Review Article

Synthetic Routes to Nickel Oxide Nanoparticles - An Overview

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Abstract

The synthesis of metal oxide, especially, Nickel oxide in the nano range has considerable attention, nowadays, due to their potential applications in various fields. Both simple and technically assisted method have been reported for the synthesis of Nickel oxide nanoparticles. This review article mainly focuses on the synthesis of NiO nanoparticles both by one step and multistep procedures using top-down and bottom-up procedures.

Keywords: Nickel oxide, Solution method, Synthesis, Metal oxides, Nanoparticles.

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Introduction

The synthesis of metal oxide materials at nano dimension integrates the materials science and technology, which leads more and more applications in Science and Technology. One step and multi-step synthesis methods of metal oxide nanoparticles by bottom-up and top-down procedures have been reported (**Table 1**).

Table 1 Various synthetic methods for the synthesis of Nanoparticles

Top-Down Procedures (Physical methods)	Bottom-Up Procedures (Chemical Methods)
Laser Ablation	Sol-Gel method
Chemical Vapour	Spray pyrolysis
Deposition (CVD)	Thermolysis
Electro Deposition	Micro Emulsion

Various methods have been reported in the literature for the synthesis of nano crystalline NiO from different precursor materials. Along with the simple methods such as sol-gel processes, thermal decomposition, chemical routes, precipitation methods, some technically assisted method like spray pyrolysis, template process, microwave assisted method, solvothermal method have also been reported.

This review focused mainly on the synthesis of NiO nanoparticles by various methods, because the nano crystalline NiO have many potential applications in various fields like nano scale optoelectronic devices such as electro chromic display [1], optical fibers, photovoltaic applications [2], sensors [3,4], environmental remediation [5], magnetic carriers for drug targeting and catalysis [6,7]. Moreover, the nano crystalline NiO exhibit novel and significant mechanical, electronic, magnetic and optical properties in comparison with their bulk counterparts [8]. Further nano crystalline NiO possesses peculiar magnetic properties related to the size and surface effects [9-12]. Therefore NiO in the nano range has been received considerable attention during the past decades. The number of research articles published for the synthesis of NiO in the nano scale for the few decade under the Chemistry and Material Science fields is shown in **Table 2**.

The number of research articles is increasing dramatically since 2011; it indicates that the need of the synthesis of NiO nanoparticles. Hence a variety of methods have been proposed for the synthesis of NiO nanoparticles. Moreover, the industrial demands have tempted to generate new method for the synthesis of size tunable, well dispersed, stable Nickel Oxide nanoparticles. The solution based methods are becoming more popular since they can yield high purity products at low cost, starting from easily available materials.

Table 2 Publication data for the synthesis of nano scale NiO under Chemistry and Material Science fields. (Ref: Sciencedirect.com)

Year	Number of publications	
	Chemistry	Material Science
1980-1990	4	6
1990-2000	137	176
2000-2010	1185	1185
2010-till date	3436	3436

Thermal Decomposition Method

Nickel oxide nanoparticles with an average diameter of about 9 nm were synthesized by Xiong Wang et al via thermal decomposition of NiC_2O_4 precursor at 450ºC [13]. Monodisperse spherical Ni nanoparticles with diameters of 2 nm, 5 nm, and 7 nm were synthesized from the thermal decomposition of a Ni–oleylamine complex. Ni nano crystals super lattices were generated via the controlled evaporation of solvent. The nanoparticles were successfully used as catalysts for the Suzuki coupling reaction, and were readily oxidized to produce NiO nanoparticles; the study was reported by J.Park, E.Kang, S.U.Son [14].

N. Dharmaraj et al. have reported the synthesis of Cubic nickel oxide (NiO) nanoparticles using a mixture of nickel acetate and poly (vinyl acetate) as precursor followed by heat treatment at 723 K. The TEM images showed that NiO nanoparticles have uniform size around 40–50 nm and well dispersion [15]. The thermal decomposition of $[Ni(acac)_2]$ in HAD at 220°C was rapid, resulting in a black precipitate which proved to be a Ni metal. In a mixture of TOPO and HAD the system was somewhat less reactive. Though the resulting material was less pure but contained well defined nano crystals of NiO; the work reported by Yang Li [16]. Since the 2D nano structures have variety of applications due to their large surface area Zhang K et.al have presented a simple and convenient method to realize two-dimensional NiO nano walls by thermal treatment of a Ni thin film deposited by sputtering onto a stainless steel substrate [17]. Their investigation reports Fatemeh Davar have found that the novel synthesis of nanoparticles Ni and NiO using bis(2-hydroxy aceto phenato) nickel(II) as precursor by thermal decomposition [18]. The preparation and characterization of single phase NiO nanoparticles was reported by Masoud Salavati-Niasari; In their study at first, nickel-o-phthalate complexes as precursor were synthesized through semisolid phase reaction method and then NiO nanoparticles were obtained via a solid-state decomposition procedure of layered coordination nickel-o-phthalate complexes formulated as $[Ni(pht)(H_2O)_2]$ and $[Ni(pht)_2]$ [19]. Nickel Oxide (NiO) nanoparticles have been synthesized by self-propagating low temperature combustion synthesis method using Nickel salt with polyethylene glycol as fuel and conformed the formation of NiO nanoparticles in the range of 20-40nm [20].

NiO nanoparticles with sizes of 3.5–12.4 nm were grown by thermal decomposing of nickel acetate at different temperatures in NaCl and Li_2CO_3 alkali salts had reported by W. J. Duan [21]. Various metal oxides were synthesized at lower decomposition temperature using metal acetyacetonates $M[(acac)_n]$, (M=Fe, Mn, Co, Ni, Cr ; n=2 or3, acac=acetyacetonate); the precursors are metal cupferronates [22-24] and metal carbonyls [25].

Sol Gel Process

Markus Niederberger have made an attempt to synthesize nanoparticles through non-aqueous Sol–gel route to metal oxide nanoparticles. The use of organic solvents under exclusion of water have become a versatile alternative to aqueous methods [26]. Naidu V.Seetala studied the synthesis of NiO nanoparticles via alumina supported Sol-Gel process [27]. A Olad and his team prepared NiO nanoparticles by the sol-gel method using Citric acid as a ligand

[28]. Nanocrystalinne NiO has been prepared by P.Jeevanantham via thermal decomposition of precursors ($NiCl_2.6H_2O$) using Sol-Gel process. The crystalline size of NiO is about 19nm [29]. In an interesting study Alagiri and M.Ponnusamy have synthesized Nickel oxide nanoparticles in the presence of agarose polysaccharide by sol-gel method [30].

In another study Lay Gaik Teoh, Kun-Dar Li have prepared, NiO nanoparticles by sol–gel method, the synthesis was accomplished by using poly(alkylene oxide) block copolymer as the surfactant, and $Ni(NO_3)_2{\cdot}6H_2O$ as the inorganic precursor [31]. P.Mallick reported the synthesis of NiO nanoparticles by sol-gel method using, nickel nitrate as precursor and ethanol as solvent [32].

Chemical Precipitation Method

NiO/MWNTs nano composites have been prepared by chemical precipitation method with the aid of sodium dodecyl sulfate (SDS) studied by Xiao-NingLiaoa [33]. Chemical precipitation is the approach utilized for the production of NiO; Materials mainly used in this project are nickel nitrate hexahydrate, sodium hydroxide, and polymeric (PVP, PEG) and cationic (CTAB) surfactants [34]. The $Ni(OH)_2$ precursors were prepared via the precipitation transformation method, which originated from $Na_2C_2O_4$, $NiSO_4 \cdot 6H_2O$ and urea. The NiO samples were successfully obtained by calcining the $Ni(OH)_2$ precursor with different calcinations methods [35].

In their research work M. M. Kashani Motlagh and his team reported the synthesis of $Ni(OH)_2$ and NiO nanoparticles with narrow size distribution and uniform shape by complexation-precipitation method using ammonia as complexing agent [36]. Nano crystalline NiO has been prepared via thermal decomposition of Nickel sulphate using homogeneous precipitation. The Crystalline size of NiO is ~2nm [37]. Avnish Kumar Arora et al synthesized nano sized NiO using precipitation method [38]. Core shell CaO-NiO mixed metal oxide nanoparticles in which CuO is the core and NiO is the Shell have been synthesized by homogeneous precipitation [39]. NiO nano materials were prepared by a homogeneous precipitation method with an aqueous solution of nickel nitrate hexhydrate and urea [40].

Regazzoni.A.C. reported the preparation of nano crystalline NiO using homogeneous precipitation method [41]. K.O. Moura made a study on the synthesis of Pure and Fe-doped NiO nanoparticles with different particles sizes by the co-precipitation method [42]. Production of NiO nanoparticles by chemical precipitation is the approach utilized in a work [43].

Chemical Methods

In a work, single crystalline, defect free metal oxide (NiO) nanoparticles with diameter ~40nm was synthesized through chemical synthesis route [44]. Crystalline cubic NiO rods with diameter ranging from nanometer to few hundred nanometers and lengths up to 10mm have been realized from a simple chemical route. The chemical reaction, of aqueous solutions of nickel chloride and sodium hydroxide with different molar ratios of $NiCl_2$ and NaOH, formed the nickel hydroxide precursor which on thermal dehydration resulted in NiO nano crystals with rod like morphology [45].

The Nano crystalline nickel zinc ferrite was prepared via chemical synthesis; Zinc nitrate, Nickel nitrate, iron nitrate, citric acid and ethylene glycol were used as precursor materials [46]. An urea-melt assisted route was designed for the synthesis of ~10nm sized Ni/NiO nanoparticles. The method consists of the thermal decomposition of a urea melted medium containing a Ni^{2+} salt and involves the olygomerisation of self combustion of the organic matrix for the formation of the Ni/NiO nanoparticles [47]. Pravistin reported the synthesis of NiO by Ethylene Glycol route. In this method NiO was prepared using $NiCl_2.6H_2O$ as precursor, ethylene glycol as solvent and agglomeration presenting agent and ammonium bicarbonate as precipitant [48]. Nano crystalline NiO has been prepared successfully by a simple chemical route using $NiCl_2{\cdot}6H_2O$ and NaOH aqueous solution at a temperature of 70°C [49]. Face centered cubic Nano crystalline nickel nanoparticles prepared at 60ºC from $NiCl_2$ precursor using hydrazine hydrate as reducing agent and EG as capping agent [50].

An attempt had been made by Feifei Tao and his team to fabricate flower-like NiO hierarchical structures with 2–5 μm diameter assembled from nano sheet building blocks via a wet-chemical method combined with thermo decomposition technology [51]. Melted $Ni_{60}Zr_{40}$ alloy, synthesized via arc melting, was quenched in liquid nitrogen and subsequently oxidized at 1073 K for 2 h. The amorphous phase, resulting from quenching, is characterized by large open porosity and consists of agglomerates of small particles. Preferential oxidation and segregation induce distinct NiO and ZrO_2 agglomerates, which are composed of nano grains. The near spherical NiO particles have a diameter in the range 10–50 nm had been synthesized [52]. Ultrafine, equated and Monodisperse oxide particles with

an average grain diameter in the range of 1–10 nm have been prepared by a two-step chemical approach: the chemical reduction of metallic salts by activated sodium hydride in tetrahydrofuran solvent, followed by oxidation of the metallic species with small amounts of O_2–N_2 gas [53].

A nickel salt–urea–H_2O ternary system has been developed for the large-scale synthesis of hierarchical α-$Ni(OH)_2$ microspheres, the solid precursor for the subsequent topotactic transition to NiO upon calcination. In this facile synthetic system, hierarchical structure is self-assembled under the cooperative direction of urea and anions in nickel salts. Thus, simply tuning the Ni salts leads to the selective construction of urchin and flowerlike hierarchical α-$Ni(OH)_2$ and NiO microspheres consisting of radial 1D nano wires and 2D nano plates, respectively [54]. NiO nanoparticles have been prepared by the decomposition of the hydroxide [55,56,57]. According to B.J. Park NiO nanoparticles have been synthesized by the oxidation of Ni nanoparticles [58]. W.Xiong et.al reported the synthesis of NiO nanoparticles by the decomposition of Nickel Oxalate [59]. T. Theivasanthi reported a work related to chemical capping synthesis of nano sized particles of nickel oxide. They found that the nanoparticles size 12nm and specific surface area 74 m^2g^{-1} [60].

Polymerized Complex Methods

Ajeet Kumar reported the synthesis of spherical, size tunable, well dispersed, stable nickel and nickel oxide nanoparticles by reduction of nickel nitrate at room temperature in a TX-100/n-hexanol/cyclohexane/water system by a reverse micro emulsion route [61]. Nano particles of fcc-NiO phase were obtained by heating the dried resin resultant of a mixture of gelatin and $NiCl_2.6H_2O$ in aqueous solution; the work was reported by A.O.G.Maia and coworkers [62]. A novel approach had been made by C.Y.Meneses and his team where the dispersed nickel oxide nanoparticles were obtained using a mixture of gelatins as organic precursor and $NiCl_2.6H_2O$ as Ni source [63]. Nickel oxide (NiO) nano crystallites with a crystal size of around 54 nm have been synthesized via the polymerized complex method [64]. Nanoparticles of nickel oxide have been prepared through a new mixed reverse micro emulsion route. Quaternary micro emulsion (water/surfactant/co surfactant/ oil-phase) was used to synthesize nickel oxide nanoparticles. The micro emulsion was prepared by Tween-80, Aerosol-OT, n-Propanol, Cyclohexane, and Nickel Chloride [65].

Masoud Salavati-Niasari and his team focused the preparation and characterization of single phase NiO nanoparticles. At first, nickel-o-phthalate complexes as precursor were synthesized through semisolid phase reaction method and then NiO nanoparticles were obtained via a solid-state decomposition procedure of layered coordination nickel-o-phthalate complexes formulated as $[Ni(pht)(H_2O)_2]$ and $[Ni(pht)_2]$ [66]. Ting Wang et al reported the synthesis of NiO nanoparticles on the silica surface by adsorption phase synthesis. The adsorption and preparation experiments of different reactants were designed to select suitable reactants for the synthesis of NiO particles [67].

Thermolysis Methods

Using hydrothermal method, nano structures of various kinds of oxides: ZnO, CuO, NiO, and Ga_2O_3 were fabricated from aqueous solutions of the respective metal nitrate hydrate and hexamethylenetetramine by hydrothermal method [68]. A.G.Al-Schemi et al demonstrated the hydrothermal calcinations method for the synthesis of NiO nanoparticles by using $Ni(NO_3)_2.6H_2O$, HMT and NaOH as starting materials [69]. Nanoparticles of NiO with average diameters in the 3–24 nm range have been prepared by the decomposition of nickel cupferronate or acetate under solvothermal conditions [70].

K. Anandan and V. Rajendran have synthesized without and with cationic surfactant (CTAB) assisted NiO nanoparticles via facile solvothermal process [71]. The present experimental design is to synthesize novel nanoparticles of Iron and Nickel oxides to be used as catalysts for in situ removal of different pollutants discharged from various industries. Particle size of synthesized iron and nickel oxides nanoparticles was characteristic of ~28-36 nm and ~48-56, respectively [72].

Sonochemical Synthesis

Nickel oxide (NiO) nano powder was obtained during a Sonochemical method. Nickel hydroxide precursor, prepared by addition of sodium hydroxide (NaOH) to nickel nitrate $Ni(NO_3)_2$ solution and vigorously stirring until the pH becomes 7.2. Ultrasounds waves were applied during precipitation period and after drying in oven nano powder NiO was achieved [73]. A sonochemistry-based synthetic method was used to produce nano crystalline nickel oxide powder with 20 nm average crystallite diameter from $Ni(OH)_2$ precursor. Ultrasound waves were applied to the

primary solution to intensify the $Ni(OH)_2$ precipitation. Dried precipitates were calcined at 320ºC to form Nano crystalline NiO particles [74]. A sono chemistry based synthetic method was used to produce Nano crystalline nickel oxide powder with ~20nm average crystalline diameter from $Ni(OH)_2$ precursor. Using Sonochemical waves resulted in lowering of the size of the NiO crystallites [75].

Nickel oxide nanoparticles 6-24 nm size have been prepared by a levitation –Jet method based on metal vapour condensation in a mixture of gaseous streams of helium and air (or Oxygen) [76]. Nanoparticles of ZnO, MgO and NiO were produces from droplets of aqueous salt solution in the flame spray pyrolysis reactor. The authors reported that nanoparticles are produced from nitrate as well as acetate salt precursor solution when propane- oxygen diffusion flame is used to decompose aqueous aerosol droplets [77]. Seo. D.J and his team prepared the NiO nanoparticles via spray pyrolysis method [78].

Laser Ablation Methods

Nickel oxide nanoparticles were fabricated by a laser ablation technique using the third harmonic of an Nd:YAG laser and sintered NiO targets in an on-axis configuration under argon pressures of 0.67, 1.33, and 2.00 Pa [79]. Pulsed laser ablation in liquid (PLAL) has been widely applied for the generation of nanoparticles. The authors have reported on the generation of NiO nanoparticles using a high-power, high-brightness continuous wave (CW) fiber laser source at a wavelength of 1,070 nm [80]. NiO nanoparticles with average particle size of 25 nm were prepared by anodic arc method [81].

Microwave-Assisted Preparations

By using a domestic microwave furnace and depending on the nickel precursor used, either tetra hydrated nickel acetate or dehydrated nickel formate, different nano sized materials- Ni/NiO composites, Ni metal, or NiO are obtained [82]. Edward K.Nyuttu and his coworkers reported the synthesis of spinel metal oxide nanoparticles by microwave assisted in-situ mixing [83]. NiO has been synthesized by microwave induced chemical synthesis route using metal organic complex of nickel in a domestic type microwave oven (2.45 GHz).A novel metal organic complex of nickel, viz., a β-ketoester of nickel, synthesized and characterized as a part of this work, was employed as the precursor material [84]. Nanoparticles of nickel oxide with an average crystalline size of 45-55nm have been prepared by microwave irradiation using nickel nitrate and sodium hydroxide solutions as the starting materials. The precipitation of nickel hydroxide after dry was irradiated by microwave radiation for short time [85]. Nickel oxide (NiO) nano-particles were produced via a rapid microwave-assisted method using $Ni(OH)_2$ precursor which is obtained from nickel nitrate and sodium hydroxide [86]. Nano-sized NiO was synthesized by microwave firing through the thermal decomposition of nickel carboxylate precursor employing glycine as a fuel [87]. Nanoparticles of ZnO, MgO and NiO were produced from droplets of aqueous salt solution in the flame spray pyrolysis reactor. The authors reported that nanoparticles are produced from nitrate as well as acetate salt precursor solution when propane–oxygen diffusion flame is used to decompose aqueous aerosol droplets [88].

Chemical Vapour Deposition Methods

The ultrahigh pseudo capacitive nickel oxide nano particulate films on the nickel foils are prepared through a two-step in which nickel hydroxides are electrodeposited on the nickel substrate in 0.08 M $Ni(NO_3)_2$ aqueous solution and then they are thermally transformed into uniform nickel oxide nanoparticles [89]. Ni/NiO nanoparticles were synthesized by metal organics chemical vapor deposition of nickel acetylacetonate in an externally heated tube flow reactor at moderate temperatures, up to 500°C. Particle production and characteristics were studied by evaluating the effects of reactor temperature, precursor concentration, and flow rate through the reactor [90].

Solution Methods

Nickel oxide (NiO) nano wire was synthesized by a simple aqueous solution method with urea as precipitant [91]. A size series of ligand-stabilized Ni nanoparticles with diameters between 8−24 nm was prepared by solution chemistry, followed by solution-phase oxidation with atmospheric oxygen at 200°C to form Ni(core)/NiO(shell) nanoparticles with shell thicknesses of 2−3 nm [92]. The nano crystalline nickel oxide (NiO) particles have been successfully prepared by a simple, fast, economical and eco -friendly solution -combustion method using $Ni(NO_3)_2$ $.6H_2O$ (oxidizer) and sugar (dextrose as fuel) [93]. Nano crystalline metal oxides (ZnO, NiO, and SnO2) powders with an average particle diameter of 18–55 nm have been successfully prepared with the surfactant-mediated method. The

cationic surfactant (cetyl trimethyl ammonium bromide, CTAB) and the hydrous metal chlorides $ZnCl_2$. $2H_2O$, $NiCl_2.6H_2O$, and $SnCl_4.5H_2O$ [94]. A new method was developed to grow Zinc oxide nano flower via organic solvent assisted growth technique from solution method, without using surfactant, complexing agent or stabilizer [95-98].

Conclusion

The research in the synthesis of nickel oxide nanoparticles is increased recently due to the novel characteristics and properties. Further the nickel oxide in the nano scale has a wide variety of applications in various fields. Although a number of publications are witnessing the synthesis of nano crystalline nickel oxide by different methods, new methods and/or simplified methods are needed for the synthesis. Based on this review, further research efforts have to be made, in future, for the synthesis of Nickel oxide nanoparticles.

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