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## THE SYNTHESIS METHODS OF NICKEL OXIDE NANOSTRUCTURES – A BRIEF REVIEW

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Supercapacitors, Li-ion batteries, gas sensors, and electrochromic devices are expected to play a major role in the development of sustainable technologies. Recent progress has demonstrated that nanostructured nickel oxides are very promising candidates for efficient energy conversion and storage systems. Recently, there is a growing interest in nickel oxide nanoparticles due to their unique physical and chemical properties. In this work, the synthesis of nickel oxide nanoparticles is primarily categorized with the preparation method. This review also provides a comparative overview of the influence of technological conditions on the properties of nickel oxide nanoparticles.

**Keywords:** *nickel oxide, nanoparticle, crystallite size.*

### 1 INTRODUCTION

In recent years, among transition metal oxides nickel oxide (NiO) nanostructures have attracted much attention owing to their unique optical, electrical, catalytic, and magnetic properties. It is a promising candidate for a wide range of applications like gas sensors, supercapacitor, anode material for Li-ion batteries, catalyst, dye-sensitized photocathodes, optoelectronic devices etc. [1–6]. Moreover, hierarchical porous NiO architectures were found to be effective adsorbents for the removal of congo red (CR) pollutant from wastewater by Zeng et al. [7], which is an important issue to decontaminate the organic dye-containing water from an environmental viewpoint. Flower-like NiO microspheres exhibited maximum adsorption capacity ( $535 \text{ mg g}^{-1}$ ) in comparison to other adsorbents for CR [8]. NiO is also considered a potential candidate for application in smart windows, drug delivery and MRI agent [9–10]. Bulk NiO adopts a rock salt structure and it is a p-type semiconductor with a wide band gap in the range of 3.6–4.0 eV [11]. Nanostructured NiO exhibits electronic, dielectric behavior and high reversible capability because of quantum size confinement and high surface energy. NiO with a variety of morphologies such as nanosheets, nanorods, nanowires, hollow microspheres, nanoflowers, nanotubes, nanoplatelets, nanofibers has been reported in the literature [2, 8, 12–17]. Properties change with altering the size, shape and morphology of nanoparticles. So that bulk NiO is antiferromagnetic with a Neel temperature of 523 K, while a reduction in particle size to nanometer-scale results in superparamagnetic, ferromagnetic behavior due to the defects in the crystal lattice. Extensive studies have been carried out to synthesize NiO nanostructures. The preparation methods of NiO nanoparticles include sol-gel, hydrothermal, precipitation, combustion, electrochemical, pulsed laser ablation, electrospinning techniques [16–20] etc. By this review paper, we could get more information about nanostructured nickel oxide, its basic synthesis techniques and applications in many fields.

### 2 SYNTHESIS METHODS OF NiO NANOPARTICLES

The preparation of NiO nanostructures with control-

led size, shape and morphology have always been the focus of researchers' attention for various applications. Up to date, many attempts have been made to synthesize nanostructured NiO. In this review, we will selectively summarize the main and general synthesis methods of NiO nanoparticles.

#### 2.1 Precipitation method

The precipitation method is simple and inexpensive so that it has the potential for the synthesis of nickel oxide and other transition metal oxides. Particle size can be easily controlled by changing the parameters like concentration of reagent, pH of the solution using this method. This method is based on precipitating the nickel salts with different precipitating agents such as NaOH,  $\text{NH}_3$ ,  $(\text{NH}_4)_2\text{CO}_3$ ,  $\text{NH}_4\text{HCO}_3$ , urea, LiOH and ethanolamine [21–27]. The stabilizing agent was not used in most experiments. Xiang Yi Deng [26] synthesized NiO nanoparticles with an average size of 9 nm using  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  and  $\text{NH}_3 \cdot \text{H}_2\text{O}$  as the raw materials and  $\text{H}_2\text{O}$  as a solvent. It was determined reaction conditions for synthesis in this research work. In another work [27], NiO nanoparticles were synthesized using nickel nitrate hexahydrate and sodium hydroxide as a nickel source and precipitating agent in the presence of various surfactants (polyvinylpyrrolidone, polyethylene glycol and cetyltrimethylammonium bromide). To learn the effect of calcination temperature on particle size, the powders were calcined at 300 °C, 450 °C and 600 °C for 2 h. Particle size increased with increasing calcination temperature. Different surfactants do not affect the morphology of the particles, sphere-like morphology was observed in all cases. The results indicated that CTAB is weaker than the other two stabilizers. Conducting nanocomposite of NiO with polyaniline was obtained in the aqueous medium using polyvinyl alcohol and hydroxypropyl cellulose as a surfactant by Aleahmad et al. [28] They investigated the electrical conductivity and thermal stability of nanocomposite as a function of the NiO concentration in the reaction solution. NiO nanotubes were obtained using dimethylglyoxime as a precipitant. The obtained nanotubes display good antibacterial properties and electrochemical capacitance [29]. Flower-

like NiO nanostructure was synthesized using  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  and ammonia as starting materials. The nanostructure was then applied as the modifier of a carbon paste electrode to study electrocatalytic oxidation of choline [30]. Albert Irudayaraj et al. [31] obtained undoped and Fe-doping NiO nanoparticles by this method. For synthesis, 0.5M nickel nitrate and 1M aqueous sodium hydroxide solution were used. The effect of the dopant on the structural, optical and magnetic properties of synthesized nanoparticles had been investigated. According to Vibrational Sample Magnetometer (VSM) results, NiO nanoparticles without Fe doping exhibit superparamagnetic behavior, while Fe-doped NiO nanoparticles exhibit ferromagnetic behavior at room temperature. NiO nanoparticles were prepared using nickel acetate tetrahydrate  $[\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}]$  and  $\text{NH}_3$  solutions following calcination at 400 °C for 2 h. Structural and dielectric properties were investigated depending on compositional variability. To vary the composition of NiO, the experiment was also carried out in the presence of 1%  $\text{H}_2\text{O}_2$ . Non-stoichiometric samples ( $\text{Ni}_{40}\text{O}_{60}$ ) with cubic structure and near stoichiometric samples ( $\text{Ni}_{48}\text{O}_{52}$ ) with amorphous nature were obtained without and with 1%  $\text{H}_2\text{O}_2$ , respectively. The dielectric constant was estimated as 35 and 40 for  $\text{Ni}_{40}\text{O}_{60}$  and  $\text{Ni}_{48}\text{O}_{52}$  nanoparticles [32]. Ethylene glycol was used as a solvent and dispersing agent for the synthesis of NiO nanoparticles and the reaction mechanism of the chemical process was proposed [33]. NiO nanoparticles with different sizes were synthesized in the presence of various nickel salts and the impact of anions on the magnetic properties was investigated [22]. It had been determined that using nickel sulfate and nickel acetate lead to smaller crystallite size (~2 nm) and these nanoparticles show higher saturation magnetization (~1.2–1.8 emu/g), while the use of nickel nitrate or nickel chloride leads to relatively bigger crystallite size (~4–6 nm) showing lower saturation magnetization values (~0.1–0.4 emu/g).

## 2.2 Thermal decomposition method

Thermal decomposition is one of the most known methods to synthesize different metal oxide nanoparticles.

El-Kemary et al. [34] successfully synthesized NiO nanoparticles by the reaction of nickel chloride with hydrazine at room temperature and thermal decomposition of the nickel hydroxide ( $\text{Ni}(\text{OH})_2$ ) at ~400 °C. They mainly studied the interactions between NiO nanoparticles and glucose to improve the biomedical applications of NiO. It was revealed that optical properties and stability of glucose changes with NiO nanoparticles. NiO nanoparticles were obtained utilizing only nickel nitrate and polyvinyl pyrrolidone as a precursor and capping agent [35]. The influence of thermal treatment temperature on the particle size, morphology, optical and magnetic properties was investigated. Particle size increased from 15 nm to 35 nm with increasing temperature from 500 to 800 °C. Optical band gaps of nanoparticles were estimated as 3.60, 3.57, 3.55, and 3.51 eV for samples calcined at 500, 600, 700 and 800 °C. Porous cubic nickel oxide nanostructures having excellent electrochemical properties were obtained thermal treatment of nickel oxalate at 350 °C for 10 min [36]. A new precursor nickel octanoate  $\text{Ni}(\text{octa})_2$  was proposed by Fereshteh et al. [37] for the synthesis of NiO nanoparticles. Using this method, nanoparticles with a crystallite size of 25 nm were obtained in the presence of oleylamine ( $\text{C}_{18}\text{H}_{37}\text{N}$ ) and triphenylphosphine ( $\text{C}_{18}\text{H}_{15}\text{P}$ ) as surfactants. Oleylamine acts as both the medium and stabilizing agent. As one type of thermal decomposition, solid-state decomposition was also used for the synthesis of NiO nanostructures. Salavati-Niasari and co-workers [38] reported the synthesis of NiO nanoparticles from nickel-o-phthalate complexes  $[\text{Ni}(\text{pht})(\text{H}_2\text{O})_2]$  and  $[\text{Ni}(\text{pht})_2]$ . They learned the influence of calcination temperature and metal-to-ligand ratio on the particle size and morphology. The smallest particle size (15 nm) was observed in the condition of  $\text{Ni}/\text{pht} = 1:2$  precursor at 500 °C calcination temperature, while nanoparticle size increased (29 nm) at 700 °C due to the agglomeration of particles. When the metal to ligand ratio was  $\text{Ni}/\text{pht} = 1:1$ , nanoparticle size was estimated as 24 nm. In this process, O-phthalic acid was used as a chelating ligand. For this reason, in the condition of metal to ligand ratio 1:2 since the number of phthalate

Table 1. Synthesis of nickel oxide nanoparticles by precipitation method

#	Chemical compos.	Structure	Morphology	Particle size (nm)	Remarks (t, pH, C)	Ref.
1	NiO	cubic	nanocrystal	9 (by XRD and TEM)	$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}/\text{NH}_3 \cdot \text{H}_2\text{O} = 1:1.15-1.30$ , pH=6.5–9.5, at room temperature, stirring time=20–30 min	[26]
2	NiO	—	sphere like	<55 (by SEM)	$\text{NiNO}_3 \cdot 6\text{H}_2\text{O}$ as a source, NaOH (as a precipitator), PVP (as surfactant)	[27]
				35–45	PEG	
				>45	CTAB	

Table 2. Synthesis of nickel oxide nanoparticles by thermal decomposition method

#	Chemical compos.	Structure	Morphology	Particle size (nm)	Remarks (t, pH, C)	Ref
1	NiO	cubic	plate-like	10–40 (by SEM and TEM)	$\text{Ni}(\text{II})$ Schiff base complex as a precursor, T=450 °C for 3 h in air atmosphere	[40]
2	NiO	hexagonal	nanoplate	d=300 thickness=20	T=300 °C for 3 h in air	[41]

molecules is relatively high, metal ions were capped completely. Thus, aggregation of particles was prevented. Spherical NiO nanoparticles in the range of 10–20 nm were prepared by thermal decomposition of  $\text{NiCl}_2(2,9\text{-Dimethyl-1,10-phenanthroline})\cdot\text{H}_2\text{O}$  complex at 400 °C [39]. Plate-like shape NiO nanoparticles were synthesized from the nickel (II) Schiff base complex as a new precursor by Khalaji [40].

### 2.3 Chemical bath deposition (CBD)

CBD is a simple and cost-effective method, which is possible to control the nanoparticle size and film thickness by varying the reagent concentration, pH of the solution, bath temperature and deposition time. In this method, thin films were deposited immersing the substrates vertically in the solution containing precursors. NiO thin films with different morphologies were successfully synthesized on various substrates such as glass, silicon wafer, carbon cloth [42–45]. Generally, for the synthesis of NiO thin films, nickel salt and urea were used as starting materials. On the other hand, reports of NiO films by CBD employing complexing agents such as ammonium and triethanolamine are studied [43]. This method is also convenient for large area deposition with well-specified nano-porosities, which make NiO attractive for applications including dye-sensitized solar cells, electrochemical supercapacitor [44]. For the first time, ternary oxide thin films composed of Ni and Co oxides were deposited on glass and steel substrates by Ezema et al. [45]. For the synthesis, 0.1M of  $\text{CoSO}_4$  and  $\text{NiSO}_4$  were used as the cobalt and nickel ion sources. The pH of the solution was adjusted to ~12 by ammonia solution. NiO thin films were deposited on a microscopic glass slide at various deposition times using nickel nitrate solution [ $\text{Ni}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$ ] as a cationic precursor and urea [ $\text{CO}(\text{NH}_2)_2$ ] as a complexing agent by Mitra et al.. The film thickness increased from ~120 nm to ~307 nm with increasing deposition time from 30 min. to 120 min. Particle size and optical band gap energy decreased with increasing deposition time. This is attributed to an enhanced degree of crystallinity and additional energy levels due to the incorporation of more oxygen atoms in the films [46]. Highly porous nickel oxide thin films were successfully synthesized on indium tin oxide (ITO) glass by Xia et al. [47] and electrochromic properties were mainly discussed in this work. It was found that the film annealed at 300 °C showed the high coloration efficiency (CE) of  $42 \text{ cm}^2 \text{ }^\circ\text{C}^{-1}$  at 550 nm, with a variation of transmittance up to 82%. The porous structure plays an important role for the enhancement of electrochromic properties. Using the same precursors, only decreasing their concentration Yang et al. [48] synthesized porous

NiO thin films and it exhibited a high CE value of  $99.51 \text{ cm}^2/^\circ\text{C}^{-1}$ .

### 2.4 Successive ionic layer adsorption and reaction (SILAR)

Successive ionic layer adsorption and reaction (SILAR) is a simple, environmentally friendly and reproducible technique for the deposition of metal oxides in the form of thin films on different substrates. This method is a modified chemical bath deposition (CBD) process and the difference between these methods is that thin films are synthesized by dipping a substrate into separately placed cationic and anionic precursor solutions. Particle size, morphology and film thickness can be easily controlled by varying the precursor concentration, pH of the solution, number of deposition cycles, adsorption, reaction and rinsing time durations. NiO thin films were grown on a glass substrate at room temperature using this method [49]. In this experiment, alkaline solution of nickel chloride ( $\text{NiCl}_2$ ) with ammonia (25–28%) and hot water (90 °C) were used as cationic and anionic precursors, respectively. It has been determined that the film thickness influences structural, optical, electric and surface properties. The porous nanoflake-like structure of NiO thin films was synthesized on stainless steel substrate for supercapacitor applications [50]. To optimize the reaction parameters, the experiment was carried out at various reaction temperatures (318K, 333K, 348K) and time periods (20, 30, 40 seconds). The strongly adherent and uniform thin films were obtained at 333K temperature and time period of 30 seconds. NiO thin films with platelet-type morphology were synthesized on glass substrate using nickel (II) chloride and ammonia as precursors for gas sensor application [51]. K. S. Klepikova et al. deposited NiO and Li-doped NiO thin films with activation energy  $E_a = 0.1 \text{ eV}$  and  $E_a = 0.25\text{--}0.31 \text{ eV}$  on glass substrate using the same precursors. In order to obtain NiO:Li films, the glass substrates covered by NiO films were immersed into lithium hydroxide (LiOH) aqueous solution at room temperature for 20 min following annealed at 550 °C. [52] Sachindranath Das and co-workers used 0.1M nickel nitrate [ $\text{Ni}(\text{NO}_3)_2$ ] in ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) (pH~9.0) as a cationic precursor and 1% hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) maintained at a temperature of 90 °C–100 °C as an anionic precursor for the deposition of NiO thin films [53]. They investigated the influence of the dipping cycle on the structure, morphology and electrochemical performance of NiO films. It has been concluded that thin films deposited at 40 cycles show the highest specific capacitance of  $1341 \text{ F}\cdot\text{g}^{-1}$  as compared to previous results.

Table 3. Synthesis of nickel oxide nanoparticles by a chemical bath deposition method

#	Chemical compos.	Structure	Morphology	Remarks (t, pH, C)	Ref
1	NiO	cubic	porous structure with flakes	0.25M $\text{Ni}(\text{NO}_3)_2$ and 0.25M urea, pH=5, T=350 °C for 2 h in air	[44]
2	NiO	rhombohedral	porous nanoflake	0.37M $\text{NiSO}_4\cdot 6\text{H}_2\text{O}$ , 0.06M $\text{K}_2\text{S}_2\text{O}_8$ and ammonia (20–30%), T=400 °C for 3 h	[48]

Table 4. Synthesis of nickel oxide nanoparticles by SILAR method

#	Chemical compos.	Structure	Morphology	Particle size (nm)	Band gap	Remarks (t, pH, C)	Ref.
1	NiO	cubic	thin film	D = 8.8–9.3 (by XRD) thickness = 125–312 (depending on deposition cycle)	3.7 eV	at room temperature	[49]
2	NiO	cubic	thin film	—	—	0.1M NiCl <sub>2</sub> ·6H <sub>2</sub> O was used as a source of Ni, pH=12, onto glass slides	[51]

Table 5. Synthesis of nickel oxide nanoparticles by sonochemical method

#	Chemical compos.	Structure	Morphology	Particle size (nm)	Remarks (t, pH, C)	Ref.
1	NiO	cubic	spherical	D = 20–150 nm (by XRD)	0.1 and 0.2M of Ni(CH <sub>3</sub> COO) <sub>2</sub> ·2H <sub>2</sub> O, 0.1M NaOH and PEG as surfactant, T=500 °C for 30 min.	[59]
2	NiO	face-centered cubic	spherical	D = 6 nm (250 °C), 21 nm (450 °C), 41 nm (650 °C) [by XRD]	1M NiCl <sub>2</sub> ·6H <sub>2</sub> O and 2M NaOH, T=250 °C (450 °C, 650 °C) for 3 h	[60]

## 2.5 Sonochemical method

The basic principle of sonochemistry is related to the acoustic cavitation phenomenon, which occurs in several steps including nucleation, growth and finally the implosive collapse of bubbles in a liquid medium. This causes local hot spots with a temperature of around 5000 °C under pressures of 500 atm, heating and cooling rates of more than 10<sup>10</sup> K/s and as a result of these extreme conditions high-energy chemical reactions occur. The main advantage of this method is that the nanoparticle size, shape and morphology can be controlled by changing the parameters like ultrasonic power, current density, sonication time, pH and reaction temperature. Ghobadifard et al. synthesized NiO nanoparticles by this method using nickel acetate [Ni(CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O], sodium hydroxide (NaOH) as precursors and polyethylene glycol (PEG) as surfactant [54]. Particle size was influenced by varying the precursor concentration, sonication time and ultrasound power. The smallest size (20 nm) was obtained at 0.1 M solution of nickel salt, 12–18 W ultrasound power and 3 h sonication time. Gas sensing activity of synthesized nanoparticles towards NO<sub>2</sub> and CO gases was investigated. NiO nanoparticles were synthesized using nickel sulfate (NiSO<sub>4</sub>·6H<sub>2</sub>O) and NaOH in the presence of CTAB as stabilizing agent followed by calcination at 600 °C for 2 h. The particle size between 35 nm and 117 nm was reported [55]. Ultrasonic irradiation of mixed aqueous solution of nickel sulfate hexahydrate and urea and then calcination of the NiO<sub>2.45</sub>C<sub>0.74</sub>N<sub>0.25</sub>H<sub>2.90</sub> precursor at 500 °C for 3 h leads to the formation of porous NiO microsphere [56]. Ultrasound-assisted synthesis of PVA/NiO nanocomposite film was reported by Anbarasan et al. [57] Cubic shape NiO nanoparticles with average crystallite size of ~20 nm were synthesized using 0.1 M nickel nitrate Ni(NO<sub>3</sub>)<sub>2</sub> and 0.1 M NaOH followed by calcination of Ni(OH)<sub>2</sub> precipitation at 320 °C for 1 h [58].

## 2.6 Sol-gel method

The sol-gel method is a simple, inexpensive, low-cost technique and this route has been widely used for the

synthesis of NiO nanoparticles due to these characteristics. Spherical NiO nanoparticles with an average diameter of about 32.9 nm were synthesized in the presence of Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O, isopropanol alcohol and polyethylene glycol. Triton X-100 [C<sub>14</sub>H<sub>22</sub>O (C<sub>2</sub>H<sub>4</sub>O)<sub>n</sub>] was used as a stabilizer to prevent aggregation of particles. The optimum calcination temperature was identified as 450 °C by thermal analysis [18]. Jahromi and co-workers [61] obtained NiO nanoparticles by this method using nickel nitrate and gelatin as precursor and polymerization agent, respectively. They investigated the influence of annealing ambient, including air, O<sub>2</sub> and annealing temperature on structural, morphological and magnetic properties of synthesized nanoparticles. It was concluded that the samples annealed in O<sub>2</sub> atmosphere were more crystalline than the air annealed due to the reduction O-vacancy and strain in the lattice. For this reason, the magnetization of the air-annealed NiO nanoparticles has been found to be higher than the O<sub>2</sub>-annealed nanoparticles. Bose et al. [62] reported the synthesis of mesoporous NiO nanosheets by this method using Aerosol-OT, Bis(2-ethyl hexyl) sulfosuccinate sodium salt (AOT) as anionic surfactant. The effect of different amounts of surfactant on the morphology and catalytic activity towards CO oxidation was investigated. 1mmol solution of surfactant was selected as an optimum concentration due to the higher surface area of NiO nanosheet for catalytic oxidation. In another experiment [63] poly(alkylene oxide) block copolymer was used as a surfactant. NiO nanoparticles with a crystallite size of 10.2 nm were synthesized by this method in a gelatin medium. With increasing calcination temperature from 400° C to 700° C, agglomeration was occurred because of the high surface energy of particles and particle size increased to 48.6 nm [64]. Saleh et al. [65] investigated the effect of annealing temperature on the size and electrocatalytic activity of NiO nanoparticles prepared using nickel nitrate and citric acid. The enhancement in electrocatalytic properties was observed for NiO nanoparticles annealed at 200 °C due to the smallest size. Using the same precursors Ying Wu and co-workers

[66] learned the influence of the ratio of the precursors, pH of the solution, heating rate, calcination temperature and time on physical properties of synthesized NiO nanoparticles. The smallest size (8.1 nm) with the highest surface area ( $105 \text{ m}^2 \text{ g}^{-1}$ ) was obtained at  $400 \text{ }^\circ\text{C}$  calcination temperature for 4 h in 1 ratio of citric acid to nitrate. NiO thin films were deposited on a glass substrate by a sol-gel spin coating method using nickel acetate  $[\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}]$  and methanol as precursors following sintered between  $400 \text{ }^\circ\text{C}$  and  $700 \text{ }^\circ\text{C}$  [67].

### 2.7 Hydrothermal/solvothermal method

The hydrothermal method has been widely used for the synthesis of NiO nanostructures with different sizes and morphology. NiO nanowires were successfully synthesized by a hydrothermal reaction of  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  and sodium oxalate ( $\text{Na}_2\text{C}_2\text{O}_4$ ) at ethylene glycol (EG) media at  $200 \text{ }^\circ\text{C}$  for 24 h [68]. Shah [69] prepared NiO nanoparticles by a soft reaction of nickel powder and water at  $100 \text{ }^\circ\text{C}$ . Organic dispersant or capping agent wasn't used in this experiment and water acted both as a solvent and a source of oxygen. The formation of oxide nanostructure occurred by the evolution of hydrogen gas. It was shown that the reaction time has a significant effect on the particle size and morphology. Nanoparticle size increased with increasing reaction time from 12 h to 24 h. In 36 h of reaction time, changes in morphology were observed from spherical to flower-like. Porous NiO nanoslices, nanoplates and nanocolumns were prepared at three different pH conditions (pH~12, 13, and 14) in an autoclave maintained at  $160 \text{ }^\circ\text{C}$  for 8 h and subsequent calcination of  $\beta\text{-Ni}(\text{OH})_2$  [70]. Morphology and size control of NiO nanoparticles was achieved by hydrothermal route using various precipitating agents such as  $\text{NH}_3$  and  $\text{NaOH}$  [71]. The choice of surfactant plays a critical role according to a report by Zeng et al. [72]. Nanocrystalline NiO nanoplates were formed in an autoclave using nickel sulphate and triethylamine (TEA) [73]. Using urea as a hydrolysis-controlling agent, coralloid nanostructured nickel hydroxide hydrate formed within just 3 minutes by employ-

ing microwave-assisted hydrothermal route, which after calcination at  $400 \text{ }^\circ\text{C}$  for 3 h resulted in NiO [74]. V. Rajendran and K. Anandan discussed the optical properties of NiO nanocrystals with different morphologies like spherical, rod and hexagonal obtained using various ionic surfactants like CTAB, sodium dodecyl sulfate (SDS) and PEG by solvothermal method at  $180 \text{ }^\circ\text{C}$  [75]. Self-assembled 3D rose-like NiO nanostructure was prepared by Lai and colleagues utilizing nickel chloride and sodium acetate under solvothermal conditions in the presence of PEG as surfactant [76]. They found that nanoparticle size and morphology were strongly influenced by altering the reaction time and molecular weight of surfactant. Lamellar morphology was observed in the absence of PEG while using surfactant flower-like architecture formed. Small size particles with rod-like morphology were obtained at  $100 \text{ }^\circ\text{C}$  because of the strong adsorption of PEG on the nanoparticle surface and strong coordination between EG and  $\text{Ni}^{2+}$  ions. As the temperature was raised to  $190 \text{ }^\circ\text{C}$ , nanoplatelets aggregate into self-assembled rose-like microstructure due to the weak adsorption of surfactant on the crystal surface.

### 3. CONCLUSION

This review article focuses on the various synthesis techniques of the nickel oxide nanostructures. Seven kinds of typical and most used methods for nickel oxide with different nanostructures are systematically summarized. The precipitation method is very often used to obtain NiO nanostructures because it is a facile and cheap approach. Comparatively, NiO nanoparticles with different morphologies were obtained by the hydrothermal/solvothermal method. The physical and chemical properties of nanoparticles also depend on morphology. However, the disadvantage of this method is that needs high temperature, pressure, and it requires a long reaction time. The main reaction parameters that influence the size, structure, composition, shape and morphology are described above. Temperature and pH play an important role in obtaining nanoparticles.

Table 6. Synthesis of nickel oxide nanoparticles by sol-gel method

#	Chemical compos.	Structure	Morphology	Particle size (nm)	Band gap	Remarks (t, pH, C)	Ref.
1	NiO	face-centered cubic	nanoparticle	$10 \pm 0.2$ (by TEM)	—	$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ as a source, gelatine, water bath $T=80 \text{ }^\circ\text{C}$ for 12 h	[64]
2	NiO	cubic	thin film	cryst. size = 11.02 ( $400 \text{ }^\circ\text{C}$ )	3.86 eV	Ni(CH <sub>3</sub> COO) <sub>2</sub> ·4H <sub>2</sub> O as a source of Ni, 40 ml methanol, $60 \text{ }^\circ\text{C}$ for 1 h	[67]
				27.80 ( $500 \text{ }^\circ\text{C}$ )	3.69 eV		
				37.80 ( $600 \text{ }^\circ\text{C}$ )	3.60 eV		
				38.14 ( $700 \text{ }^\circ\text{C}$ )	3.47 eV		

Table 7. Synthesis of nickel oxide nanoparticles by hydrothermal/solvothermal method

#	Chemical compos.	Structure	Morphology	Particle size (nm)	Remarks (t, pH, C)	Ref.
1	NiO	cubic	nanoplate	96 (by FE-SEM, HR-TEM)	$\text{NiSO}_4$ as a source, 0.01M TEA solution, stirring time=2 h, $T=600 \text{ }^\circ\text{C}$ for 6 h	[73]
2	NiO	cubic	nanowire	$d=60$ (by SEM)	$T=200 \text{ }^\circ\text{C}$ for 24 h	[68]
3	NiO	—	nanorod	$d=25\text{--}65$	Ni foam act both as a substrate and Ni source, $T=140 \text{ }^\circ\text{C}$ for 24 h	[77]

We should pay special attention to the specific surface area, size, and phase purity of the nickel oxide nanostructures. As the size decreases, the specific surface area of the particles increases and it causes more surface reactive sites, which is the main requirement for gas sensor applications. Depending on the application area, the choice of an appropriate synthesis method for NiO nanoparticles with desirable properties is an essential factor.

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## НИКЕЛЬ ОКСИДІНІҢ НАНОҚҰРЫЛЫМДАРЫН СИНТЕЗДЕУ ӘДІСТЕРІ – ҚЫСҚАША ШОЛУ

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Суперконденсаторлар, литий-ионды батареялар, газ қадағалары және электрохроматикалық құрылғылар тұрақты технологияны дамытуда маңызды рөл атқарады деп күтілуде. Кейінгі кезде қол жеткізілген прогресс нанокұрылымды никель оксидтерінің энергияны конверсиялау мен сақтаудың тиімді жүйелері үшін келешегі бар кандидат екенін көрсетті. Соңғы уақытта никель оксидінің нанобөлшектеріне олардың ерекше физикалық және химиялық қасиеттеріне байланысты қызығушылық артып келеді. Бұл жұмыста никель оксидінің нанобөлшектерінің синтезі ең алдымен өндіріс әдісімен жіктеледі. Сондай-ақ бұл жұмыста технологиялық жағдайлардың никель оксиді нанобөлшектерінің қасиеттеріне әсері туралы салыстырмалы шолу жасалған.

**Түйін сөздер:** никель оксиді, нанобөлшектер, кристалл көлемі.

## МЕТОДЫ СИНТЕЗА НАНОСТРУКТУР ОКСИДА НИКЕЛЯ – КРАТКИЙ ОБЗОР

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Предполагается, что суперконденсаторы, ионно-литиевые батареи, газовые датчики и электрохроматические устройства будут играть важную роль в разработке устойчивых технологий. Достигнутый в последнее время прогресс показал, что наноструктурированные оксиды никеля являются весьма перспективными кандидатами для эффективных систем преобразования и хранения энергии. В последнее время интерес растет к наночастицам оксида никеля ввиду их уникальных физических и химических свойств. В данной работе синтез наночастиц оксида никеля в первую очередь классифицируется методом получения. В данном обзоре также дается сравнительный обзор влияния технологических условий на свойства наночастиц оксида никеля.

**Ключевые слова:** оксид никеля, наночастицы, кристаллический размер.