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METHODS FOR SYNTHESIS OF NICKEL NANOPARTICLE PROPERTIES FROM PREGNANT LEACH SOLUTION OF LATERITE

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Abstract

In recent years, the synthesis of nanoparticles has been primarily focused on metal oxides because of their large surface area and good adsorption properties. The metal oxide commonly used in industry is nickel oxide (NiO) because it can be used for various applications, such as electrodes, catalysts, and sensors, and in multiple applications ranging from electrochemical detection to energy storage and environmental remediation. The method in the process of synthesizing nickel oxide nanoparticles which are considered to produce optimal products is the sol-gel method. The sol-gel method is a chemical method for synthesizing nanostructures. The process starts

Keywords

Synthesis, nickel oxide, nanoparticle, sol-gel, polyethylene glycol

from precursor molecules dissolved in water or alcohol and then converted into gels through heating and stirring with hydrolysis or alcoholysis. Then the resulting gel is dried by the appropriate method depending on the desired properties and application. The sol-gel method was chosen because it produces products with high purity without needing a lot of energy. Therefore, this review paper will discuss research on the traditional and green synthesis of nickel oxide nanoparticles using the sol-gel method with the precursor used, namely polyethylene glycol, as an agglomeration barrier, including possible improvements and weaknesses of this method

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Introduction. Recently, the science of nanotechnology has attracted public attention. Nanotechnology is considered one of the main areas supporting technological progress in the Industrial Revolution Era 4.0. Even though nanotechnology is still a relatively new in Indonesia, it can be said that this field makes a significant contribution, such as in the fields of energy, environment, medical, sensors and electronics. So, the potential is quite promising and the research area is quite broad. Basically, nanotechnology is the study of how to manipulate and create materials at the atomic and molecular level with particle sizes of 1–100 nm. This focus of interest in nanotechnology creates opportunities to develop applications in the catalytic field as desired [1]. To get nano-sized products, so we needed a synthesis process. Synthesis is an integration of two or more existing elements that produce a new result.

In chemistry, chemical synthesis is a process of forming a particular molecule from added "precursors". In recent years, the synthesis of nanoparticles has focused on metal oxides due to their large surface area and good adsorptive properties [2]. Several metals, such as Ag, Co, Ni, and Pd have been studied in the form of nanoparticles, with unique properties that support their use in different applications within each element. However, Nickel nanoparticles are of great interest and are applied as catalysts, both photo-catalysts and electro-catalysts, heat exchangers and biosensors. Nickel nanoparticles are of interest because of their properties, namely high reactivity, good biocompatibility, abundance of concentrates, bacterial resistance, anti-inflammatory activity, cost-effectiveness, and environmental compatibility. Nickel is a transition metal element useful in the chemical industry. This element has several important characteristics in its application, such as resistance to corrosion and heat, high impact strength and ductility, relatively low thermal and electrical conductivity, and the ability to form alloys with other metal elements. In industry, nickel is widely used for battery applications, catalysts, nonferrous alloys and super-alloys, electrical coatings, and the production of stainless steel and alloy steel [3].

Overview of method for the synthesis of nickel nanoparticles. Nano-particle synthesis can be carried out by two methods, namely top-down and bottom-up methods [3]. The top-down approach starts with large materials and tries to break them down into nanomaterials through physical methods. The top-down method consists of mechanical milling, nanolithography, laser ablation, sputtering and thermal decomposition. In the mechanical milling mechanism, the material is crushed to a powder and followed by fine-grained particles up to tens of nanometers in size. Furthermore, the refined powder is synthesized to obtain the final product. While the bottom-up method is the most developed method at this time. Because in this method, nanoparticles can be controlled chemically in the solution phase.

The bottom-up method consists of sol-gel, spinning, chemical vapor deposition (CVD), pyrolysis, and biosynthesis. The method synthesis nanoparticles using the bottom-up method has better stability than the top-down method. Bottom-up synthesis has several advantages over top-down approaches, namely having greater control over the size, shape and composition of the resulting nanostructures [4, 5]. The top-down method has a weakness, namely the imperfection of the surface structure. Conventional top-down methods can cause crystallographic damage to the pattern during processing and cause additional defects to appear during the etching step. The top-down method is not suitable for use in large-scale production because it creates internal stresses, in addition to surface defects and contamination [6]. The classification of nanoparticle method can be seen in Fig. 1.

Overview of sol-gel method. The sol-gel process is a chemical method used to synthesize a nanoparticle-sized solid material or small molecules. In the sol-gel method, the precursor used is usually a metal alkoxide, in which the precursor is dissolved in water or alcohol through a hydrolysis or alcoholysis step and then converted into a gel by heating and stirring. The gel produced from these stages is wet, so the gel is dried using the existing method according to the desired gel application. The sol-gel method is a conventional method for the synthesis of nanoparticles, because this method is more cost-effective than other methods and can control the chemical composition of the product at low reaction temperatures [7].

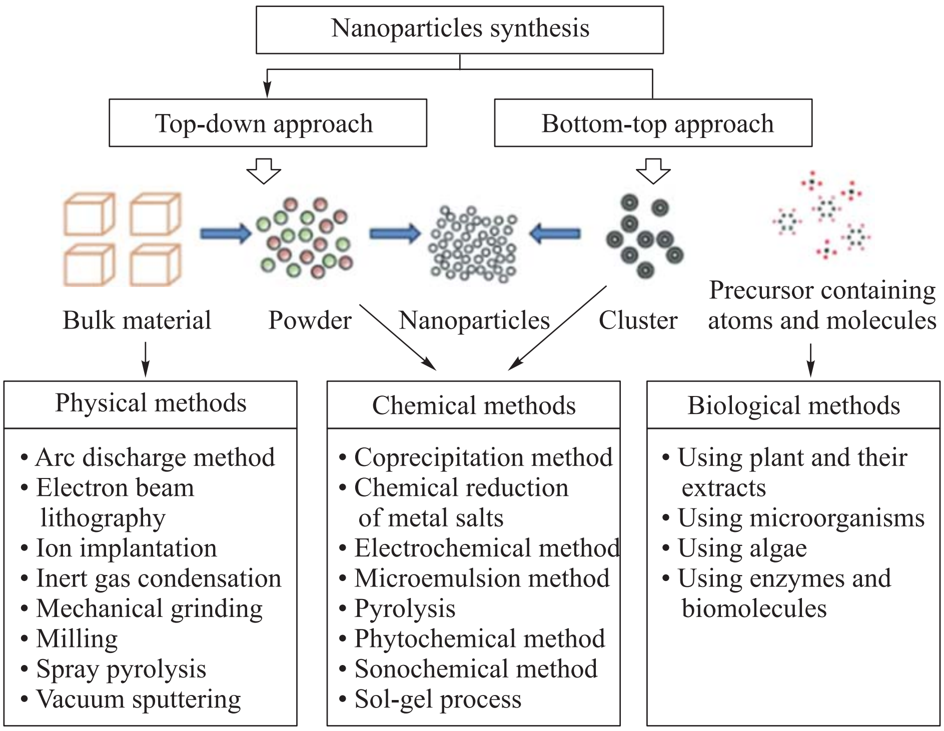


Fig. 1. Classification diagram of nanoparticle synthesis method [6]

The advantage of the sol-gel method is that it has higher popularity and industrial application compared to other synthesis methods, this is because the sol-gel method has unique properties and characteristics, where this method can produce nanoparticles with high quality and the same size as the industry scale [7]. Several studies have applied this sol-gel method in synthesizing nickel oxide nanoparticles. Based on Shamim's research, 2019 showed that nano-sized nickel oxide powder was successfully synthesized using a simple and low gypsum sol-gel method. The materials used are nickel(II) nitrate hexahydrate and 0.5 M sodium hydroxide. In the process, 3–4 grams of nickel nitrate hexahydrate is dissolved in 100 ml of deionized water and stirred. Next, the salt solution is titrated with 0.5 M NaOH. Then, a precipitate will form at pH = 11 which is marked in green. The precipitate is washed and dried to remove water content at 95 °C, then the dry precipitate is calcined at 550 °C for three hours in a furnace. The resulting product is characterized by X-ray diffraction (XRD), energy dispersive spectroscopy (EDX), and scanning electron microscopy (SEM). From Fig. 2, the resulting particle size of 45 nm indicates the formation of pure NiO NPs without impurities and the results obtained from the morphology show that nickel oxide nanoparticles can be applied to the fields of lithium-ion batteries, semiconductor materials and wastewater purification [2].

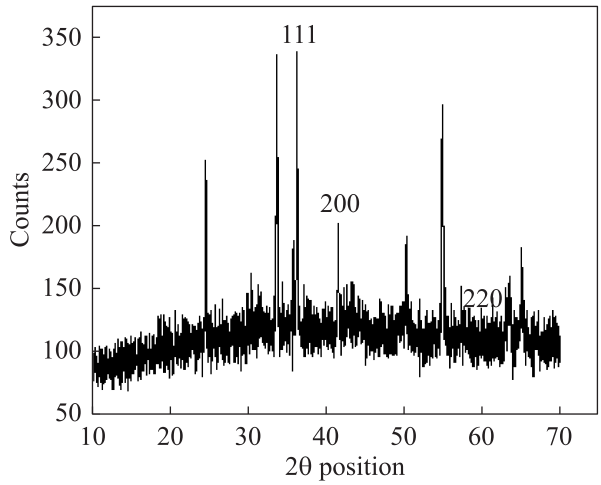


Fig. 2. XRD data of NiO nanoparticles [2]

Study [8] discusses the synthesis of NiO nanoparticles using the sol-gel method, where the precursors used are ammonium hydroxide and nickel nitrate. NiO nanoparticles were calcined at 400 and 1000 °C. Then the results were characterized using Fourier transform infrared spectroscopy (FT-IR), vibrating sample magnetometer (VSM), XRD, differential thermal analysis (TGA/DTA). The results obtained are in accordance with the XRD data that NiO has high purity and forms good crystals. The XRD data showed that the crystal size increased as the calcination temperature increased in Fig. 3. The crystal size obtained ranges from 12–20 nm when calcined at 400 and 1000 °C. Fourier transform infrared spectroscopy data shows the formation of NiO nanoparticles and their chemical composition.

Research [9] on the manufacture of NiO using the sol-gel process has been carried out using nickel acetate tetrahydrate as a precursor, methanol as a solvent and urea and NaOH as a precipitation agent. Nickel oxide nanopowder produced by drying at a temperature of 100–110 °C for 1 hour and dissolved by calcination at a temperature of ± 450 °C for ± 1.5 hours. The result obtained is powder or black powder. The XRD pattern shows that the NiO product formed is a cubic structure with a crystal size of about 50.53 nm. SEM micrographs of 1–10 μm and the crystal length of about 5–20 μm with very small sizes can be displayed as adsorbents and catalysts.

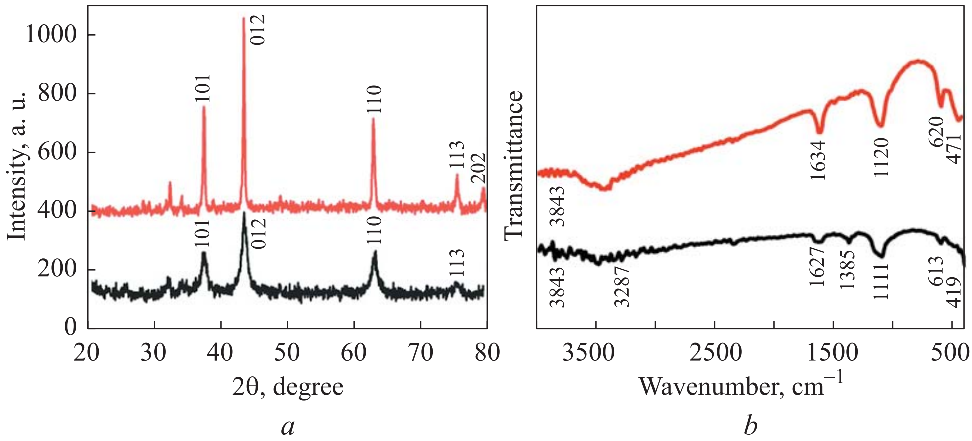
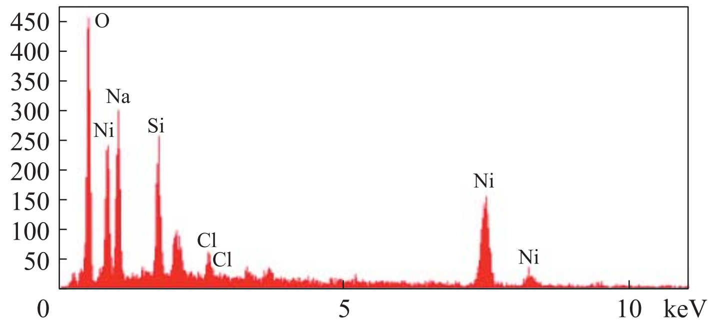


Fig. 3. XRD (*a*) and FT-IR (*b*) patterns of nickel oxide nanoparticles obtained at annealing temperatures of 400 °C (—), and 1000 °C (—) [8]

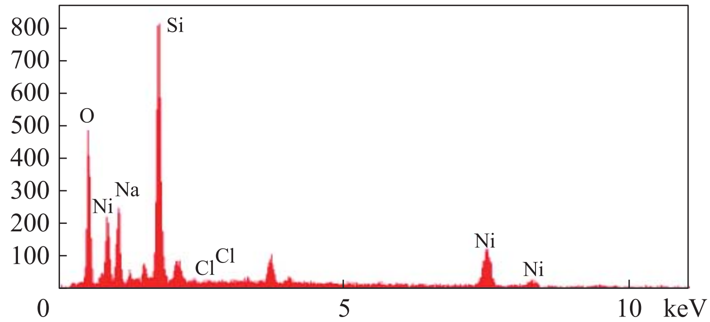
In another study [10] studied the magnetic and structural properties of NiO nanoparticles synthesized using the sol-gel method, which used two precursors in acidic and alkaline media deposited on a glass mat, namely nickel nitrate hexahydrate and nickel chloride hexahydrate. with the same concentration and optimal temperature. Judging from the X-ray diffraction patterns or XRD data in Fig. 4, the synthesized nickel nano-oxide powder has a sample structure in the form of a simple cubic with high crystallinity and the particle size is calculated using the Debye — Scherrer equation, for samples of nickel nitrate hexahydrate or alkaline medium, it has the smallest crystal size of 23 nm, and the number of nickel elements is more than the other samples. When viewed from its morphology, nickel oxide nanoparticles have a spherical shape, and the size of the nanoparticles ranges from 52–54 nm where the samples are arranged in a pattern on the glass mattress fibers. According to magnetometer analysis, only nickel nitrate samples in acidic media acquire ferromagnetic properties.

In another study [11] explaining the synthesis process using sol-gel namely NiO powder was synthesized with modifications of the precursors used, namely citric acid, nickel acetate and ethylene glycol. The pH was adjusted to 8 with the addition of 30 % ammonium hydroxide (NH_4OH), then the solution was evaporated at 90 °C for 4 hours with constant agitation, the process aims to support hydrolysis, polyesterification, esterification and gel formation



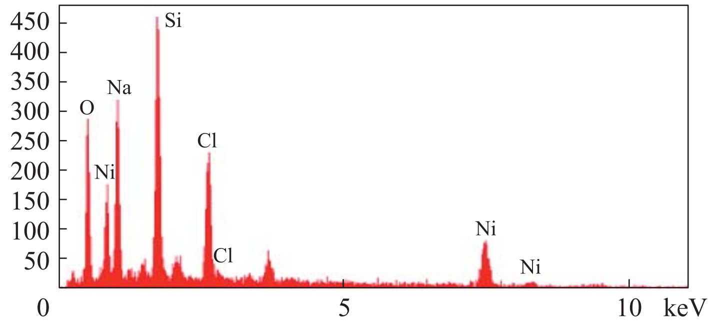
ELT	W%	A%
O	33.72	54.82
Na	17.59	19.90
Si	6.54	6.05
Cl	1.87	1.38
Ni	40.28	17.85

a



ELT	W%	A%
O	39.95	55.22
Na	12.48	13.34
Si	21.33	18.67
Cl	0.39	0.27
Ni	29.85	12.50

b



ELT	W%	A%
O	32.99	50.40
Na	18.53	19.70
Si	14.92	12.99
Cl	10.74	7.40
Ni	22.82	9.50

c

Fig. 4. EDX results related to deposited glass mat with sample A (*a*) with nickel nitrate in basic medium, sample B (*b*) with nickel nitrate in acidic medium, sample C (*c*) with nickel chloride in basic medium [10]

reactions. Furthermore, the resulting gel was heated for pre-calcination at 250 °C for 2 hours and for calcination at 800 °C for 1 hour. The results obtained through scanning electron microscopy are particles after pre-calcination measuring 50 m and after calcination measuring 10 m. Energy dispersive X-ray spectroscopy (EDS) data detects the presence of nickel in Fig. 5, oxygen, and carbon before calcination, while after calcination the composition only contains Ni and O. Furthermore, X-ray photoelectron spectroscopy (XPS) data detects a composition in nickel oxide close to stoichiometry, while the

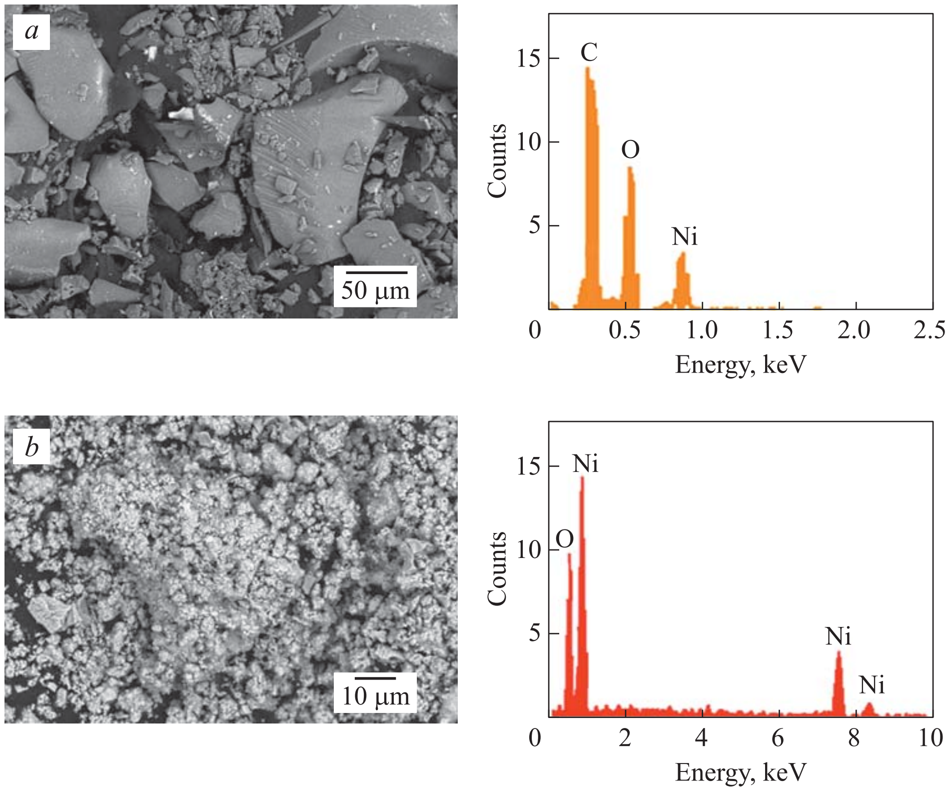


Fig. 5. SEM image and EDS spectrum of NiO_x annealed 250 °C (*a*), 800 °C (*b*) [11]

Raman and Fourier transform infrared spectra data only show the characteristics of the nickel oxide itself. The crystal structure of the particles that have been calcined is cubic in shape as seen from the XRD data. While the characterization tests carried out by Mateos' research, namely XRD, EDS, FT-IR XPS, and Raman, showed that the synthesized nickel oxide had high purity, the method carried out was low cost and the method was very effective.

Research [12] describes the synthesis of NiO nanoparticles by sol-gel method and for application to dye-sensitized solar cells. Nickel oxide nanoparticles were synthesized with the participation of nickel(II) nitrate hexahydrate and citric acid. Then the results obtained are uniform nickel oxide nanoparticles with a spherical shape with nanoparticle diameters not exceeding 25 nm from XRD and TEM analysis. The resulting nanoparticles were successfully used as semiconductor coatings in dye-sensitized solar cells, according Fig. 6. The light harvesting efficiency of the electrodes across the range studied was more than 90 %.

There are other studies [13] using the sol-gel method also in the synthesis process, but the difference is that this research was carried out with auto combustion, where $Ag^{+}/Mn^{2+}/Cr^{3+}$ is doped with nano-sized spinel ferrite Ni–Cu–Zn, then the chemical composition is

$$Ni_{0.4}Cu_{0.3}Zn_{0.3}Ag_{0.4x}Mn_{0.3x}Cr_{0.3x}Fe_{2x}O_4 \ (x = 0, 0.05, 0.10, 0.15).$$

X-ray diffractometry and field emitting scanning electron microscopy (FESEM) identified the formation of a nano-sized single-phase spinel ferrite structure. For lower dopant concentrations (x = 0, 0.05, 0.10) the crystal size decreased from 19 to 12 nm, while for high concentrations (x = 0.15), the crystal size increased to 18 nm. From the results obtained the crystal size is less than 50 nm, this indicates that the results of the synthesis process of Ni–Cu–Zn ferrite can be used to obtain the ratio of noise to signal in high-density recording media. FT-IR characterization detected the formation of characteristic absorption peaks around 418.1–434.9 cm^{-1} and 571.2–592.5 cm^{-1}. Meanwhile, magnetic characteristics were analyzed according to structural and morphological properties. The results showed that increasing the concentration of dopants, Ms and nB could decrease from 66 emu/g to 54 emu/g and 2.82μ to 2.33μ. In addition, the high Ms value and lower Hc value results in soft magnetic particle properties of Ni–Cu–Zn, making it suitable for multi-layer chip inductor (MLCI) applications.

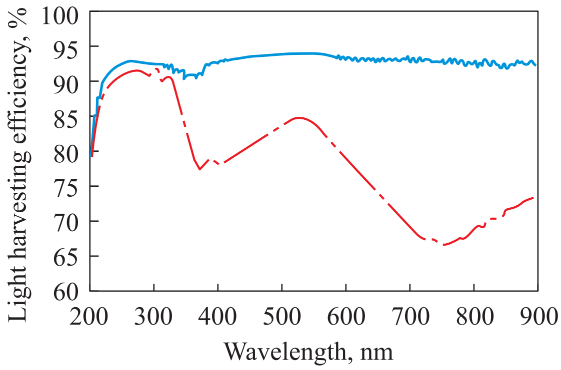


Fig. 6. Light absorption efficiency spectra for individual layers NiO + N_3 (——) and TiO_2 + N_3 (– · –) of the photo anode of dye sensitized solar cell [12]

There are also other studies [14] that synthesize nickel oxide nanoparticles (NiO NPs) via photo irradiation as a new method. The method used is considered simple and cost-effective. In the process, potassium nickel sulfate hexahydrate $K_2\ (Ni(SO_4)_2 \cdot 6H_2O)$, HCL, deionized water are mixed to form a solution, a chemical used without any purification. Nickel oxide nano-particles were prepared using the photo-irradiation method, in which the irradiation cell was used as a source of nickel oxide nanoparticles. The results of the study in the form of particle size and morphology were analyzed by TEM, SEM, AFM characterization and crystallinity or XRD and Gaussmeter analysis to determine the strength of the sample's magnetic field. The results of the XRD data show that the nickel oxide nanoparticles have high crystallinity, then the size of the nickel oxide particles produced is 12 nm according Fig. 7. Furthermore, the value of the inhibition zone shows that the nanoparticles have an effect on different bacteria. The results of this study are considered as a new synthesis method of NiO nanoparticles to obtain antimicrobial agents against bacteria.

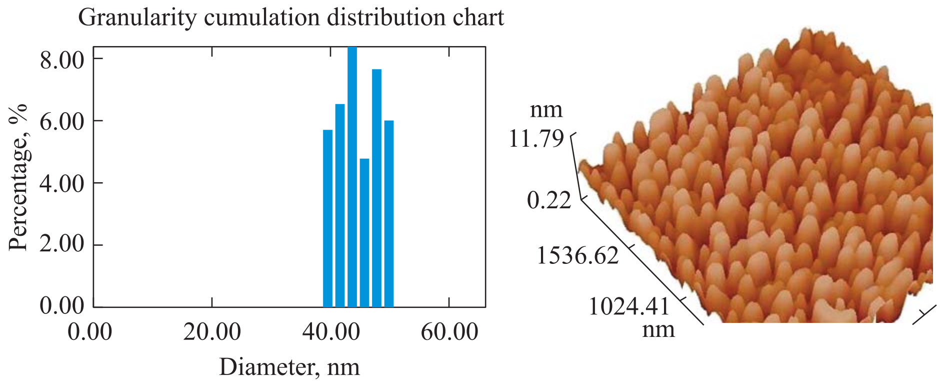


Fig. 7. AFM microscopy diameter distribution of Ni–NPs [14]

Research [15] describes the synthesis of NiO nanoparticles using the sol-gel method with variations of three different pH values, namely pH = 1, 7 and 11, the calcination temperature is carried out at 450 °C. The effect of different pH values on the physical properties of NiO nanoparticles is seen from PSA, FESEM and XRD characterization. At pH = 1, the cubic structure of NiO nanoparticles was obtained without secondary phase and for pH = 7 and pH = 11, the secondary phase peaked. Morphological observations of pH = 7 and pH = 11 formed more agglomerates compared to pH = 1. The average diameter of NiO nanoparticles with pH = 1, 7 and 11 ranged from 19–26 nm, 21–28 nm, and 24–30 nm, respectively. The synthesized NiO powder with pH = 1 was then used to make NiO NPs–$BaCe_{0.54}Zr_{0.36}Y_{0.1}O_{2.95}$ (BCZY) composite anodes. Unfortunately, the NiO NPs–$BaCeO_3$–$BaZrO_3$ composite obtained was not BCZY and was composed by agglomerates with sizes in the range of 70–300 nm.

In study [16], the synthesis of NiO nanoparticles by sol-gel method was carried out using $Ni(NO_3)_2$–$6H_2O$ served as inorganic precursor and poly(alkylene oxide) block copolymer served as surfactant. Then, the results of the effect of variations in calcination temperature and H_2O concentration on the formation of NiO nanoparticles were obtained. TGA, XRD, SEM, TEM, and N_2 adsorption-desorption isotherms were used to characterize the microstructure and specific surface area of the samples. Following Fig. 8, the results showed that the nanoparticles formed at 923 K were the purest, while those formed at 823 K had the lowest purity. In addition, this study shows that the specific surface area of NiO nanoparticles increases with the increase in the amount of water added.

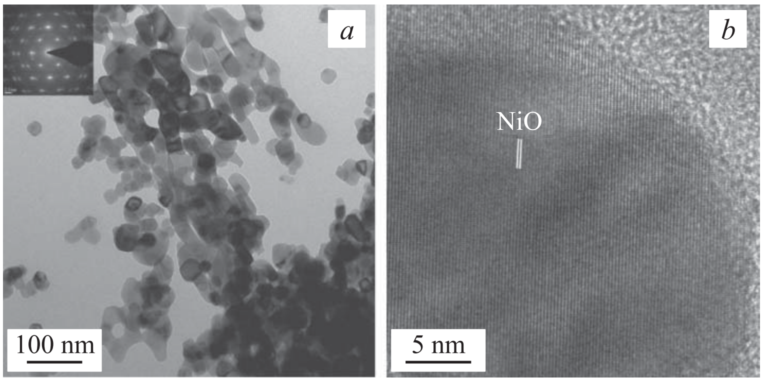


Fig. 8. The bright field and dark field of the TEM images (*a*), and HRTEM of NiO prepared with 10 mass % H_2O and calcined at 723 K for 3 h [16] (*b*)

Another study [17] discussing the synthesis of nickel nanoparticles using the sol-gel method. In contrast to previous research, this study used polyethylene glycol (PEG) as a barrier to agglomeration. This study describes the synthesis of sol-gel with a precursor in the form of PEG which is used as a hole selective layer. Furthermore, a high temperature post-annealing process was carried out on the NiO layer which is useful for obtaining high crystallinity according the data in Fig. 9. The NiO layer has a uniform and smooth surface for charge

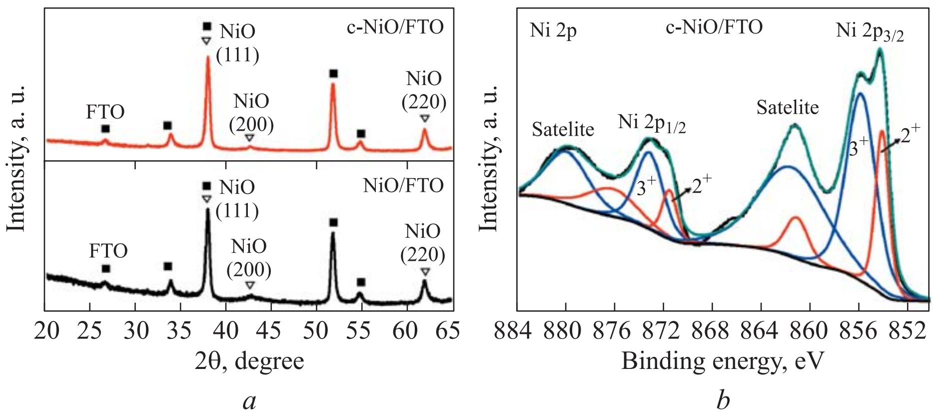


Fig. 9. XRD pattern of nickel oxide coating on FTO glass without using PEG (*a*) and using PEG (*b*) [17]

transport in organic solar cells. In this study, the difference was seen between the nickel oxide (NiO) coating on fluorine-doped tin oxide (FTO) glass using PEG and those without.

In research [18], he explained the synthesis of NiO nanoparticles using the sol-gel method in an alkaline state or pH = 11, with the precursors used, namely PEG and Triton® X-100, then the calcination temperature was around 450 °C. The results of structure, particle size, and particle morphology of NiO found that NiO has a cubic structure without any impurities. Then the morphology of NiO shows the average diameter size is around 32.9 nm. Kooti's research shows that NiO_2 and NiO nanoparticles can be easily produced through surfactant-assisted chemical reactions. The surfactant used is Triton® X-100 GR which is incorporated into the reaction mixture of $Ni(NO_3)_2{-}6H_2O$ and ClO^-. Then, the resulting NiO_2 nanoparticles can be converted into NiO nanoparticles by simply washing with acetone. NiO_2 and NiO nanoparticles were obtained with high purity. After the washing process, no calcination process is required as is usually applied in other methods [19]. It describes a synthesis method that uses Triton® X-100 surfactant as a non-ionic surfactant to synthesize ZnO nanoparticles and shows that Triton® X-100 can improve gel homogeneity and optimize the formation of pore structures in sol-gel materials [20, 21].

Research [22] explains that large-scale NiO synthesis can be carried out using PEG as a fuel through combustion reactions with nickel oxalate as a precursor. Dried nickel oxalate is ground to obtain powder and mixed with PEG in a ratio of 1:5. Characterization carried out after NiO has been synthesized in the form of crystal structure, morphology, and bonding through XRD, SEM, and FT-IR. The thermal behavior of the synthesized NiO was seen using TGA and DSC. The super paramagnetic behavior of the synthesized was studied by magnetic hysteresis.

Research [23] shows that the synthesis of ZnO using the sonication method (low intensity) with dihydrate zinc acetate precursor for 2 and 4 hours at room temperature produced XRD data patterns with pure ZnO phase. The results of the morphology of ZnO particles that were sonicated for 120 min were fine when compared to the sonication time of 4 hours, with a needle-shaped shape with a diameter of 80–100 nm. Another study said that the length of sonication time affected the average molecular mass (Mv), namely a decrease in Mv at 120 min of sonication [24]. Similar to research [25] which showed that the optimum time to produce high purity silica sand was 120 min with an oxalic acid concentration of 1.5 g/L.

Sonication is a treatment method by utilizing ultrasonic waves in which the process is an ultrasonic power generator in the form of an electrical signal which is then converted into physical vibrations, causing a cavitation effect on the solution which causes the solution molecules to break apart. This sonication method has the advantage of producing a very small particle size, preventing creaming or sedimentation during the storage period, and producing a large surface area with a transparent color so that it can accelerate the penetration of the active ingredient and facilitate dispersion [26]. The sonication method aims to prevent oxidation of metal ions which causes the formation of amorphous particles [27]. The duration of the sonication process affects the morphological properties of a particle. The results show that the longer the sonication time, the particle size tends to be more homogeneous and smaller which will eventually produce a stable nanoparticle size with less agglomeration. This is because the shock waves that occur in the sonication method can separate particle agglomeration so that perfect dispersion occurs with the addition of surfactant as a stabilizer [25].

Ultrasonic treatment has several functions in the synthesis process of the sol-gel method, including: improving the homogeneity of the mixture, increasing the reaction speed, producing cavitation effects to break large particles into smaller particles. Increase the density and strength of the material due to the cavitation effect which can cause high pressure and temperature which can accelerate the polymerization process and increase the binding of material particles [28, 29].

Green synthesis. There is an environmentally friendly nanoparticle synthesis method, usually called green synthesis. Green synthesis is a method for synthesizing metal nanoparticles using plants, microbes, algae, bacteria, fungi and yeast. This green synthesis method has advantages besides being environmentally friendly, namely the process is easy to do, and the resulting product has low toxicity. The green synthesis method has three main factors, namely a harmless stabilizing agent, a good reducing agent, and the use of environmentally friendly solvents. Plants produce nanoparticles that are more stable and biocompatible than those made by other methods [30].

One of the studies that used the green synthesis method was Pandian [31]. The study explained that nickel nanoparticles were synthesized by the green synthesis method using *Ocimum Sanctum* leaf extract. NiG serves as an adsorbent to remove dyes such as crystal eosin Y (EY), orange II (OR), anionic pollutants nitrate (NO_3^-), violet (CV), sulfate (SO_4^{2-}) from aqueous solutions. Variables of NiGs were examined in the form of pollutant concentration, contact time, different pH, initial dye and dose of NiGs. In the Pandian study, the leaves of *O. sanctum* were washed thoroughly with distilled water and then dried for 3 days at 25 ± 1 °C, then mechanically milled and 15 mesh sieved, then the powder was washed with 2 % HCl solution. The leaf extract used was 1 gram of leaf powder in 50 ml of double distilled water. Then, shaking was done for 2 hours and the precipitate was removed by filtration. Filtrate. The filtrate is used as an extract for the synthesis of NiGs. NiG was synthesized by vigorous stirring from 10 ml of *Ocimum sanctum* leaf extract added with 1 mmol · L–1 $Ni(NO_3)_2$ solution, for 3 hours at 60 °C. Furthermore, the solution was dried for 24 hours, and nanoparticle powder was obtained [31].

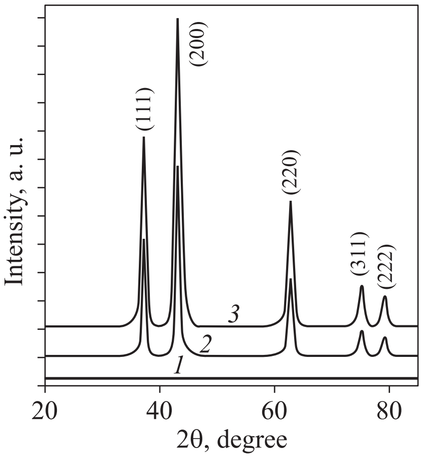


Fig. 10. XRD pattern of a precursor (*1*), NiO nanoparticles calcined at 400 °C (*2*) and NiO nanoparticles calcined at 500 °C (*3*) [32]

There is research explaining that nickel oxide nanoparticles have been synthesized by adding agarose polysaccharide in a sol-gel method. Characterizations such as structure, morphology, optical and magnetic properties of the products were examined by X-ray diffraction, transmission electron microscopy, UV-visible spectrophotometer, and superconducting quantum interference device magnetometer. TEM images show the nickel oxide nanoparticles have a spherical shape of about 3 nm in size. Magnetic measurement results show that NiO nanoparticles are ferromagnetic at 4.2 K and superparamagnetic at 300 K in Fig. 10 [32].

There are other studies [33] that discuss the synthesis of nickel nanoparticles but using other methods, namely green and convenience as a magnetic mirror with antibacterial activity. The simple, green, and efficient method uses antibacterial NMMNP, namely *E. coli* and *S. aureus* bacteria using the diffusion method. Both bacteria were confirmed by inactive colony method in the presence of NMMNP. The NMMNPs results showed a higher inhibition zone than *E. coli* bacteria. The synthesized mirror has less than 7 % transmittance in applicable UV light. The NMMNP results are more reflective than ordinary glass. Ni-NP samples can be used as an alternative mirror with toxic materials such as Hg because Ni-NP has low toxicity. In addition, NMMNP also has magnetic properties, therefore NMMMP can be applied as a glass coating as a highlight mirror feature.

Other studies [34] investigated the process of synthesizing nickel oxide nanoparticles (NiO-NPs) using the biosynthetic or green synthesis method using

Cydonia oblonga extract both from phase, the method can be seen in Fig. 11, morphological and shape studies using various techniques. The results of the evaluation of the biocompatibility of these nanoparticles aim to obtain safety data on the L929 cell line of these particles which results are non-cytotoxic. In addition, the XRD results showed a face-centered cubic (FCC) construction of NiO with a space group (Fm3m), then FESEM and EDAX observed the morphology and analyzed the elements of the NiO-NPs. The result is that NiO-NPs are superparamagnetic which can act as a photocatalyst (4.3–5.2 eV).

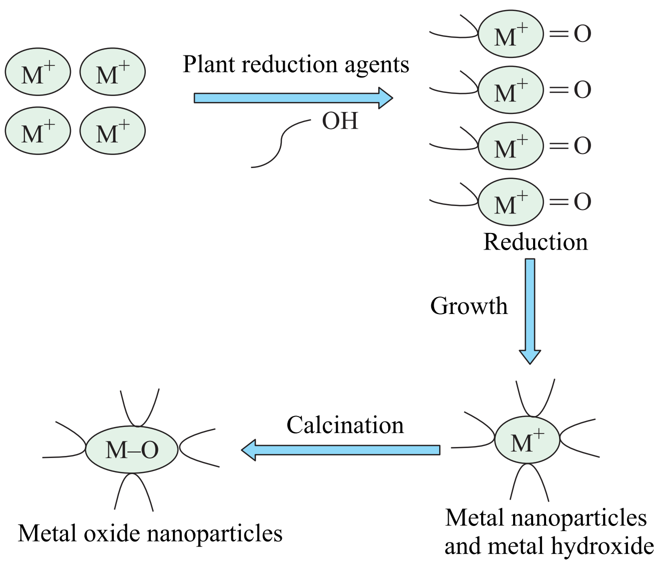


Fig. 11. Mechanism of plant mediated synthesis of metal oxide nanoparticles [34]

Research describes the green synthesis of nickel oxide (NiO) nanoparticles using *Moringa oleifera* plant extract because it is more cost-effective and environmentally friendly. X-ray diffraction, FT-IR, high-resolution transmission electron microscopy (HRTEM), energy-dispersive X-ray analysis (EDX), and photoluminescence (PL) spectroscopy were used to characterize the nanoparticles. The synthesized nickel oxide nanoparticles were single crystals with a cubic phase and two intense photoluminescent emissions at 305.46 and 410 nm. *In vitro* cytotoxicity and cell viability of human HT-29 cancer cells and antibacterial studies against various bacterial strains were performed with various concentrations of nickel oxide nanoparticles prepared from *Moringa oleifera* plant extract. MTT assay measurements on cell viability and morphology studies proved that the synthesized nickel oxide nanoparticles have cytotoxic activity against human cancer cells and various zones of inhibition, indicating that they are effective against various Gram-positive and Gram-negative pathogenic bacteria [35] or can also be synthesized using *Sageretia thea* (Osbeck) leaf extract [36]. In addition, *Areca catechu* leaf extract can also be used for the prevention of diabetes and cancer [37].

In research [38], nickel nanoparticles were synthesized by the green synthesis method using *Fumaria officinalis* water extract. *Fumaria officinalis* leaves were characterized by FESEM, EDX, UV-Vis, FT-IR, and XRD. The resulting nickel nanoparticles are spherical in shape with a particle size of 16.85–49.04 nm according Fig. 12. Then, NiNPs–*Fumaria officinalis* were tested for antioxidants, IC50, and BHT against DPPH free radicals yielding 253, 145, and 107 mg/mL, respectively. In addition, NiNPs–*Fumaria officinalis* was tested by MTT for 48 hours regarding its cytotoxicity and anti-cancer properties in the human ovary. IC50 test results from NiNPs–*Fumaria officinalis* were 375, 225, 246, and 279 mg/mL against the cell lines PA1, Caov-3, SW-626, and SK-OV-3, respectively. The results obtained are that nickel nanoparticles containing aqueous extract of *Fumaria officinalis* leaves can be used as a new chemotherapy drug formula to treat several types of ovarian cancer in humans.

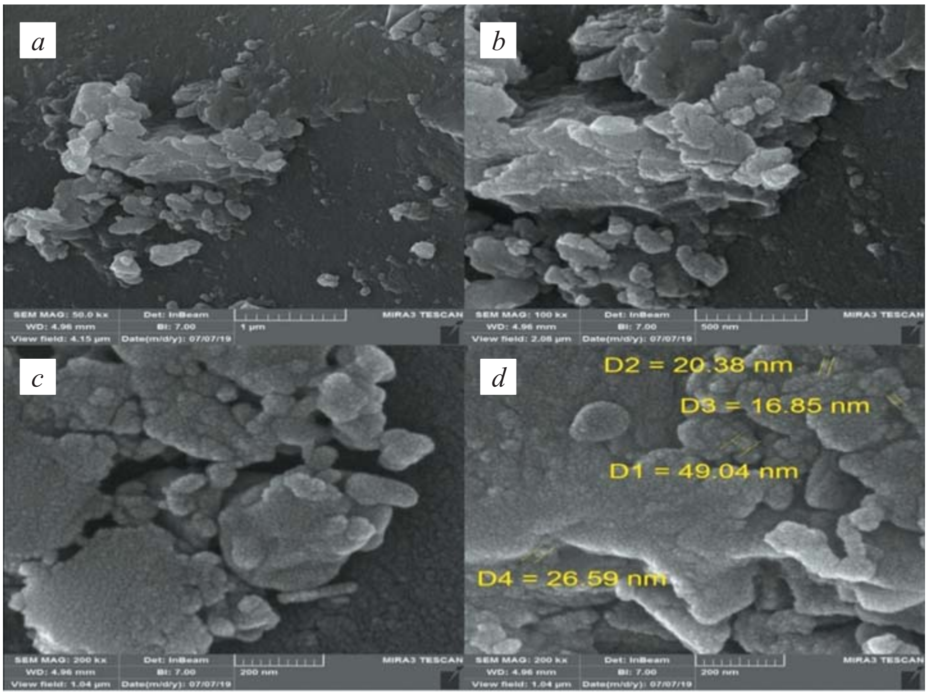


Fig. 12. FE-SEM images of NiNPs–*Fumaria officinalis* [38]

Pure single-phase NiO nanoparticles were successfully synthesized using the natural extract of *Agatosma Betulina* as an effective chelating chemical. The process was carried out at 500 °C for 2 hours under normal air conditions, resulting in highly crystallized single-phase cubic NiO nanoparticles. Stencils fabricated from *n*-type/*p*-Si NiO nanoparticle junctions showed adequate photovoltaic & photodetection response especially in the red region of the spectrum. Follow-up studies will examine the physical and chemical mechanisms involved in the formation of NiO nanoparticles during the interaction between nickel precursors and active compounds of *Agathosma Betulina* natural extracts [36]. There is another study [39] that made nanoparticles from *Salvia hispanica L.* (chia) seed extract as a capping agent. The results showed that the nanoparticles were spherical and had a size of about 30 nm.

In another study [40], using *Azadirachta indica* extract to synthesize MnO_2NPs by green synthesis, and Mn_2O_3NPs were successively synthesized by thermal decomposition. In this investigation, Mn_2O_3NPs were used as heterogeneous catalysts for the removal of organic contaminants, particularly RhB. There are other uses of extracts that can be synthesized through green synthesis of NiO nanoparticles, such as *Nigella sativa* [41], *Berberis* [42], *Gymnema sylvestre* [43], tea plant extract [44], and *Rhamnus virgata* [45]. These extracts are environmentally friendly and offer a sustainable alternative to traditional chemical synthesis.

Recent development recent development about sol-gel method for synthesis of nickel nanoparticle. Based on the literature survey, the sol-gel method is still the best method for the synthesis of nanoparticles. This is because the sol-gel method can produce materials with higher purity and strength than other methods. In addition, the sol-gel method has several advantages such as inexpensive equipment, homogeneous products and easy processing. The sol-gel method will produce a small particle size and uniform particle distribution with high homogeneity [46]. The advantage of NiO synthesis using the sol-gel method is that it can produce optimum particle size and maintain phase purity [47]. The disadvantages of the sol-gel method include the high cost of precursors, the use of organic solvents that can be harmful to the environment. In addition, it can be time-consuming and tedious due to its multi-step nature. In addition, the sol-gel method may not be suitable for all materials and applications, as it requires careful control of factors such as pH, temperature, and concentration [48, 49].

Among the many types of magnetic materials, the most widely used in current technology are metals and metal oxides. In the process of synthesizing nanoparticles, from some literature, nickel oxide is used as the metal oxide to be synthesized. Applications of nickel oxide nanoparticles can be found in various fields, including in semiconductors, gas sensors, magnetic materials, supercapacitors, battery cathodes, catalysts, pharmaceuticals, and solid oxide fuel cell anodes.

In addition, these NiO nanoparticles can also be applied as photocatalyst semiconductors in managing organic dye waste, one of which is Methylene Blue [50]. Non-stoichiometric nickel oxide has defects that can be used in semiconductors with *p*-rip forms and is found in applications namely hydrogen gas sensors. At temperatures above 523 K, it was found that NiO has an FCC structure with the NaCl type [9].

Conclusion. The sol-gel process is a wet chemical method in the process of synthesizing a material in nanostructures, especially in metal oxide nanoparticles. In this method, two important phases are applied, consisting of a sol and a gel. The working principle of this sol gel method is the formation of an initial compound (precursor) consisting of organic salts or organic metal compounds, then the polymerization of the solution occurs and then to the drying and calcination process to remove organic compounds and form inorganic materials in the form of oxides. Nickel oxide nanoparticles were synthesized using this method, because this method can produce high purity and strength of nickel. The results of the synthesis of NiO nanoparticles can be applied in various fields, including supercapacitors, gas sensors, semiconductors, magnetic materials, battery cathodes, catalysts, pharmaceuticals, solid oxide cell anodes and so on, according the green one or traditional.

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