

UNIVERSITI PUTRA MALAYSIA

INFLUENCE OF CALCINATION TEMPERATURE ON NICKEL OXIDE NANOPARTICLES SYNTHESIZED BY THERMAL TREATMENT

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By MANAL AHMED HASHEM

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Master of Science

June 2015

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Abstract of thesis presented to the senate of Universiti Putra Malaysia in fulfilment of the requirement for the Degree of Master of Science

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June 2015

Chair : Professor Elias Saion, PhD Faculty : Faculty of Science

Recently, semiconductor nanoparticles have received immense interests due to their peculiar and outstanding physical and chemical properties which are different from their bulk counterparts. Nanostructures nickel and its oxides have potential applications in battery electrodes, catalysts, piezoresistors, thermistors, gas sensors, stain glass, and ceramic additives. Nickel oxide (NiO) nanoparticles have a wide direct band gap (3.56 eV) is a stable material and exhibits p-type semiconducting behavior. They also can be superparamagnetic and superanti-feromagnetic in their properties.

Several methods have been used and developed for synthesizing crystalline NiO nanoparticles in recent years, including chemical reaction, electrodeposition, sputtering, pulsed laser deposition, oxidation of nickel at high temperatures, solution growth techniques, spray pyrolysis, and sol–gel route. In many of these methods, the main objectives are to produce controlled nanoscale materials, high purity, low cost, and environmentally friendly for particular technological applications. However, most of these synthesis methods have some drawbacks to meet these objectives due to complicated procedures involved, longer reaction times, high reaction temperatures, and toxic chemical precursors used.

In the present work, crystalline nickel oxide (NiO) nanoparticles have been synthesized using a simple thermal treatment method from a well-mixed solution containing only nickel nitrate as a metal precursor, polyvinyl pyrrolidone as a capping agent, and deionized water as a solvent before proceeded by drying at 80 °C for 24 h, grounding, and calcination at different temperatures range from 500 to 800 °C. The morphological, structural, optical and magnetic properties of the obtained nanoparticles were studied using various techniques. The surface electron microscopy (SEM) images showed that surface morphology of the samples consists of monocrystalline grains with almost uniform shape. The X-ray diffraction (XRD) and Fourier Transform

Infrared (FTIR) spectra exhibited that the samples were amorphous at room temperature but transformed into a crystalline structure during calcination. The XRD spectra showed that the average particle size and the degree of crystallinity increased with increasing calcination temperature. The average particle sizes from the transmission electron microscopy (TEM) images were about 15 and 35 nm at calcination temperatures of 500 and 800 °C respectively. The elemental composition of the samples was determined by energy dispersed X-ray spectroscopy (EDX) which confirmed the presence of Ni and O in the final products. The optical properties were determined by UV-Vis reflection spectrophotometer and showed a decrease in the band gap from 3.60 to 3.51 eV with increase of calcination temperature from 500 to 800 °C. The magnetic properties were also investigated by electron spin resonance (ESR) spectroscopy, which confirmed the presence of unpaired electrons. The resonant magnetic field decreased from 296.4 to 289.7 Oe and the g-factor increased from 2.28167 to 2.29367 with increase of calcination temperature from 500 to 800 °C.

Abstrak tesis yang dikemukakan kepada senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

PENGARUH SUHU KALSINASI KE ATAS NANOPARTIKEL NIKEL OKSIDA DISEDIAKAN DENGAN KAEDAH RAWATAN TERMA

Oleh

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Jun 2015

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Baru-baru ini, nanopartikel semikonduktor telah menerima tumpuan yang besar kerana sifat fizikal dan kimia mereka yang luar biasa berbeza daripada keadaan pukal rakan-rakan mereka. Nanostruktur nikel oksida adalah nanobahan yang mempunyai potensi kegunaan dalam elektrod bateri, pemangkin, piezoresistors, termistor, sensor gas, kaca noda, dan bahan tambahan seramik. Nanopartikel nikel oksida (NiO) yang mempunyai jurang tenaga yang luas (3.56 eV) adalah bahan stabil dan mempamerkan tingkah laku semikonduktor jenis-p. Mereka juga boleh bersifat superparamagnetik dan superanti-feromagnetik.

Beberapa kaedah telah digunakan dan dibangunkan untuk mensintesis kristal nanopartikel NiO pada tahun-tahun kebelakangan ini, termasuk tindak balas kimia, pengenapan, pemercikan, pemendapan laser berdenyut, pengoksidaan nikel pada suhu yang tinggi, teknik pertumbuhan penyelesaian, semburan pirolisis, dan laluan sol-gel. Dalam banyak kaedah-kaedah ini, objektif utama adalah untuk menghasilkan bahan-bahan bersaiz nano terkawal, ketulenan yang tinggi, kos penyedian rendah, dan mesra alam untuk aplikasi teknologi tertentu. Walau bagaimanapun, kebanyakan daripada kaedah sintesis mempunyai beberapa kelemahan untuk memenuhi objektif tersebut yang disebabkan oleh prosedur rumit yang terlibat, masa tindak balas lebih lama, suhu tindak balas yang tinggi, dan prekursor kimia toksik yang digunakan.

Dalam kajian ini, nanopartikel kristal oksida nikel (NiO) telah disintesis dengan menggunakan kaedah rawatan terma yang mudah dari satu larutan campuran yang mengandungi hanya nikel nitrat sebagai pelopor logam, polyvinyl pyrrolidone sebagai ejen menetapkan penyalut, dan air ternyahion sebagai pelarut sebelum diteruskan oleh pengeringan pada 80 °C selama 24 jam, dikisar, dan pengkalsinan pada suhu yang berbeza antara 500-800 °C. Ciri-ciri morfologi, struktur, optik dan magnet nanopartikel diperolehi telah dikaji dengan menggunakan pelbagai teknik. Mikroskopi elektron permukaan (SEM) menunjukkan imej yang morfologi permukaan sampel terdiri daripada bijirin



monohabluran dengan bentuk hampir seragam. Spektrum pembelauan sinar-X (XRD) dan speltrum inframerah Fourier Transform (FTIR) dipamerkan bahawa sampel amorfus pada suhu bilik tetapi berubah menjadi struktur kristal semasa proses kalsinan. Spektrum XRD menunjukkan bahawa saiz zarah purata dan darjah penghabluran meningkat dengan peningkatan suhu pengkalsinan. Saiz zarah purata dari mikroskop elektron transmisi (TEM) imej kira-kira 15 dan 35 nm pada suhu pengkalsinan 500 dan 800 °C masing-masing. Komposisi unsur sampel telah ditentukan oleh spektroskopi tenaga tersebar X-ray (EDX) yang mengesahkan kehadiran Ni dan O dalam produk akhir. Ciri-ciri optik ditentukan oleh spektrofotometer refleksi ultraunggu-tampah dan menunjukkan penurunan dalam jurang jalur tenaga 3.60-3.51 eV dengan peningkatan suhu pengkalsinan 500-800 °C. Sifat-sifat magnet juga disiasat oleh spektroskopi resonan spin elektron (ESR), yang mengesahkan kehadiran elektron tidak berpasangan. Medan magnet salunan menurun 296.4-289.7 Oe dan faktor-g meningkat 2.28167-2.29367 dengan peningkatan suhu pengkalsinan 500-800 °C.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF SYMBOLS AND ABBREVIATIONS

н	Applied field
H _r	Resonant magnetic field
Pa	Pascal
Φ	Magnetic flux
A/m	Ampere per meter
μ_{B}	Bohr magnetron
Fd3m	Space group in cubic structure
f	Frequency
Х	Susceptibility
ΔH_{pp}	Peak-to-peak line width
H_{hrf}	Hyperfine spine interaction Hamiltonian
g-factor	Gromagnetic ratio
h	plank constant
М	Magnetization
eV	Electron volt
Ean	Magnetocrystalline anisotropy energy
Ni	Nickel
°C	Degree Celsius
а	Lattice parameter
ESR	Electron spin resonance
TEM	Transmission electron microscopy
FTIR	Fourier transform infrared
SEM	Scanning Electron Microscopy

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- XRD X-ray diffraction
- TGA Thermo gravimetric analysis
- PVP Poly (vinyl pyrrolidone)
- UV-vis Ultraviolet visible
- KM Kubelka–Munk

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CHAPTER 1

INTRODUCTION

1.1 Introduction of Nanoscience and Nanotechnology

After the emergence of nanoscience and nanotechnology, new dimensions of basic sciences were created and new branches of technology developed (Hornyak et al., 2011). Nanotechnology can be best defined as a design, fabrication and implementation of nanostructures or nanomaterials. Apparently, nanotechnology deals with miniature-sized materials and small structures between 1 and 100 nm. One nanometer is approximately as long as 5 silicon atoms aligned (Patil et al., 2008). Going down in size such metal particles, containing 1000 atoms that can be used to produce advanced materials. Their characteristics are considerably different from the bulk material, and in result assembling them to macroscopic chunks creates materials with novel behavior. 1 nm is about the smallest structures used in nanotechnology (Binns, 2010).

There are two approaches to synthesize nanomaterials and fabricate nanostructure: bottom-up and top-down approaches. Milling or attrition is considered a common top-down method to produce nanoparticles, whereas a good example of the bottom-up approach is the colloidal dispersion which contributes to the nanoparticle synthesis. Both approaches play crucial roles in modern industry and nanotechnology. However, both approaches have some advantages and disadvantages (Chan et al., 2011).

The imperfection of the surface structure is found as the biggest problem of the top-down approach. Obviously, the traditional top-down systems like lithographer might lead to major crystals damages to the processed patterns and even further defects might emerge during intaglio step. For instance, nanowire which is synthetic by lithographer is not smooth at all and they might have lots of structural defects and impurities on surface. These imperfections can affect physical features, and the surface chemistry of nanomaterials and nanostructures. Notwithstanding of any defects like the surface imperfections of the top-down approach, they can represent an important roles in the nanomaterials and nanostructure synthesis (Mijatovic et al., 2005).

In nanotechnology literature, the bottom-up approach is most often emphasized despite the fact that there is nothing new in bottom-up approach for the synthesis of nanomaterials. In fact, bottom-up approach represents the material build-up of atom by atom, jot by jot, or mass by mass. The best models for this built-up from bottom is the production of nitrate and salt in chemical industry. In most cases, there is not much difference between the physical characteristics of materials irrespective of their synthesis routes. Even though bottom-up approaches are not indeed novel, they represent a significant function in a fabricating and transformation the nanomaterial and nanostructure by giving a better chance to produce nanostructures which are less defective, while being more homogeneous regarding their chemical composition with better short and long range configuration (Hawker and Russell, 2005).

1.2 What is Interesting in Nanoparticle and Nanotechnology?

Being small in size allow for more functionality at a given space; however, nanotechnology should not be merely considered like a simple development of miniaturization from micron meter to nanometer scale. Most of the time, materials in the micrometer scale show some physical features like that of the bulk form, but in contrast, those materials in the nanometer scale might show physical properties different from that of bulk. Materials which fall in this size range show outstanding properties like the transition from atoms or molecules to bulk form within this size. Thus, there is a high interest between 100 nm to atomic level (approximately 0.2 nm). This behavior transition occurs basically for two major reasons: Surface area expansion (per unit mass) which leads to an increment in the chemical reactions accordingly, and the dominant quantum effects, of which the size of the material is minimized to tens of nanometers. The quantum effects can influence the material's electrical, optical, and magnetic features. For instance, the melting point of crystals which are in nanometer scale are low (the difference between ions get significant fraction of the total frequency of atoms). The ions and the surface energy play a crucial role in thermal stability (Daniel and Astruc, 2004). The Crystal structures which are stable at high temperatures are stable in much lower temperature when they are in nanometer size. Thus, when the materials are minimized to the nanometer scale, the ferroelectrics and ferromagnetics might lose their ferromagnetism and ferroelectricity. When the dimensions are small enough in a couple of nanometer, the bulk semiconductor changes to insulators. When the copper nanocrystals minimize to about 10 nm diameter they can be simply used to produce a surface luster, and when the single particle gets tinier to a diameter size of 7 nm, it becomes visible atomic planes with high level of crystallinity (Binns, 2010). Another class of nanoparticles is noble metal nanostructures which have much interest due to their unique properties, such as big optical area improvements that lead to the high scattering and absorption of light (Jain et al., 2008).

Every substance, including solid pieces of metal, has a grain structure. Controlling the grain structure leads to the production of higher performance materials like magnetic films with pretty high magnetization, stronger metals, nanoparticle suspension with magnetic films with a very high magnetization, suspensions of nanoparticles with tailored features, and some others (Varghese et al., 2008). Nanoparticles are getting more popular in research and industry because of their improved features in comparison with bulk materials. Some of the advantages of nanoparticles are like increased electrical conductivity, ductility and toughness, and increased metal and alloy strength and hardness. (Grzelczak et al., 2010).

Another aim of analytical nanotechnology and nanoscience is using nanomaterials like analytical tools and apply them in analysis. Practically, the

nano size structured matters with special features can be used in nanotechnology-based analytical processes. The analytical systems of nanometer scale which are based on nanosized materials are weight and size bound. The most current applications of analytical nanoscience is exploitation of exceptional physiochemical characteristics of nanomaterials (Smith et al., 2004).

The use of tools which provide molecular and elemental information with high spatial resolution is getting increasingly common in the development of nanoscience and nanotechnology such semiconductor nanoparticles as analysis tool. The available analytical techniques used for the recognition of the atomic and molecular configuration and structure at the bulk level are not usually applicable for analyzing such materials (Smith et al., 2004).

Nickel oxide (NiO) nanoparticles are popular in a lot of studies. NiO is known with a wide range of specialized applications. It is roughly pure substance suitable for specialized applications, and metallic grade, that makes is basically useful to produce alloys. It is most commonly useful to the ceramic industry to produce porcelain glazes, frit and ferrite. To make nickel steel alloy, sintered oxide is used. Moreover, nickel cement can be prepared to make the anode layer of solid oxide fuel cells and it is also used in lithium nickel oxide cathodes for lithium ion micro-batteries (Varghese, et al, (2008); (Songli, et al, (2003). As it can be noticed, nickel acid has a wide variety of applications (Salimi et al., 2007).

1.3 Semiconductor Quantum dots

The quantum dots are sufficiently small particles which make the electron energy level diverging bigger than thermal energy-the quantum size effect. The nanoparticles of semiconductor materials can be described, if stimulated, emit light much like a nanoscale light -emitting diode (LED) (Gammon and Steel, 2002).

In primarily, the electronic and optical behaviors of semiconductors are controlled by an electron energy gap between the empty conduction band and the filled valence band (Gammon and Steel, 2002). The materials are an insulator case, unless electrons are somehow stimulated and given enough energy to cross the gap. When the gaps are sufficiently narrow at room temperature, there will be enough thermal energy to excite some electrons inside the conduction band and the material obtains a very low conductivity. The gap makes them fluoresce due to an electron excited across the gap by, for instance, absorption of enough energetic photon will leave a hole in the valence band but will recombine with it in a short time, releasing the gap energy as a photon. The process is clear in the three steps shown in Figure 1.1. Initially, the incoming photons enhance the electrons across the gaps, and they leave a positively charged holes in the valence band, so the electrons interact with the holes because of their opposite charges and form a bound pair called an exciton (Figure 1.1 a). Although the electron may recombine with the hole from where it find itself, this is not likely and during the lifetime, the

absorbed photon may have any energy higher than the gap energy, and the electron can be excited well even inside the conduction band.

(a)By absorption of enough energetic photon, an electron will be enhanced from the valence band to the conduction band. The interaction of electrons and positively charged holes with each other left behind to form a bound particle pair called an exiton. (b) The energy of electron decays down through the continuation of energy levels into the conduction band even up to as a fluorescent photon and has the gap energy. So, a large group of photon energies are absorbed but mostly one wavelength can be emitted. As a matter of fact in phase (b) the electron also decays in the excitonic energy levels below the bottom of the conduction band, but in many states they are very near to the bottom of the conduction band and the energy of the fluorescent photon is not much influenced. At this case the exciton crumble that is the electron and hole recombine and the energy issued (the gap energy) is given off as a photon of light (Figure 1.1 c). So, that semiconductor fluorescence is characterized by the absorbing a large range of photon energies but the emission in one wavelength provided by the energy of gap (Chris, 2010).



Figure 1.1: Process of Fluorescence from a semiconductor

This major process happens in both nanoscale and bulk semiconductor particles quantum dots. The recombination energy is shown in Figure 1.1c. After the electron was decayed down into the conduction band states and reach to the gap edge, it then find itself at the top of another staircase of energy levels that belong to the exciton.

The quantum size effect may create unique optical properties of nanomaterial. In addition, semiconductor nanoparticles are created of up to 100–2000 atoms. These unique kinds of nanoparticles have both chemical and physical properties which are defined by two factors. The first one that the high surface (volume ratio of the nanocrystals). The second one is the particle size, that can determine the physical and electronic properties for each materials (Lu et al., 2013). When the de Broglie wavelength is bigger than the size of one crystal nanoparticle, an electron and hole are spatially bound and each electric dipole is formed, and separated electronic energy levels will be formed in each material. The separation of energy between neighboring levels can be increased by reducing size. In the semiconductor nanoparticles, the quantum size effect is most clearly observed, in which the band gap reduces by increasing the size, causing in the interband transition switching to higher frequencies (Guzelian et al., 1996). For semiconductor nanoparticles, the difference of energy between the empty conduction band and the totally filled valence band is of the order of some electron-volts and increases quickly by reduce the size (Ma et al., 2003). So it is clear that the quantum dots affect the optical properties, because the fluorescent photon color and the size of the primary gab can be easily controlled with change of quantum dots size.

The semiconductor quantum dots have a wide use in many scientific areas. The use of fluorescent molecular markers still in biological and medical research, however, quantum dots have many advantages such as high quantum yield (outer numbers of fluorescent photon, internal numbers of excitation photon). Moreover the colloidal fluorescent semiconductor quantum dots is used as immensely lighting spots, the fluorescence microscopy use is expanded for behind the fixed cells use and tissues, that can represent a possible tool to quick and accuracy diagnostic of cancer and other diseases (Farias et al., 2007).

Other applications of semiconductor nanoparticles is its strong effect to increase the sensitivity of fluorescent detection since they generate a phenomenon commonly known as enhanced fluorescence which helps to increase the lifetime of fluorescence and quantum yields. They also cause the localized surface plasmon resonance (LSPR). Many problems related to organic fluorophores in near infrared spectroscopy can be solved by quantum dots which consist of an inorganic core and metal shell with an outer coating of organic material. The presence of the inorganic core and shell makes the fluorescence tuning possible with a narrow bandwidth. This enables multiplexed identification of molecular target (Schummer, 2005). Moreover, nanoscale carbon-based materials, including multiple walled nanotubes (MWNTs), single walled nanotubes (SWNTs), and carbon nanotubes, and fibers can be applied in detection and sensing. Besides, these materials can be well used for applications in sorption and preconcentration of heavy metals ions and organic molecules before they are recognized by spectroscopic. Semiconductor nanoparticles are used also in optical sensors such as oxides nanoparticles these ions (Cu^{2+} , Mn^{2+} , Ni^{2+} , Hg^{2+}) (Lu et al., 2013).

1.3.1 Semiconductor nickel oxide nanoparticles

Semiconductor nickel oxide nanoparticles has band gap ranging from (3.50 to 4.0 eV). Nickel oxide nanoparticle shows remarkable properties which distinguishes it from bulk material. Particularly, nickel oxide nanomaterials were found to show higher reactivity catalytic activities. But bulk nickel oxide is an antiferromagnetic insulator at a Néel temperature (NT) of 523 K (Sathishkumar et al., 2014), while NiO nanoparticles is known as a p-type semiconductor which has a stable large band gap (3.5– 4.0 eV) and paramagnetic behavior (Salavati et al., 2009).

Diacritical optical properties of nanomaterials might arise from another phenomenon called the quantum size effect. This depending on the volume of the nanoparticle, especially when the particle size is less than the excitation Bohr radius of the material. Excitation Bohr radii are usually on the order of few nm for semiconductor nanoparticles. When the size of the bulk material is reduced to nano-scale, the chemical, physical, optical, and electronic properties differ a lot from those in the bulk size. In this study, the effect of size was a clear on the optical and magnetic properties of NiO nanoparticles. It is found that, the value of optical band gaps reduced from 3.60 to 3.51 eV for the specimens calcined at temperatures from 500 to 800 °C, as the particles size increased. For magnetic properties the values of resonant magnetic field were reduced from 296.4 to 289.7 G when the calcination temperature increased from 500 to 800 °C. The g-factor was also increased in the value ranging from 2.28167 to 2.29367 with the increase of calcination temperature at 500 to 800 °C. This high temperature has affected the internal magnetic field in which it increases as the particle size increases.

Nickel oxide can be synthesis by several methods at 400 °C and above. The successful way of synthesis is by pyrolyzing the nickel components such as carbonate, nitrate, and hydroxide. Various approaches for manufacturing ultrafine nickel oxide powder has been reported by previous researchers such as; the sol-gel method, hydrothermal method ,co-precipitation method , ball milling method, chemical method , decomposition method, spray pyrolysis and microwave irradiation method.

NiO nanocrystals has wide range of applications such in nickel steel alloy, sintered, anode layer of solid oxide fuel cells, lithium ion microbatteries. NiO nanocrystals is also used to prepare cathode materials in smart windows, electrochemical capacitors, alkaline batteries, and materials for gas or temperature sensors and formaldehyde sensors (Palchik et al., 1999). In addition, it has been found that magnetic nanomaterials show some strange features like huge magneto resistance, unusually high magneto caloric effect and some others.

1.4 Problem statement

Transition metal oxide nanoparticles are a broad category of materials which have undergone extensive researches because of their remarkable electronic,

catalytic, and magnetic characeristics. Nickel oxide nanoparticle is a transition metal oxide which has the formulation of NiO. Bunsenite, the mineralogical form of NiO is categorized as a basic metal oxide, and it is quite rare. Several million kilograms of NiO are produced in different quality per year, and they are mainly used as an intermediate to produce nickel alloys.

NiO can be produced through multiple methods, including microwave irradiation, chemical, sol-gel, ball milling, co-precipitation, Spray pyrolysis and de combustion method. However, application of some of these methods is not easy in a larger scale production due to some flaws. Such as hydrothermal and sol-gel methods are found time consuming and require highly unstable alkoxides and high temperatures. Spray pyrolysis method needs higher pressure. Ball milling method needs wet condition and it is found costly. Microwave irradiation and decomposition method keeping its reaction conditions is shown to be difficult, production big size particles. To overcoming of those disadvantages, the synthesis of nickel oxide by the thermal treatment method in this study.

Following the above literature, it is felt that there is a need to develop further researches on the preparation techniques of metal oxide nanoparticles. In the present study, the synthesis of NiO nanoparticles through thermal treatment by a solution holing nickel nitrate, deionized water, and polymer is focused. The solution got dried at low temperature 80 °C for 24 h prior to grinded and calcined at a temperature range of 500 to 800 °C. The advantage of this method is its lower expenses. There will be unwanted by-products any more, and it is environmentally justified. This method can be used in large scale production.

Calcination is one of the important steps involved in completing the procedures involved in the synthesis of nanoparticles by thermal treatment method. Calcination in this study started from 500 °C to show compounds of PVP still in the sample. When the calcination temperature was increased in range 600, 700, 800 °C, the PVP content and undesired anions were completely removed from the sample and all oxygen compounded with other elements such as OH, NO, NO₂, CO, CO₂ are removed during calcinations. So gradually high purity NiO nanoparticles form and begun to merged to increase the particle size with increasing calcination temperature. The effect of calcination temperature followed properties change of NiO nanoparticles which are studied in this research.

1.5 Hypothesis

During thermal treatment, nickel ions (Ni^{+2}) will react with oxygen (O_2) from the air to produce NiO atoms. These atoms due to vibration (energy) will aggregate with each other to form NiO nanoparticles when the capping PVP is removed during calcination.

1.6 Objectives of the study

This research is aimed at characterizing and synthesizing nickel oxide nanocrystals by the use of thermal treatment route. The objectives of the present study are as the following:

- i To produce high purity nickel oxide nanoparticles using thermal treatment method.
- ii To study the morphology and structural properties of nickel oxide nanoparticles.
- iii To study optical properties of nickel oxide nanoparticles.
- iiii To study magnetic properties of nickel oxide nanoparticles.

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LIST OF PUBLICATIONS

SUBMITTED ARTICLE

Manal A. Hashem, Elias B. Saion, Naif Mohammed Al-Hada, Suriati B. Paiman. "A simple thermal treatment route for fabrication of semiconductor nickel oxide (NiO) nanoparticles".

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