

**EFFECT OF CETYLTRIMETHYLAMMONIUM BROMIDE ON THE  
PROPERTIES OF NICKEL OXIDE NANOPARTICLE BY SOL-GEL  
METHOD**

**HAYMAN SAEED SALIH**

**UNIVERSITI TEKNOLOGI MALAYSIA**

EFFECT OF CETYLTRIMETHYLAMMONIUM BROMIDE ON THE  
PROPERTIES OF NICKEL OXIDE NANOPARTICLE BY SOL-GEL METHOD

HAYMAN SAEED SALIH

A dissertation submitted in partial fulfillment of the  
requirement for the award of the degree of  
Master of Science (Chemistry)

Faculty of Science  
Universiti Teknologi Malaysia

FEBRUARY 2014

*With lots of love and gratitude, I dedicate this to my beloved parents, my wife and my lovely daughter and closest friend.*

*Each of you always makes me feel special and thank you for believing in me.*

## ACKNOWLEDGEMENT

In the name of Allah, the Entirely Merciful, the Especially Merciful.

First of all, I would like to thanks Allah, for giving me the strength, patience and health to go through all obstacles in order to complete this research.

With a deep sense of gratitude, I wish to express my sincere thanks to my supervisor, Professor Dr. Madzlan Aziz, who has been helpful and offered invaluable assistance, advice, guidance, and high level inspirations for me in completing this research. This thesis would not have been possible without the guidance and the help of several individuals who in one way or another contributed and extended their valuable assistance in the preparation and completion of my study.

Many thanks for librarian of UTM. Not to be forgotten to all labratories assistance and the staff of Department of Chemistry, thanks for every helps given.

My greatest appreciation goes to my beloved parents, Hajj Saeed Salih and Hajj Sabrya Mahmood, who have raised me with their greatest love to be the person I am today, I would thanks my beloved wife, Shokhan for supporting me spiritually throughout my studies. My special appreciation extends to my brothers and sisters.

Alot of thank to my friends and my colleagues for their helps, support and advices. Last but not least, I want to extend my appreciation to the people who directly and indirectly contributed in the completion of this project.

## ABSTRACT

Metal oxide semiconductors have been widely studied due to its wide properties and applications. Among them, nickel oxide (NiO) is an important metal oxide which contributes to a wide range of applications in gas sensors, fuel cell electrodes, catalysis and optoelectronic devices. In the present study, the critical micelle concentration (CMC) of CTAB surfactant obtained was equal to  $9 \times 10^{-4}$  M. Nickel oxide nanoparticles were successfully prepared without and with CTAB surfactant by sol-gel method. Based on characterization techniques, the effect of different concentrations of cationic surfactant (cetyltrimethylammonium bromide, CTAB) on NiO nanoparticle properties were studied. Fourier transform infrared spectroscopy (FTIR) showed a wide band at  $430\text{--}530\text{ cm}^{-1}$  corresponding to the Ni-O asymmetric vibration. The resulting nickel oxide particles were highly crystalline within the range of 19.3–23.0 nm as calculated using Scherrer equation. Below (CMC) gave the smallest particle size. Moreover, X-ray diffraction (XRD) pattern showed a face centered cubic (FCC) structure. The optical properties were studied using UV-Vis-NIR scanning spectrophotometer, which shows the band gap energy of prepared NiO samples decreased with increasing CTAB concentration, indicating the increase in the conductivity. The result showed that at lower (CMC), the NiO sample displayed uniform morphology and smaller particle size as shown by the FESEM analysis.

## ABSTRAK

Semikonduktor oksida logam telah dikaji secara meluas oleh para penyelidik disebabkan oleh kepelbagaian sifat dan aplikasinya. Antaranya, nikel oksida (NiO) yang merupakan oksida logam penting yang menyumbang kepada pelbagai aplikasi dalam sensor gas, sel solar, pemangkin dan peranti optoelektronik. Dalam kajian ini, kesan kepekatan surfaktan (setiltrimetilammonium bromida, CTAB) atas sifat nanopartikel NiO telah dikaji. Kepekatan kritikal misel (CMC) bagi CTAB yang diperolehi dalam kajian ini adalah  $9 \times 10^{-4}$  M. FTIR menunjukkan jalur yang sepadan dengan getaran tak simetri Ni-O. Zarah nikel oksida yang terhasil adalah berhablur tinggi iaitu dalam julat 19.3-23.0 nm yang dikira daripada persamaan Scherrer. Kepekatan di bawah CMC telah memberikan saiz zarah yang paling kecil. Tambahan pula, XRD menunjukkan corak struktur kiub berpusat muka. Sifat optik telah dikaji dengan teknik spektrofotometer pengimbasan UV-Vis-NIR, menunjukkan bahawa tenaga jurang jalur sampel NiO yang disediakan mengalami penurunan dengan peningkatan kepekatan CTAB, serta menunjukkan peningkatan kekonduksian. Keputusan yang diperolehi menunjukkan bahawa pada kepekatan CMC yang lebih rendah, sampel NiO mempunyai morfologi seragam dan saiz zarah yang lebih kecil seperti yang ditunjukkan oleh analisis FESEM.

## TABLE OF CONTENTS

CHAPTER	TITLE	PAGE
	<b>DECLARATION</b>	ii
	<b>DEDICATION</b>	iii
	<b>ACKNOWLEDGEMENT</b>	iv
	<b>ABSTRACT</b>	v
	<b>ABSTRAK</b>	vi
	<b>TABLE OF CONTENTS</b>	vii
	<b>LIST OF TABLES</b>	x
	<b>LIST OF FIGURES</b>	xi
	<b>LIST OF SYMBOLS / TERMS</b>	xiii
	<b>LIST OF ABBREVIATION</b>	xiv
	<b>LIST OF APPENDICES</b>	xv
<b>1</b>	<b>INTRODUCTION</b>	
1.1	Introduction	1
1.2	Problem Statement	3
1.3	Objectives of study	5
1.4	Scope of the Study	5
1.5	Significance of Research	6
<b>2</b>	<b>LITERATURE REVIEW</b>	
2.1	Introduction	7
2.2	Nickel oxide	7
2.3	Nickel oxide preparation methods	9
2.3.1	Sol-gel method	10

a. Sol-gel processing	11
b. Advantage of sol-gel	13
c. Disadvantage of sol-gel	13
2.4 Particle size control principle	13
2.5 Surfactant theory	14
2.5.1 Properties of CTAB as surfactant	15
2.5.2 Critical Micelle Concentration (CMC)	16
2.5.3 Surfactant adsorption	17
2.5.4 Particle size control using surfactant	18
 <b>3 RESEARCH METHODOLOGY</b>	
3.1 Experimental Procedure	21
3.2 Chemical materials and apparatus	21
3.3 Determination of Critical Micelle Concentration of CTAB surfactant	23
3.4 Preparation of Nickel Oxide (NiO) nanoparticles	23
3.4.1 Preparation of Nickel Oxide (NiO) nanoparticles without surfactant	23
3.4.2 Preparation of Nickel Oxide (NiO) nanoparticles in the presence of CTAB with different concentration	24
3.5 Characterization Techniques	24
3.5.1 Structural information	25
3.5.2 Morphological studies	27
3.5.3 Molecular spectroscopy	28
3.5.4 Optical properties	29
3.5.5 Thermal analysis	30
 <b>4 RESULTS AND DISCUSSION</b>	
4.1 Critical Micelle Concentration (CMC) of Cetyltrimethy lammonium Bromide (CTAB)	31
4.2 Thermogravimetric (TG)/Differential thermal analysis (DTA) studies of nickel oxide nanoparticles	33



4.3	X-Ray Diffraction (XRD) Studies of Nickel oxide Nanoparticles	34
4.4	FTIR Analyses of Nickel oxide nanoparticles	41
4.5	Field Emission Scanning Electron Microscope (FESEM) Studies of Nickel oxide nanoparticles	43
4.6	UV-Vis-NIR Studies of Nickel oxide nanoparticles	46
4.7	Summary of CTAB effect	50
<b>5</b>	<b>CONCLUSIONS AND SUGGESTIONS</b>	
5.1	Conclusions	51
5.2	Suggestions for Future Work	52
	<b>REFERENCES</b>	53
	<b>APPENDIX</b>	62

**LIST OF TABLES**

<b>TABLE NO.</b>	<b>TITLE</b>	<b>PAGE</b>
2.1	Chemical and physical properties of NiO	9
4.1	Calculated particle sizes of each sample at 500°C from XRD and FESEM in different conditions	39

## LIST OF FIGURES

FIGURE NO.	TITLE	PAGE
1.1	Crystal structures of cubic NiO	1
1.2	Schematic representation of nanoparticles stabilization by (a) steric repulsion/interaction, (b) electrostatic repulsion	4
2.1	Crystal structures of cubic NiO	8
2.2	Schematic diagram for sol-gel routes; preparation of metal oxide gel through sol-gel method	12
2.3	Chemical structure of CTAB surfactant	15
3.1	Schematic exemplification of experimental procedure	22
3.2	General calculation of height halfway between background and the peak maximum	26
4.1	Plot of conductivity against CTAB concentration	32
4.2	TG and DTA curves of NiO	34
4.3	XRD diffractogram for the NiO nanoparticle prepared without CTAB surfactant calcined at 500°C	37
4.4	XRD diffractogram for the NiO nanoparticle calcined at 500°C for 1hr below CMC	38
4.5	XRD diffractogram for the NiO nanoparticle calcined at 500°C for 1hr at CMC	39
4.6	XRD diffractogram for the NiO nanoparticle calcined at 500°C for 1hr after CMC	40

4.7	FT-IR spectra for the NiO nanoparticle calcined at 500°C: (a) without CTAB. (b) below CMC. (c) at CMC. (d) after CMC.	42
4.8	FESEM micrographic for the prepared NiO nanoparticle without CTAB	44
4.9	FESEM micrographic for prepared NiO nanoparticle with CTAB (below CMC)	44
4.10	FESEM micrographic for prepared NiO nanoparticle with CTAB (at CMC)	45
4.11	FESEM micrographic for prepared NiO nanoparticle with CTAB (after CMC)	46
4.12	Bandgap energy of NiO NPs without CTAB	48
4.13	Bandgap energy of NiO NPs with CTAB (below CMC)	48
4.14	Bandgap energy of NiO NPs with CTAB (at CMC)	49
4.15	Bandgap energy of NiO NPs with CTAB (after CMC)	49

**LIST OF SYMBOLS**

eV	-	Electron Volt
°C	-	Degree Celcius
μm	-	Micrometer
nm	-	Nanometer
Å	-	Angstrom
g.mol <sup>-1</sup>	-	Gram per mole
h	-	Hour
mol/L	-	Mole per liter
cm	-	Centimeter
K	-	Kelvin
min	-	Minute
θ	-	Angle Theta
%	-	Percentage
g	-	Gram
mL	-	Mili Liter
M	-	Molarity
mW	-	Milliwatt
v	-	volume

## LIST OF ABBREVIATION

CTAB	-	Cetyltrimethylammonium bromide
FTIR	-	Fourier Transform Infrared Spectroscopy
FESEM	-	Field Emission Scanning Electronic Microscope
XRD	-	X – Ray Diffractometer
MW	-	Molecular Weight
TG	-	Thermogravimetric
FWHM	-	Full-Width at Half Maximum
NP	-	Nanoparticle
TX-100	-	Triton X-100
CMC	-	Critical micelle concentration
TTAB	-	Tetradecyltrimethylammonium bromide

**LIST OF APPENDICES**

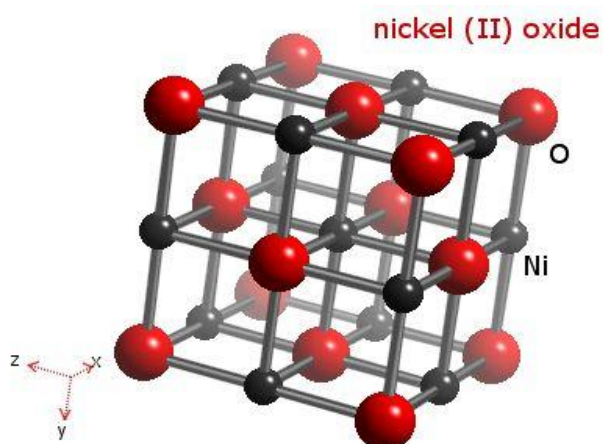
<b>APPENDIX</b>	<b>TITLE</b>	<b>PAGE</b>
	Calculation of nanoparticles size using Scherrer equation of XRD diffraction	63

## CHAPTER 1

### INTRODUCTION

#### 1.1 Introduction

Nickel oxide (NiO) is a green and well characterized oxide that crystallises with the cubic structure and has a *p*-type wide band gap (3.6-4.0 eV) semiconductor [1]. Figure 1.1 shows the six coordination number both for nickel and oxygen atoms and NiO belongs to NaCl structure, so-called rock salt structure [2]. Nickel oxide is an important antiferromagnetic semiconductor with excellent properties such as gas-sensing, catalytic and electrochemical properties. NiO nanoparticles are widely used in different fields such as catalysis, electrochromic films and fuel cell electrodes due to their mechanical, electronic, magnetic and optical properties [3].



**Figure 1.1:** Crystal structure of cubic NiO.



Bulk NiO showed antiferromagnetic character below the neel temperature of 523 K and the magnetic properties can be modified with reduction of particle size. The antiferromagnetic character of NiO is super paramagnetic (300 K) when the particle size reaches approximately 100 nm [4].

Various techniques can be used to synthesize NiO nanoparticle some of which including: sol–gel, laser ablation, hydrothermal, solvothermal, chemical vapor deposition, anodic plasma, precipitation, microemulsions technique, sonochemical and thermal decomposition [5, 6]. Among all the above mention techniques the sol gel method is most suitable for the synthesis of nickel oxide nanoparticle because it offers several advantages over other methods. Some of the advantages of sol-gel method posses over other methods including: low processing temperature, better homogeneity, controlled stoichiometry, low processing cost, better control over the particle size of the crystallite, flexibility of forming dense monoliths and thin films, or nanoparticles.

Surfactants are used in the preparation of crystalline metal oxide in the nanoscale to control crystal growth and to provide solubility. Surfactants help to organize the small oxide crystallites into a sufficiently ordered structure to generate regular pores and low angle of Bragg reflection [7]. The use of surfactants, generally consisting of a coordinating head group and a long alkyl chain, offers several advantages. The coating of the nanoparticles prevents agglomeration during synthesis and results in good colloidal stability of the final product in organic solvents. Dynamic adsorption and desorption of surfactant molecules onto particle surfaces during particle growth, sometimes combined with selectivity toward specific crystal faces, enables control over particle size, size distribution and morphology [8]. Moreover, the surfactants can be exchanged against other ones in a post synthetic step, allowing the chemical modification of the surface properties of the nanoparticles [9]. Although the organic species strongly influence the structural, compositional and morphological characteristics of the inorganic product and thus offer a versatile tool for tailoring particle size, shape, composition and surface properties, this is only possible on an empirical basis as long as the organic-inorganic interface is not completely

understood on a molecular level and as long as there is no detailed information available about how organic molecules bind and pack on nanocrystal surfaces [10].

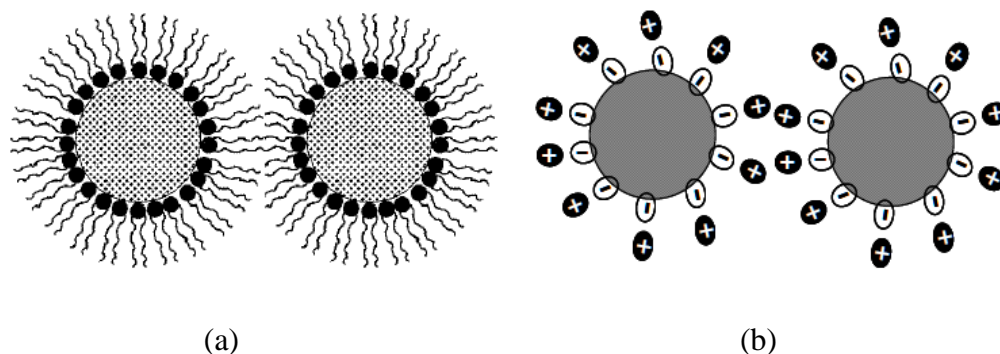
The surfactants have to be removed firstly by washing the product with distilled water and then completely removed by burning the samples at higher temperature to improve thermal stability and the purity of the nickel oxide nanoparticle [11]. The main purpose of the present research is to study the influence of cationic surfactant (CTAB) on the nickel oxide nanoparticle properties such as particle size and morphology.

## **1.2 Problem statement**

Semiconductor nanocrystals have unique properties of controlling size and shapes which contain thousands of atoms with the periodicity of bulk semiconductors among the other known molecular and solid-state regime materials. However, the structure of electronic levels and the resulting electrical and optical properties can be strongly modified as the physical dimensions of the nanocrystals confine the wave functions of electrons and gaps. Reduction of the nanocrystals size causes a characteristic blue shift of the band gap [12].

Nanoparticles are thermodynamically unstable because of large surface free energy. In order to obtain stable nanoparticles, they have to be stabilized kinetically during the reaction by adding stabilizing agents such as organic/inorganic ligands or capping agents. Nanocrystals dispersed in liquids are either charge-stabilized or sterically stabilized [13]. In the case of sterically stabilized nanoparticles, capping agent is adsorbed on the surface of the particles either by forming chemical bonds or by weak interactions. These capping agents act as stabilizers due to repulsion between their tail groups as shown in Figure 1.2a.

In the case of charge stabilized nanoparticles, an electric double layer is formed on the surface of the particle. This double layer acts as stabiliser due to electrostatic repulsion between layers of different particles as shown in Figure 1.2b [13].



**Figure 1.2:** Schematic representation of nanoparticles stabilization by: (a) Steric repulsion/interaction. (b) Electrostatic repulsion.

The wide band gap makes NiO an attractive matrix for electrically activated emission, since wide band gap semiconductors have higher excitation ionization energy [14]. Recently, it was found that small NiO nanoparticle has many defects such as oxygen vacancies because of a large area. This will lead to an increase in the trap of emission in the visible region [15]. These ideal surfaces do not exist in the case of nanoparticles, thereby increasing the number of unreconstructed surfaces, dangling bonds, and surface vacancies that induce acceptor or donor levels at the grain surface [16].

However, it was still difficult to control the size of nanoparticles. Recently, it has been noted that anionic, cationic and nonionic surfactants can be used to assist the formation of nanoscaled materials [17]. Hence, this study focuses on the study of nickel oxide nanoparticles properties with an organic compound, specifically the CTAB surfactant, because CTAB is a cationic surfactant and ionizes completely in aqueous solution. The resulted cations were a tetrahedron with a long hydrophobic tail. Therefore, ion-pairs between  $[\text{Ni}(\text{OH})_4]^{2-}$  and  $\text{CTA}^+$  could form due to electrostatic interaction [18].

In the crystallization process, surfactant molecules adsorbed on the crystal nuclei not only serve as a growth director but also as a protector to prevent from aggregation of the product. As a result, NiO nanoparticles were produced. According to above results, the smallest sized and well-dispersed NiO nanoparticles were obtained successfully with cationic surfactant CTAB [19].

### **1.3 Objectives of study**

The objectives of this study are:

1. To determine the critical micelle concentration (CMC) of cationic surfactant used.
2. To synthesize nickel oxide nanoparticle without and with CTAB surfactant.
3. To characterize the product using TGA, XRD, FTIR, FESEM and UV-visible spectroscopy.
4. To study the effect of CTAB concentration (below, at and after CMC) on the nickel oxide properties.

### **1.4 Scope of the Study**

In this study, the type of surfactant used is cationic surfactant (cetyltrimethylammonium bromide (CTAB)). This research focuses more on the effect of surfactant concentration on the nickel oxide properties. Initial part of the study to determine the critical micelle concentration (CMC) of CTAB surfactant, followed by preparation of nickel oxide nanoparticle using sol-gel method without surfactant (CTAB). Furthermore preparing nickel oxide nanoparticle using CTAB as surfactant by adding different concentration (below, at and after CMC) of the surfactant to the reaction mixture. Characterizations include X-ray diffraction (XRD)

to study the structure and the size of crystals via Debye-Scherrer's equation, field emission scanning electron microscope (FESEM) for imaging the morphology of the surface, fourier transformed infrared (FTIR) spectroscopy were employed to shows the type of chemical bonds are present in the sample and UV-Vis-NIR scanning spectrophotometer for studying optical properties.

## **1.5 Significance of Research**

The importance of this study is to provide an alternative method of sol-gel preparation to produce nickel oxide nanoparticle, which have nanostructure grain in order to extend its application, furthermore to improve the properties of nickel oxide nanoparticle by the addition of surfactant at different concentration depending on the critical micelle formation. Micellization is observed in surfactant solution when concentration exceeds the critical micelle concentration (CMC), whereas the physicochemical properties of the aqueous solution change abruptly, different physical and chemical properties such as electrical conductivity, interfacial tension and detergency dramatically change. The solubility of hydrocarbons is greatly increased if the concentration of surfactant is above the CMC. At this concentration, the polar and nonpolar groups become oriented and organized to form clusters in the solution which referred as micelles. The hydrophilic groups point outwards from within the micelles and the hydrophobic groups point inwards within the micelles and often have hydrocarbon character [20]. Therefore practical applications of surfactants depend on accurate knowledge of CMC [21]. So in the present study, the properties of nickel oxide nanoparticle had studied by varying the concentration of surfactant above the CMC value, equal to CMC and also below CMC.

## REFERENCES

1. Mohammadyani, D., Hosseini, S. A. and Sadrnezhad, S. K. (2012). Characterization of nickel oxide nanoparticles synthesized via rapid microwave-assisted route. *International Journal of Modern Physics: Conference Series*. **5**, 270-276.
2. [http://www.webelements.com/compounds/nickel/nickel\\_oxide.html](http://www.webelements.com/compounds/nickel/nickel_oxide.html).
3. Patil, V., Pawar, S., Chougule, M., Godse, P., Sakhare, R., Sen, S. and Joshi, P. (2011). Effect of annealing on structural, morphological, electrical and optical studies of nickel oxide thin films. *Journal of Surface Engineered Materials and Advanced Technology*. (1), 35-41.
4. Alagiri, M., Ponnusamy, S. and Muthamizhchelvan, C. (2012). Synthesis and characterization of NiO nanoparticles by sol-gel method. *Journal of Materials Science: Materials in Electronics*. **23**(3), 728-732.
5. Srivastava, N. and Srivastava, P. C. (2010). Synthesis and characterization of (single-and poly-) crystalline NiO nanorods by a simple chemical route. *Physica E: Low-dimensional Systems and Nanostructures*. **42**(9), 2225-2230.
6. Pilban Jahromi, S., Nay Ming, H., Rasat Muhamad, M. and Hong Ngee, L. (2012). Green gelatine-assisted sol-gel synthesis of ultrasmall nickel oxide nanoparticles. *Ceramics International*. **39**(3), 3909-3914.
7. Severin, K. and Abdel-Fattah, T. (1998). Supramolecular assembly of mesostructured tin oxide. *Chemical Communications*. 1471-1472.
8. Kumar, S. and Nann, T. (2006). Shape control of II-VI semiconductor nanomaterials. *Small*. **2**(3), 316-329.

9. Niederberger, M. (2007). Nonaqueous sol–gel routes to metal oxide nanoparticles. *Accounts of Chemical Research*. **40**(9), 793-800.
10. Yin, Y. and Alivisatos, A. P. (2004). Colloidal nanocrystal synthesis and the organic–inorganic interface. *Nature*. **437**(7059), 664-670.
11. Wang, Y. D., Ma, C. L., Sun, X. D. and Li, H. D. (2002). Preparation of nanocrystalline metal oxide powders with the surfactant-mediated method. *Inorganic Chemistry Communications*. **5**(10), 751-755.
12. Millo, O., Katz, D. and Yun, W. C. and Banin, U. (2001). Tunneling and Optical Spectroscopy of InAs and InAs/ZnSe Core/Shell Nanocrystalline Quantum Dots. *Physica Status Solidi (b)*. **224**, 271–276.
13. Rao, C. N. R., Thomas, P. J. and Kulkarni, G. U. (2007). Nanocrystals: synthesis, properties and applications. *Springer*. **95**, 3-4.
14. Hayakawa, T. and Nogami, M. (2005). High luminescence quantum efficiency of Eu<sup>3+</sup>-doped SnO<sub>2</sub>–SiO<sub>2</sub> glasses due to excitation energy transfer from nano-sized SnO<sub>2</sub> crystals. *Science and Technology of Advanced Materials*. **6**(1), 66.
15. Arik, k., Simanta, K. and Amitava, P. (2011). Surface Defect-Related Luminescence Properties of SnO<sub>2</sub> Nanorods and Nanoparticles. *Journal of Physics Chemistry C*. **115**, 118–124.
16. Raul, D. D. (2002). Tin Oxide Gas Sensors an Electrochemical Approach. Report *Dissertation of Doctor in Chemistry*. Barcelona University. Dubaray **16**.
17. Gnanam, S. and Rajendran, V. (2010). Anionic, cationic and nonionic surfactants-assisted hydrothermal synthesis of Tin oxide nanoparticles and their photoluminescence property. *Digest Journal of Nanomaterials and Biostructures*. **5**(2), 623.
18. Sun, X. M., Chen, X., Deng, Z. X. and Li, Y. D. (2003). A CTAB-assisted hydrothermal orientation growth of ZnO nanorods. *Materials Chemistry and Physics*. **78**(1), 99-104.
19. Anandan, K. and Rajendran, V. (2012). Structural, optical and magnetic properties of well-dispersed NiO nanoparticles synthesized by CTAB assisted

- solvothermal process. *Nanoscience and Nanotechnology: An International Journal*. **2**(4), 24-29.
20. Li, E., Zhang, G., Dong, J., Zhou, X., Yan, X. and Lou, M. (2004). Estimation of critical micelle concentration of anionic surfactants with OSPR approach. *Journal of Molecular Structure*. **710**, 119-126.
  21. Chun, H. T., Zhen, J. H. and Xu, G. H. (2010). Rapid determination of surfactant critical micelle concentration in aqueous solutions using fiber-optic refractive index sensing. *Analytical Biochemistry*. **401**, 144-147.
  22. Sawatzky, G. A. and Allen, J. W. (1984). Magnitude and origin of the band gap in NiO. *Physical Review Letters*. **53**(24). 2339.
  23. <http://www.chemexplore.net/ReO3-NiO.htm>.
  24. Wen, G., Hui, K. S. and Hui, K. N. (2012). High conductivity nickel oxide thin films by a facile sol-gel method. *Materials Letters*. **92**, 291-295.
  25. Kumar Rai, A., Tuan Anh, L., Park, C. J. and Kim, J. (2013). Electrochemical study of NiO nanoparticles electrode for application in rechargeable lithium-ion batteries. *Ceramics International*. **39**, 6611-6618.
  26. Avendano, E., Berggren, L., Niklasson, G. A., Granqvist, C. G. and Azens, A. (2006). Electrochromic materials and devices: Brief survey and new data on optical absorption in tungsten oxide and nickel oxide films. *Thin Solid Films*. **496**(1), 30-36.
  27. Bouessay, I., Rougier, A., Poizot, P., Moscovici, J., Michalowicz, A. and Tarascon, J. M. (2005). Electrochromic degradation in nickel oxide thin film: A self-discharge and dissolution phenomenon. *Electrochimica Acta*. **50**(18), 3737-3745.
  28. Agrawal, A., Habibi, H. R., Agrawal, R. K., Cronin, J. P., Roberts, D. M., Caron-Popowich, R. S. and Lampert, C. M. (1992). Effect of deposition pressure on the microstructure and electrochromic properties of electron-beam-evaporated nickel oxide films. *Thin Solid Films*. **221**(1), 239-253.



29. Mohseni Meybodi, S., Hosseini, S. A., Rezaee, M., Sadrnezhad, S. K. and Mohammadyani, D. (2012). Synthesis of wide band gap nanocrystalline NiO powder via a sonochemical method. *Ultrasonics Sonochemistry*. **19**(4), 841-845.
30. Soleimanpour, A. M. and Jayatissa, A. H. (2012). Preparation of nanocrystalline nickel oxide thin films by sol–gel process for hydrogen sensor applications. *Materials Science and Engineering*. **32**(8), 2230-2234.
31. Skandan, G. and Singhal, A. (2006). 2 Perspectives on the Science and Technology of Nanoparticle Synthesis. 1-15.
32. Wandstrat, M. M. (2006). Materials and Modification of Electrodes for the Detection of Biological Molecules (Doctoral dissertation, Miami University). 1.
33. Rao, M. S. and Dave, B. C. (1998). Selective intake and release of proteins by organically-modified silica sol-gels. *Journal of the American Chemical Society*. **120**(50), 13270-13271.
34. Rao, M. S., Dubenko, I. S., Roy, S., Ali, N. and Dave, B. C. (2001). Matrix-assisted biomimetic assembly of ferritin core analogues in organosilica sol-gels. *Journal of the American Chemical Society*. **123**(7), 1511-1512.
35. Rao, M. S. and Dave, B. C. (2006). Use of environmentally-responsive smart glasses in controlled release: Diffusion of molecules under applied stimuli. *Journal of Sol-gel Science and Technology*. **40**(2-3), 241-251.
36. Bradley, D. C. (1989). Metal alkoxides as precursors for electronic and ceramic materials. *Chemical Reviews*. **89**(6), 1317-1322.
37. Segal, D. (1997). Chemical synthesis of ceramic materials. *Journal of Material Chemistry*. **7**(8), 1297-1305.
38. Beach III, E. R. (2009). Picoliter drop deposition of oxide nanoparticles: a route to high performance microsensor arrays. 41.
39. Korošec, R. C., Bukovec, P., Pihlara, B., Vuk, A. S., Orel, B. and Drazic, G. (2003). Preparation and structural investigations of electrochromic nanosized NiOx films made via the sol–gel route. *Solid State Ionics*. **165**, 191-200.

40. Rifaya, M. N., Theivasanthi, T. and Alagar, M. (2012). Chemical Capping Synthesis of Nickel Oxide Nanoparticles and their Characterizations Studies. *Nanoscience and Nanotechnology*. **2**(5), 134-138.
41. Wuled Lenggoro, I., Itoh, Y., Iida, N. and Okuyama, K. (2003). Control of size and morphology in NiO particles prepared by a low-pressure spray pyrolysis. *Materials Research Bulletin*. **38**(14), 1819-1827.
42. Wang, Y. D., Ma, C. L., Sun, X. D. and Li, H. D. (2002). Preparation of nanocrystalline metal oxide powders with the surfactant-mediated method. *Inorganic Chemistry Communications*. **5**(10), 751-755.
43. Wang, C. B., Gau, G. Y., Gau, S. J., Tang, C. W. and Bi, J. L. (2005). Preparation and characterization of nanosized nickel oxide. *Catalysis Letters*. **101**(3-4), 241-247.
44. Ahire, D. V., Patil, G. E., Jain, G. H. and Gaikwad, V. B. (2012). Synthesis of nanostructured NiO by hydrothermal route and its gas sensing properties. *In Sensing Technology (ICST), Sixth International Conference*. 136-141.
45. Kostecki, P. T., Calabrese, E. J. and Bonazountas, M. (1992). Hydrocarbon Contaminated Soils: Analytical Methodologies, Site Assessment, Environmental Fate, Risk Assessment, Regulatory Considerations, Remediation. (Vol. 2). *CRC Press*.
46. Thomas, S. P. (1996). Surfactant-enhanced electrokinetic remediation of hydrocarbon-contaminated soils. M.Sc. Thesis in Biosystems Engineering, University of Manitoba, Winnipeg, Manitoba, Canada.
47. Kile, D. E. and Chiou, C. T. (1989). Water solubility enhancements of DDT and trichlorobenzene by some surfactants below and above the critical micelle concentration. *Environmental Science and Technology*. **23**(7), 832-838.
48. Ger, M. D., Lee, J. L., Ke, S. T., Chen, Y. C., Kuo, S. L. and Wong, C. H. (2008). The influence of surfactant CTAB on the microstructure and material properties of nickel microelectroforming. *Key Engineering Materials*. **364**, 346-350.

49. Lai, H., Du, Y., Zhao, M., Sun, K. and Yang, L. (2014). CTAB assisted hydrothermal preparation of  $\text{YPO}_4\text{:Tb}^{3+}$  with controlled morphology, structure and enhanced photoluminescence. *Materials Science and Engineering*. **179**, 66-70.
50. Wang, Y. X., Sun, J., Fan, X. and Yu, X. (2011). A CTAB-assisted hydrothermal and solvothermal synthesis of ZnO nanopowders. *Ceramics International*. **37**(8), 3431-3436.
51. Lokajová, J., Hruška, V., Tesařová, E. and Gaš, B. (2008). System peaks in micellar electrophoresis: I. Utilization of system peaks for determination of critical micelle concentration. *Electrophoresis*. **29**(5), 1189-1195.
52. Kostecki, P. T. and Calabrese, E. J. (Eds.). (1993). Principles and practices for petroleum contaminated soils. *CRC Press*.
53. Porter, M. R. and Porter, M. R. (1991). Handbook of surfactants. London: Blackie. 55-59.
54. Steinlin, A. (2006). Determination of the critical micelle concentration of an amphiphile by conductivity measurements. PCI, Praktikum Pharmazie. 1-11.
55. Vaidya, S., Rastogi, P., Agarwal, S., Gupta, S. K., Ahmad, T., Antonelli Jr, A. M. and Ganguli, A. K. (2008). Nanospheres, nanocubes, and nanorods of nickel oxalate: control of shape and size by surfactant and solvent. *The Journal of Physical Chemistry C*. **112**(33), 12610-12615.
56. Ranjan, R., Vaidya, S., Thaplyal, P., Qamar, M., Ahmed, J. and Ganguli, A. K. (2009). Controlling the size, morphology and aspect ratio of nanostructures using reverse micelles: a case study of copper oxalate monohydrate. *Langmuir*. **25**(11), 6469-6475.
57. Ganguly, A., Ahmad, T. and Ganguli, A. K. (2010). Silica mesostructures: control of pore size and surface area using a surfactant-templated hydrothermal process. *Langmuir*. **26**(18), 14901-14908.
58. Bagwe, R. P. and Khilar, K. C. (1997). Effects of the intermicellar exchange rate and cations on the size of silver chloride nanoparticles formed in reverse micelles of AOT. *Langmuir*. **13**(24), 6432-6438.

59. Eastoe, J., Hollamby, M. J. and Hudson, L. (2006). Recent advances in nanoparticle synthesis with reversed micelles. *Advances in Colloid and Interface Science*. **128**, 5-15.
60. Eastoe, J. and Sharpe, D. (1997). Properties of phosphocholine microemulsions and the film rigidity model. *Langmuir*. **13**(13), 3289-3294.
61. Cason, J. P., Miller, M. E., Thompson, J. B. and Roberts, C. B. (2001). Solvent effects on copper nanoparticle growth behavior in AOT reverse micelle systems. *The Journal of Physical Chemistry B*. **105**(12), 2297-2302.
62. Capek, I. (2004). Preparation of metal nanoparticles in water-in-oil (w/o) microemulsions. *Advances in Colloid and Interface Science*. **110**(1), 49-74.
63. Gao, L., Wang, E., Lian, S., Kang, Z., Lan, Y. and Wu, D. (2004). Microemulsion-directed synthesis of different CuS nanocrystals. *Solid State Communications*. **130**(5), 309-312.
64. Paria, S. and Khilar, K. C. (2004). A review on experimental studies of surfactant adsorption at the hydrophilic solid–water interface. *Advances in Colloid and Interface Science*. **110**(3), 75-95.
65. Gamboa, C., Barraza, R. and Olea, A. F. (2004). Interaction between anionic polyelectrolytes and ctab micelles: a potentiometric study. *Journal of the Chilean Chemical Society*. **49**(4), 303-306.
66. Li, N., Liu, S. and Luo, H. (2002). A new method for the determination of the first and second CMC in CTAB solution by resonance Rayleigh scattering technology. *Analytical Letters*. **35**(7), 1229-1238.
67. Modaressi, A., Sifaoui, H., Grzesiak, B., Solimando, R., Domanska, U. and Rogalski, M. (2007). CTAB aggregation in aqueous solutions of ammonium based ionic liquids; conductimetric studies. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*. **296**(1), 104-108.
68. El-Kemary, M., Nagy, N. and El-Mehasseb, I. (2013). Nickel oxide nanoparticles: Synthesis and spectral studies of interactions with glucose. *Materials Science in Semiconductor Processing*. **16**(6), 1747-1752.

69. Wang, Y. D., Ma, C. L., Sun, X. D. and Li, H. D. (2002). Preparation of nanocrystalline metal oxide powders with the surfactant-mediated method. *Inorganic Chemistry Communications*. **5**(10), 751-755.
70. Qiao, H., Wei, Z., Yang, H., Zhu, L. and Yan, X. (2009). Preparation and characterization of NiO nanoparticles by anodic arc plasma method. *Journal of Nanomaterials*. **2**.
71. Wongsaprom, K. and Maensiri, S. (2013). Synthesis and Room Temperature Magnetic Behavior of Nickel Oxide Nanocrystallites. *Ciang Mai Journal of Science*. **40**(1), 99-108.
72. Farrukh, M. A., Tan, P. and Adnan, R. (2012). Influence of reaction parameters on the synthesis of surfactant-assisted tin oxide nanoparticles. *Turk Jorنال Chemistry*. **1**, 12.
73. Bryleva, E. Y., Vodolazkaya, N. A., Mchedlov-Petrosyan, N. O., Samokhina, L. V., Matveevskaya, N. A. and Tolmachev, A. V. (2007). Interfacial properties of cetyltrimethylammonium-coated SiO<sub>2</sub> nanoparticles in aqueous media as studied by using different indicator dyes. *Journal of Colloid and Interface Science*. **316**(2), 712-722.
74. Klopprogge, J. T., Hickey, L. and Frost, R. L. (2004). FT-Raman and FT-IR spectroscopic study of synthetic Mg/Zn/Al-hydrotalcites. *Journal of Raman Spectroscopy*. **35**(11), 967-974.
75. Ganachari, S. V., Bhat, R. and Deshpande, R. (2012). Synthesis and characterization of nickel oxide nanoparticles by self-propagating low temperature combustion method. *Recent Research in Science and Technology*. **4**(4), 50-53.
76. Gajendiran, J. and Rajendran, V. (2011). Size controlled and optical properties of Zn-doped SnO<sub>2</sub> nanoparticles via sol-gel process. *Optoelectronics and Advanced Materials – Rapid Communication*. **5**, 44–48.
77. Galhotra, P. (2010). Carbon dioxide adsorption on nanomaterials. The University of Iowa. 115.

78. Dharma, J., Pisal, A. and Shelton, C. T. (2009). Simple Method of Measuring the Band Gap Energy Value of  $\text{TiO}_2$  in the Powder Form using a UV/Vis/NIR Spectrometer. Application Note.
79. Barakat, A., Al-Noaimi, M., Suleiman, M., Aldwayyan, A. S., Hammouti, B., Hadda, T. B. and Warad, I. (2013). One Step Synthesis of NiO Nanoparticles via Solid-State Thermal Decomposition at Low-Temperature of Novel Aqua (2, 9-dimethyl-1, 10-phenanthroline)  $\text{NiCl}_2$  Complex. *International Journal of Molecular Sciences*. **14**(12), 23941-23954.
80. Indulal, C. R., Kumar, G. S., Vaidyan, A. V. and Raveendran, R. (2011). Effect of Thermal Annealing on the Structural and Optical Properties Nanostructured Cobalt-Manganese and Cobalt-Nickel Oxides Prepared by Co-Precipitation. *In AIP Conference Proceedings*. **1391**, 564-566.
81. Fetzer, C. M., Lee, R. T., Stringfellow, G. B., Liu, X. Q., Sasaki, A. and Ohno, N. (2002). Effect of surfactant Sb on carrier lifetime in GaInP epilayers. *Journal of Applied Physics*. **91**(1), 199-203.
82. Gleiter, H. (2000). Nanostructured materials: basic concepts and microstructure. *Acta Materialia*. **48**(1), 1-29.
83. Graeve, O. A., Fathi, H., Kelly, J. P., Saterlie, M. S., Sinha, K., Rojas-George, G. and Lopez, E. A. (2013). Reverse micelle synthesis of oxide nanopowders: Mechanisms of precipitate formation and agglomeration effects. *Journal of Colloid and Interface Science*. **407**, 302-309.