University of Kurdistan
Faculty of Science
Department of Chemistry

Title:
Fabrication and Characterization of Metal Oxide Nanostructures and their Application for Construction of Sensors and Biosensors Application of Carbon Nanotube/ Rh-Complex Nanocomposite for Preparation of Nitrite Sensor

By:
Rahman Hallaj

Supervisor:
Dr. Abdollah Salimi

Advisor:
Dr. Raof Ghavami
Dr. Saeed Soltanian

A Thesis
Submitted in Partial Fulfillment of the Requirements for the Degree of
Ph.D. in Analytical Chemistry

April 16, 2009

Abstract:

Electrochemical sensors and biosensors are very favorable due to their high sensitivity and selectivity, portable field-based size, rapid response time and low-cost. On the other hand, electrochemical sensors have had some limitations: electrochemically active interferences in the sample, weak long-term stability, and particularly troublesome electron-transfer pathways. Recently nanostructures have been significant role in development of fast, sensitive and selective electrochemical sensors and biosensors for clinical, inviromental and industrial analysis. Among various nanostructured materials, metal oxides and carbon nanotubes have wide practical applications in the field of electroichemistry in particular electrocatalysis. Furthermore electrochemical properties of nanostructres strongly depended to the construction method. Between different possible synthesis methods for preparation of metal oxide nanostructures, electrodeposition synthesis are attracting because it is safe, environmentally friendly, easy and is typically performed at low temperatures (less than 200 ^{0}C). Electrocrystalization methods and various affective parameters were explained. In addition, different characterization methods were briefly illustrated. Finally, application of metal and metal oxide nanostructures in design and construction of sensors and biosensors were studied.

In chapter 2, fabrication of three types of metal oxides was studied. Metal oxide nanostructures, including cobalt oxide (CoOx), zinc oxide (ZnO), and iridium oxide (IrOx) nanostructures, were prepared by the electrodeposition techniques. A polished and clean surface of the glassy carbon electrode is used as a substrate for electrodeposition of metal oxide nanoparticles. Preparations of cobalt and iridium oxides nanoparticles were taken place during dynamic potential condition, but static potential condition was used to

synthesis of zinc oxide nanoparticles. The morphology and structure of resultant nanoparticles were evaluated by scanning electron microscopy (SEM) and X-ray diffractometry (XRD). Electrochemical behaviors of metal oxide nanostructures were characterized by impedance spectroscopy and cyclic voltammetry. Shape and size of cobalt oxide are changed due to the variation in applied potential condition from spherical to worm like nanostructures.

In chapter 3, further characterization and different applications of cobalt oxide nanostructures was studied. Cyclic voltammograms of electrodeposited cobalt oxide nanostructures on to glassy carbon electrode showed well defined and stable redox couple due to the reduction and oxidation of cobalt oxide and hydroxide film. The electrochemical behavior of cobalt oxide film was studies in solutions with different pH value. Kinetic charge transfer properties of corresponded redox couple were evaluated. The result showed cobalt oxide could be used as an electron transfer mediator due to its fast electron transfer properties. Recorded cyclic votammograms of cobalt oxide in the presence and absence of different analyte showed excellent electrocatalytic activity toward hydrogen peroxide, insulin, guanine and arsenit ions. Furthermore, due to high porosity and good compatibility of cobalt oxide nanostructure, it can be used to immobilization of different enzymes and biomolecules. Cobalt oxide was used to immobilization of Hemoglobin (Hb), Flavin Adenine Dinucleotide phosphate (FAD) and Cholesterol Oxidase(CHox). Immobilizations of these biomolecules were taken place via consequent cyclic voltammetry. Electrochemical behaviors of constructed bio-electrodes were investigated by cyclic voltammetry, impedance spectroscopy and UV-Visible spectroscopy. Applicability of the fabricated cobalt oxide/biomolecules modified

electrodes to determination of different spices was studied. Recorded cyclic voltammograms of FAD-cobalt oxide, Hb-cobalt oxide and ChOx-Cobalt oxide modified electrode showed drastic electrocatalytic activity toward nitrite, hydrogen peroxide and cholesterol respectively. Typical amperometry and flow injection analysis were used to achieving the optimum analytical parameters.

Applicability of the electrodeposited nanostructures of zinc and iridium oxides in designing of sensor and biosensor was studied in chapter 4. Electrodeposition under potentiostatic condition was used for the fabrication of zinc oxide nanoparticles. The SEM-imaging techniques showed that the obtained ZnO nanparticles are spherical , with same size and shape. Due to the individual interaction between zinc oxide and most biomolecules, immobilization of guanine oxidation products was take place by repetitive cyclic voltammetry. Electrochemical investigations indicated that 8-oxo-guanine was immobilized onto zinc oxide nanoparticles. Direct electrochemistry of 8-oxo-guanine was studied at the surface of zinc oxide nanoparticles. Finally, electrcatalytic activities of the modified metal oxide nanoparticles were proved by voltammetric techniques. The results showed excellent electrocatalytic activity of 8-oxo-guanine modified glassy carbon electrode toward the electrooxidation of l-cysteine. Amperometric technique was used to evaluation of the fabricated sensor. Morphology of resultant Iridium oxide film during electrochemical deposition was investigated by AFM and SEM microscopy. The results showed very rough and needle like structure. Cyclic voltammograms of the Iriduim oxides nanoparticle showed well defined and very stable redox couple, due to reduction and oxidation of oxide film. In this work for the first time, iridium oxide nanoparticles were used in electroreduction process. Recorded cyclic voltammograms of iridium oxide-

glassy carbon electrode showed drastic catalytic activities toward electroreduction of iodate and periodate at unusual positive potential (+0.7V). Typical amperometry and flow injection analysis were used to evaluation of analytical parameters of modified electrode. Based on consumption of periodate in the presence of vicinal-diols, a sensitive sensor for sugars was fabricated. Cyclic voltammetry, amperommetry and flow injection analysis were used to determination of sugars via consumption of periodate.

In the last part of this thesis, the GC electrode was modified by multiwall carbon nanotube and Rh-Complex. Electrochemical behavior of the modified electrode was investigated in different pH values. The electrocatalytic behavior of modified electrode in presence of nitrite was studied by cyclic voltammetry. The results showed two well known catalytic responses corresponded to electrocatalytic oxidation (at 0.75V) and electroreduction (at -0.5V) of nitrite. Typical amperometric and Flow injection analysis were used to characterization of fabricated sensors. Anti-interferences effect of the modified electrode was proved by recording of amperometric responses in the presence of nitrite and different interferences. The performance of sensor was investigated by determination of nitrite in meat products as a real sample. The results obviously shows the advantages of nanostructures which applied in fabrication of sensors and biosensors.

Declaration

I declare that this thesis represents my own work, except where due acknowledgement is made, and that it has not been previously included in a thesis, dissertation or report submitted to this University or any other institution for a degree, diploma or other qualifications.

Signed: _____
Rahman Hallaj

Acknowledgments

I would like to express my sincerest thanks and deepest appreciation to Dr. Abdollah Salimi as my supervisors , Dr. Saied Soltanian and Dr. Raoof Gavami for their guidance, encouragement and support throughout my study at the University of Kurdistan. Special acknowledgements would be given to my friends Mr. Maroof Oraz, Mr. Ali Berenji, Mr. Jalal Oraz and Mr. Keyvan Akhtari for their kindness. I am thankful to Miss. Begard Kavosi and Shiva zandi. I am deeply grateful to my brother Rahim Hallaj Finally, I would like to thank my family for their encouragement and deep love. This dissertation is especially to my lovely and beautiful daughter, whose name is

Raman Hallaj

And finally
To whom change my life

'My wife'

List of Publications

Journal Articles:

1- Abdollah Salimi, **Rahman Hallaj**, Saied Soltanian and Hussein Mamkhezri Nanomolar detection of hydrogen peroxide on glassy carbon electrode modified with electrodeposited cobalt oxide nanoparticles. ***Analytica Chimica Acta, Volume 594, Issue 1, 26 June 2007, Pages 24-31.***

2-- Abdollah Salimi, **Rahman Hallaj**, Saied Soltanian

Immobilization of hemoglobinon electro deposited cobalt-oxide nanoparticles: Direct voltammetry and electrocatalytic activity. ***Biophysical Chemistry 130(2007)122–131***

3- Abdollah Salimi, **Rahman Hallaj**, Hussein Mamkhezri, Saeid Soltanian,

"Electrochemical properties and electrocatalytic activity of FAD immobilized onto cobalt oxide nanoparticles: Application to nitrite detection***". Journal of Electroanalytical Chemistry 619–620 (2008) 31–38***

4- Rahman Hallaj, Abdollah Salimi, Keyvan Akhtari, Saied Soltanian, Hussein Mamkhezri, "Electrodeposition of guanine oxidation product onto zinc oxide nanoparticles:Application to nanomolar detection of l-cysteine". ***Sensors and Actuators B 135 (2009) 632–641.***

5- Abdollah Salimi, Hussein Mamkhezri, **Rahman Hallaj**, Saied Soltanian. Electrochemical detection of an ultratrace amount of arsenic (III) at glassy carbon electrode modified with cobalt oxide nanoparticles. ***Sensor and Actuators B 129 (2008) 246–254***

6- Fabrication of Sensitive Cholesterol Biosensor Based on Cobalt-oxide Nanostructures Electrodeposited onto Glassy Carbon Electrode , **Electroanalysis , 2009, 24, 2693-2700**.

7- Abdollah Salimi, **Rahman Hallaj** Begard Kavosi, Highly sensitive and selective amperometric sensors for nanomolar detection of iodate and periodate based on glassy carbon electrode modified with iridium oxide nanoparticles, ***Analytica Chimica Acta. 661(2010) 28-3***

8- Rahman Hallaj , Abdollah Salimi, Amperometric and voltammetric sensor for guanine detection based on electrodeposited cobalt oxide nano- structures modified glassy carbon electrode**. *Analytical methods.***

9- Abdollah Salimi, Rahman Hallaj, Amperometric flow injection analysis of insulin at cobalt-oxide nanomaterials modified glassy carbon electrode, ***submitted to*** Solid State Electrochemistry**.**

10- Abdollah Salimi, Keivan Akhtari, Saied Soltanian Rahman Hallaj, Electrochemical &Morphological Investigation of Cobalt-oxide Nanostructures Prepared by Potentiodynamic Deposition Method, **submitted to nanotechnology**.

Conference Papers;

1) Hussein Mamkhezri, **Rahman Hallaj**, Saied Soltanian ,Abdollah Salimi, Nanomolar detection of arsenic (III) at glassy carbon electrode modified with cobalt oxide nanoparticles. ***7th Biennial, Electrochemistry Seminar of Iran (7BESI) Urmia, Sept-9 2007.***

2) Abdollah Salimi, **Rahman Hallaj ,**Hussein Mamkhezri, Keyvan Akhtari, Saied Soltanian. Immobilization of guanine on ZnO nanoparticle: Electrochemical reversibility and electrocatalytic activity. ***7th Biennial electrochemistry Seminar of Iran (7BESI) Urmia, Sept-9 , 2007.***

3) Abdollah Salimi, **Rahman Hallaj**, Saied Soltanian Hussein Mamkhezri, , Novel amperometic sensor for iodate based on glassy carbon electrode modified with iridium oxide nanoparticles. ***7th Biennial electrochemistry Seminar of Iran (7BESI) Urmia, Sept-9, 2007.***

4) **Rahman Hallaj**, Abdollah Salimi, Hussein Mamkhezri, Saied Soltanian,. Direct voltammetry and electrocatalytic activity of immobilized FAD and Hemoglobin on glassy carbon electrode modified with cobalt oxide nanoparticle. ***7th Biennial electrochemistry Seminar of Iran (7BESI) Urmia, Sept-9, 2007.***

5) Abdollah Salimi, **Rahman Hallaj** Hussein Mamkhezri, Saied Soltanian. Nanomolar detection of hydrogen peroxide on glassy carbon electrode modified with electrodeposited cobalt oxide nanoparticles. ***7th Biennial electrochemistry Seminar of Iran (7BESI)Urmia, Sept-9 , 2007***

6) Abdollah Salimi , **Rahman Hallaj** , *Fabrication of biosensors based on immobilization of enzymes and proteins onto cobalt oxide nanoparticles,* The tenth word Congress of Biosensors, **14-16 May 2008, Shanghai, China**

7) Rahman Hallaj Abdollah Salimi**,** Keyvan Akhtari **,** Begard Kavosi**,** **"***Electrochemistry of immobilized FAD onto Ionic-Liquid / MWCNTs nanocomposite"* , 8th Iranian Biannual Seminar of Electrochemistry, **University of Kurdistan-Iran, 14-16 July 2009.**

8) Rahman Hallaj, Abdollah Salimi, Saied Soltanian *"Fabrication of sensitive cholesterol biosensor based on highly porous cobalt oxide nanostructures",* **8th Iranian Biannual Seminar of Electrochemistry, University of Kurdistan-Iran, 14-16 July 2009**

9) Rahman Hallaj , Begard Kavosi , Gobad Mansori, Abdollah Salimi, *"Electrocatalytic Reduction and Oxidation of nitrite at Glassy Carbon Electrode Modified with MWCNTs and Thodium Complex: Fabrication of Highly Sensetive and selective nitrite sensor***, Iranian Biannual Seminar of Electrochemistry, University of Kurdistan-Iran, 14-16 July 2009.**

10) Rahman Hallaj Abdollah Salimi, *Highly Sensitive Flow injection Insullin Sensor Based on Cobalt Oxide Nanoparticle Electrodeposited on Glassy Carbon Electrode.***16th Iranian Seminar of Analytical Chemistry, University of Bu- -Ali- Hamadan -Iran, 28-30 July 2009.**

Table of Contents

Chapter 1 5
Introduction: 5
1.1 General Background of Nanoscience and Nanotechnology 5
1.1 Application of Metal oxide 10
1.2 Synthesis of Metal Oxide nanostructures 11
1.3 Growing of nanostructures 14
1.4 Electrochemical metal deposition and Eelectrocrystalization 21
1.4 Electrochemical /chemical metal oxide deposition 30
1.5 Characterization methods 38
1.5.1 Scanning electron microscopy (SEM) 39
1.5.2 Transmission electron microscopy (TEM) 40
1.5.3 Energy dispersive X-ray spectroscopy 40
1.5.4 X-ray diffraction 41
1.5.5 Atomic force microscopy and scaning tunneling microscopy 42
1.6. Electrochemical applications of metal oxide nanostructure 49
1.7. Function of nanoparticle in electrochemical sensors 49
1.7.1 Catalysis of electrochemical reactions 51
1.7.2 Modified electrode, advantages & application of nanomaterials 52
1.7.3 Application of Metal oxide nanoparticles for modification of electrode 55
1.7.4 Modification of metal & metal oxide nanoparticles 58
1.7.5 Immobilization of biomolecules 65
1.7.7 Enhancement of Electron Transfer 68
1.7.8 Electrochemical sensors and biosensors based on 71
Refrences: ..... **Error! Bookmark not defined.**
Chapter 2 87
2. Preparation and Characterization of Cobalt, Zinc, and Iridium 88
2.1 Cobalt oxide Nanoparticles 93
2.2 Electrochemical deposition of cobalt oxides hydroxides nanostructures 95
2.2. Preparation of cobalt oxides by potentiodynamic technique 96
2.2.1 Experimental 96

2.2.2Preparation of the cobalt oxides by potentiodynamic electrodeposition..... 97
2.3. Study of Electrodeposition..... 97
2. 4. Characterization of Cobalt oxides nanostructures..... 102
2.4.1. Electrochemical characterization 102
2.4.1 a- Cyclic voltammetry..... 102
2.4.1 b- impedance spectroscopy 104
2.4.2 Structural characterization of cobalt oxides nanoparticles..... 106
2.4.3 XRD, AFM and SEM imaging microscopy..... 112
2.5. Electrocatalytic behavior of cobalt oxide nanostructures 112
2.5 Zinc oxide nanoparticles 118
2.5.1 Background 118
2.6 Experimental 120
2.6.1 Chemicals, reagents and Apparatus 120
2.6.2. Preparation of zinc oxide modified glassy carbon electrode..... 121
2.6.3 Characterization of zinc oxide nanoparticles 122
2.7 Iridium oxide nanoparticles..... 125
2.7.1 Background 125
2.8. Experimental 127
2.8.1 Chemicals, reagents and Apparatus 127
2.8.2 preperation and characterization of modified electrode..... 127
Refrences..... 132
Chapter 3 140
3. 1 Design and Fabrication of Sensors and Biosensors based on 146
Background 146
3.1 Experimental 148
3.1.1 Chemical and reagent..... 148
3.1.2 Apparatus 148
3.2 GC/Cobalt oxide modified electrode 150
3.2.1 Electrochemical characterization of modified electrode..... 150
3.2.2 Electrochemical behavior, pH effect and stability of modified electrode..... 155
3.2.3 Determination of Hydrogen Peroxide 159

Back ground 159
3.2.4 Electrocatalytic oxidation of H_2O_2 on GC/CoOx-NPs 160
3.2.5 Amperometric response of modified electrode 170
3.3.3 Determination of Arsenic 173
Back ground 173
3.3.4 Electrocatalytic Oxidation of Arsenic (III) on GC/CoOx-NPs 175
3.3.5 Amperometric Detection of As (III) at CoOx-NPs Modified Electrode 181
3.3.6 Interference effects and Analysis of As(III) in tap water 183
3.4. Determination of insulin 186
Background 186
3.4.1 Electrocatalytic Oxidation of insulin on GC/CoOx/NPs 188
3.4.2 pH dependence of catalytic activity on GC/CoOx/NPs 190
3.4.3 Amperometric and flow injection Detection of Insulin at the Modified Electrode 193
3.4.4 Stability and reproducibility of the modified electrode 196
3.4.5 Electrode response toward Interference species 198
3.4. Determination of Guanine 200
3.5.1 Electrocatalytic Oxidation Guanine on GC/CoOx-NPs 202
3.5.2 Kinetic of electrocatalytic oxidation of guanine on GC/Co-oxide/NPs- electrode 205
3.5.3 Amperometric detection of Guanine at GC/Cobalt-Oxide/NPs electrode 209
3.5.4 Stability and repeatability 212
Introduction 214
3.6 Immobilization of FAD onto CoOx-NPs 217
3.6.1 Electrochemical preparation of FAD/Cobalt oxide electrode 219
3.6.2 Investigation of GC/CoOx-NPs/FAD modified electrode 221
3.6.3 Electrocatalytic reduction of nitrite on GC/CoOx-NPs/FAD modified electrode . 227
3.6.4 Kinetic of electrocatalytic Reduction of Nitrite of GC/Co-oxide/NPs- electrode . 231
3.6.5 Amperometric detection of nitrite at FAD/cobalt oxide –np 233
3.7 Immobilization of Hemoglobin onto GC/CoOx-NPs modified electrode and its 236
3.7.1 Immobilization of Hemoglobin onto GC/CoOx-NPS 238
3.7.2 Charecterization of GC/CoOx-NPs/Hb 238
3.7.3. Direct Electrochemistry of hemoglobin at GC/CoOx-NPs 240

3.7.4 Electrochemical behavior of modified electrode in different pH..... 244
3.7.5 Electrocatalytic activiry of modified electrode toward reduction of hydrogen peroxide and oxygen 246
3.7.6 Amperometric detection of H_2O_2 at GC/CoOx-NPs/Hb electrode 250
3.7 Immobilization of Cholesterol Oxidase onto GC/CoOx-NPs 253
Background 253
3.7.1 Immobilization of ChOx onto cobalt oxide nanoparticles 256
3.7.2 Characterizations of GC/CoOx-NPs/ChOx modified electrode..... 256
3.7.3 Voltammetric response of GC/CoOx-NPs/ChOx electrode in the presence of cholesterol 260
3.7.4 Amperometric Detection of Cholesterol 263
Refrences..... 266
Chapter 4 280
Fabrication of Novel Sensors based on..... 280
Zinc Oxide & Iridium Oxide Nanoparticles..... 280
4.1 Electrodeposition of guanine oxidation product onto zinc oxide nanoparticles 286
Background 286
4.1.1 Immobilization of oxidation products of guanine onto ZnOx..... 289
4.1.2 Kinetic characterization of GC/ ZnO/8-oxo-guanine electrode 294
4.1.3 pH dependence of GC/ ZnO/8-oxo-guanine modified electrode 295
4.1.4. Eelectrocatalytic activity of GC/ZnO-8-Oxo-G toward 297
4.1.5. Amperometric detection of l-cysteine at GC/ZnOx-8-oxo-G electrode..... 302
4.2 Iridium oxide nanoparticles based sensor 305
Background 305
4.2.1 Electrochemical properties of GC/IrOx-NPs 307
4.2.2 electrocatalytic reduction of IO_3^- and IO_4^- at GC/IrOx-NPs modified electrode ... 308
4.2.3. Amperometric detection of iodate and periodate at the modified electrode 313
4.2.3 Interfaces effect and real sample analysis..... 321
4.3 Determinations of sugar and ethylene glycol..... 323
4.3.1 Cyclic voltammograms of GC/IrOx-NPs electrode in..... 326
4.3.2 Amperometric Detection of sugars at GC/IrOx-NPs electrode..... 329

4.3.3 Continuous Flow determination of reducing sugars 332
References 335
Chapter 5 341
5 . Carbon Nanotube/Rh-Complex based sensor 342
Background 346
5.1 Preparation of GC/Rh-Complex composite-modified electrode 350
5.2 Characterization of Rh-complex-modified CNT 351
5.3 Electrocatalytic properties of GC/CNT/Rh-Complex 356
5.4 Electrochemical Investigation of the Modified Electrode as a Sensor 359
5.5 Amperometric detection of nitrite 361
5.6 Interferences 364
5.7 Flow injection analysis 367
5.8 Determination of nitrite in food samples 371
References 375
Chapter 6 378
6. Conclusions 379

Chapter 1

Introduction To Nanostrucures;

Synthesis, Charecterization And Electrochemical Applications

چکیده فصل اول

مقدمه

کاربرد روزافزون نانوساختارها در عرصه های علم و تکنولوژی مرهون خواص منحصر به فرد ذرات در ابعاد نانو است. مواد در ابعاد نانومتری دارای تعداد اتمهای سطحی زیاد، هدایت الکتریکی بالا و خواص فیزیکی و شیمیایی متفاوت با توده ماده هستند. وجود این تفاوتها در نانوساختار ها موجبات بکارگیری گسترده آنها را فراهم کرده است. روشهای مختلفی برای تهیه نانوذرات بکار گرفته شده است که می توان به روشهای چون انباشت با پالس لیزر چگالش بخار ، روشهای شیمیایی و هیدروترمال و فرایندهای حالت جامد (همانند آسیاب کردن) و نیز روشهای الکتروشیمیایی اشاره کرد. عواملی نظیر غلظت، دما، pH ، زمان همزدن و ماهیت املاح بکار رفته بر تهیه نانوساختارها اثر گذار هستند، فرایند تشکیل ذرات از محلول در طی یک واکنش شیمیایی و یا الکتروشیمیایی اتفاق می افتد که می توان آنرا به دو مرحله متمایز شامل مرحله هسته زایی و مرحله رشد بلور تقسیم کرد. فرایند انتقال یونها از درون محلول به سطح الکترود و نیز سنتیک فرایند انتقال بار بین یونها و سطح الکترود بشدت خواص الکتریکی ، فیزیکی و شیمیایی ذرات حاصل را تحت تاثیر قرار می دهد. در روش انباشت الکتروشیمیایی فرایند هسته زایی با احیاء یونها و اجتماع و اتمهای حاصل اتفاق می افتد. از میان انواع نانوساختارها ، اکسیدهای فلزی و نانولوله های کربنی،

به دلیل خواص الکتریکی و قابلیت بر همکنش با مولکولها و زیست مولکولها، کاربردهای ویژه ای در الکتروشیمی و الکتروآنالیز گونه ها دارند. از آنجاییکه روش ساخت نانو ذرات فلزی و اکسیدهای فلزی تاثیر قابل توجهی بر خواص فیزیکی، شیمیایی و الکتروشیمیایی آنها دارد مطالعه این روشها و عوامل موثر بر آن بسیار مهم است. از میان روشهای متنوع ساخت نانو ذرات اکسیدهای فلزی، انباشت الکتروشیمیایی به دلیل سادگی روش، سازگار بودن با محیط و انجام پذیری در دمای پایین(کمتر از $200\ ^{\circ}C$) بیشتر مورد توجه است. به عنوان مقدمه ابتدا مکانیسم الکتروکریستالیزاسیون و اثر عوامل مختلف موثر بر آن و در نهایت تاثیر عوامل مختلف بر شکل ، اندازه، خواص فیزیکی ، شیمیایی و الکتریکی آنها مورد مطالعه قرار می گیرد. شناسایی، تعین ساختار و خواص نانوساختارهای تولید شده از مراحل بسیار مهمی است که توسط تکنیکهای مختلف تصویر برداری (میکروسکوپ الکترونی، میکروسکوپ نیروی اتمی)، تکنیکهای طیف سنجی(اسپکتروسکوپی نور مرئی ـ ماوراء بنفش) و نیز روشهای الکتروشیمیایی (اسپکتروسکوپی امپدانس و ولتامتری چرخه ای) انجام می شود، لذا مرور کوتاهی بر روشهای مطالعه نانوساختار ضروری به نظر می رسد. در ادامه کاربردهای مختلف این نانو ساختارها بخصوص کاربرد آنها در ساخت حسگرها و زیست حسگرها مورد مطالعه قرار می گیرد. نانوساختارهای اکسیدهای فلزی اغلب بطور مستقیم و به روش الکترونباشت و در سطح الکترود تولید می شوند. این نانو ساختار یا بطور مستقیم در فرایند انتقال الکترون

درگیر بوده و اکسیداسیون و احیای گونه ها در سطح الکترود را کاتالیز کرده و یا به دلیل قابلیت اصلاح توسط سایر مولکولها و زیست مولکولها به عنوان بستری برای استقرار و فعالیت این گونه ها مورد استفاده قرار گرفته اند. نتیجه بکارگیری این ساختارها در تهیه حسگرها و زیست حسگرها رسیدن به پارامترهای تجزیه ای مطلوب نظیر حد تشخیص پایین، حساسیت زیاد و محدوده خطی وسیع و فوق پتانسیل کم است. کارهای وسیعی در زمینه بکارگیری نانو ذرات در آنالیز مولکولهای مهم نظیر داروها، قندها، انسولین، تومورمارکرها ، DNA و نیز آنیونها و کاتیونهای مهم و آلاینده های زیست محیطی مختلف صورت گرفته است. تعداد روزافزون گزارشات در زمینه ساخت و بکارگیری نانو ساختارها نشان دهنده کارایی بسیار خوب آنها است. بنابراین ساخت نانوذراتی با کارایی های جدید و مطالعه خواص و رفتار آنها علاوه بر تولید علم و ایجاد زمینه های جدید پژوهش ، منجر به توسعه کاربرد نانو ساختارها در طراحی و ساخت حسگرها و زیست حسگرهای جدید با کارایی بالا می شود. نانو ساختارها در تهیه طیف وسیعی از حسگرها و زیست حسگرها و بر اساس خواصی مانند رنگ سنجی ، پلاسمون سطح ، فلورسانس ، خواص مغناطیسی و الکتروشیمیایی بکار رفته اند. سادگی تهیه، انتخاب گری و حساسیت بالا باعث توسعه کاربرد نانو ساختارها در تهیه حسگرها و زیست حسگرها شده است. در فصل دوم ساخت سه نوع از نانوذرات اکسیدهای فلزی شامل اکسید کبالت، اکسید روی و اکسید ایریدیوم مورد مطالعه قرار گرفته است. ساخت نانوذرات اکسید فلزات فوق به روش انباشت الکتروشیمیایی و در سطح الکترود کربن شیشه ای انجام شده است