

**SYNTHESIS AND CHARACTERIZATION OF VANADIUM
PENTOXIDE MICRO/ NANO-STRUCTURES AND ITS NO₂ GAS
SENSING APPLICATION**

HEMLATA



**DEPARTMENT OF PHYSICS
INDIAN INSTITUTE OF TECHNOLOGY DELHI
SEPTEMBER 2022**

© Indian Institute of Technology Delhi (IITD), New Delhi, 2022

**SYNTHESIS AND CHARACTERIZATION OF VANADIUM
PENTOXIDE MICRO/ NANO-STRUCTURES AND ITS NO₂ GAS
SENSING APPLICATION**

by

HEMLATA

Department of Physics

Submitted

in fulfillment of the requirements of the degree of Doctor of Philosophy

to the



INDIAN INSTITUTE OF TECHNOLOGY DELHI

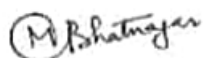
SEPTEMBER 2022

Certificate

This is to certify that the thesis entitled, “**Synthesis and Characterization of Vanadium Pentoxide Micro/ Nano-structures and its NO₂ Gas Sensing Application**”, being submitted by **Ms. Hemlata** to **Indian Institute of Technology Delhi** for the award of the degree of **Doctor of Philosophy** is a record of bonafide research work carried out by her under our guidance and supervision. She has fulfilled the requirements for the submission of the thesis, which to the best of our knowledge have reached the requisite standard and the results contained in this thesis have not been submitted in part or full to any other University or Institute for the award of any degree or diploma.

Date:

Place: New Delhi



Prof. Mukesh C Bhatnagar

(Retd. Professor)

Department of Physics

Indian Institute of Technology Delhi

New Delhi-110016

India



Dr. Pintu Das

(Associate Professor)

Department of Physics

Indian Institute of Technology Delhi

New Delhi-110016

India

Acknowledgements

In course of my Ph.D. time at Indian Institute of Technology Delhi (IIT Delhi) has been challenging as well as enjoyable. It is a gratifying aspect that now I have the opportunity to show my gratitude to all the people who contributed through their support and experience to accomplished this work.

First and foremost, I am highly grateful to almighty God by whose grace and blessing I would be able to achieve this academic endeavor in my life.

I would like to express my sincere gratitude to my thesis supervisors Prof. Mukesh C Bhatnagar and Dr. Pintu Das for their constant support and guidance. I am very thankful to Prof. Mukesh C Bhatnagar for his invaluable guidance, suggestions, encouragement and also for providing opportunity to do experimental work and to work on research ideas. I would especially like to mention that he provided a healthy and enthusiastic surrounding in the lab which kept us motivated in our research activities.

I would like to express my sincere thanks to my Ph. D. research committee members Prof. B. R. Mehta, Prof. Varsha Banerjee and Prof. Samarash Das for their insightful comments, valuable suggestion and guidance during the course of my research work.

I acknowledge the Department of Science and Technology (DST), Government of India, for providing me the research fellowship as an INSPIRE fellow. I am also thankful to Indian Institute of Technology Delhi, for providing me funding to attend the international conference from outside India. I also acknowledge the Nanoscale Research Facility (NRF) and Central Research Facility (CRF) of IIT Delhi for providing the experimental facility during the course of my research work.

I wish to put my deepest sense of regards and gratefulness to the Head of Department Prof. Ratnamala Chatterjee, all faculty members, laboratory and office staff of Physics Department, who helped me during the successful completion of my research work.

I would express my sincere gratitude and thanks to Prof. B. R. Mehta, Prof. Varsha Banerjee, Prof. Ratnamala Chatterjee, Prof. Sujeet Chaudhary, Prof. Pankaj Srivastava and Prof. Y. P. Sundriyal (HNBGU) who supported me in my difficult times, which are always remembered.

My sincere thanks to all my research group members Dr. Brajesh Nandan, Dr. Deshraj Meena, Dr. Abhishek, Dr. Mukesh Kumari and Mr. Habeebur Rahman for maintaining the friendly atmosphere in group for work and progress.

A deepest thanks goes to my friends at IIT Delhi who always supported me and gave me confidence when I was lacking it. I will never forget my friends Dr. Monika, Dr. Pratisha, Dr. Parul, Ms. Yogita, for spending good times and providing good company throughout my course of research work and also acknowledge Dr. Khushboo Agarwal and Mr. Rishabh Shukla for their useful discussions during my research work.

Finally, my deepest gratitude goes to my family members for their unconditional love and support throughout my life. The acknowledgement goes to my grandparents and parents for their love, affection, care, understanding and encouragement to do best in my life. I also like to express my deepest love to my sisters Mrs. Poonam Pokhriyal, Mrs. Geeta Nauriyal and Mrs. Anjana for their unconditional love and care in my hard time. I would like to gratefully acknowledge to my brother Mr. Vinod Dhoundiyal and Mr. Himanshu Dhoundiyal for giving me their valuable time to support me and motivate me in my difficult time. I want to express my sincere thanks to my brother-in-law Dr. Maheshanand Nauriyal Jeejaji and Mamaji Mr. Asharam Nautiyal for their valuable advice and guidance during my course of research work.


Hemlata

Abstract

Vanadium pentoxide (V_2O_5), a transition metal oxide has shown its great potential due to its specific physical and chemical properties. It is a promising material or metal oxide due to its semiconducting nature, optical, electrical and magnetic properties with thermal and chemical stability. Among the all transition metal oxides, V_2O_5 has gained huge research interest due to its layered structure, environment friendly nature, and cost effectiveness. In the present time, air pollution is the global problem for human being due to its abrupt increase because of its vast industrialization and fuel vehicle transportation. For controlling or monitoring the air pollutants, low cost and smart sensors are needed, as well as which can be operated at low working temperature. For this purpose, V_2O_5 shows the interesting results in the field of gas sensing. We set our first aim to find out the physical characteristics such as electrical and magnetic properties of hydrothermally synthesized microstructures to determine its charge carrier dynamics. After this work, we set our second aim to investigate the gas sensing application of V_2O_5 metal oxide with specified morphology such as micro flowers. Next, we compare the different morphology, micro flowers and nanoparticles to find out the best response for NO_2 gas sensing applications.

In the beginning of work, vanadium pentoxide V_2O_5 is successfully synthesized by hydrothermal method. The structural study explained the growth of prepared V_2O_5 sample in orthorhombic phase. The Raman study revealed the layered structure of V_2O_5 sample. Further, the morphological analysis of prepared V_2O_5 sample showed the flower like microstructures self-assembled by nanorods with diameter $\sim 3\mu m$. The crystalline nature of the microstructures was confirmed by SAED pattern. The electrical transport measurement confirmed the semiconducting nature of the V_2O_5 microstructure with activation energy 185 meV in the high temperature range 285 K to 380 K. whereas, the variable range hopping conduction is found in the low temperature

range 200-285 K. The experimentally observed behavior of V_2O_5 microstructure has been discussed in this work. It is evident from the M H plot that system shows the paramagnetic behavior along with small component of ferromagnetic behavior due to presence of V^{+4} oxidation state of vanadium. The magnetic characteristics of V_2O_5 system also showed its anomalous behavior in narrow temperature range 45-65 K, where the magnetic ground state is antiferromagnetic.

Subsequently, we prepared the V_2O_5 metal oxide sample by hydrothermal method with specific morphology for the gas sensing application, which is well known topic in the field of research in the recent time. The morphological analysis confirmed the flower like structures (micro flowers self-assembled by nanoplates). The structural study showed the orthorhombic phase and layered structure of V_2O_5 micro flowers. In addition, SAED pattern further confirms the polycrystalline nature of the micro flowers. The electrical transport analysis of micro flowers showed the semiconducting nature in the whole temperature range (170 - 380 K) with the activation energy 103 meV. In the gas sensing application part of V_2O_5 micro flowers, it illustrates the sharp increment in the resistance when the gas flow is switched on and reaches to the saturation value over 10 minute NO_2 gas exposure time. The sensing result also shows the better selectivity and sensitivity for the trace amount of the NO_2 gas at optimum operating temperature.

In the next part of the work, first we optimized the morphology of V_2O_5 metal oxide by hydrothermal synthesis method at different synthesis temperatures such as 150, 170, 190 °C (using the same synthesis precursors) to obtain the nanoparticle morphology of V_2O_5 . The structural characterization of all the optimized samples showed the orthorhombic phase formation of V_2O_5 sample with the layered structure. The morphological study confirmed the formation of nanoparticles at the temperature 190 °C with dimension of ~ 100 nm. The electrical transport study confirmed the semiconducting nature of the nanoparticles in the temperature regime 170 - 380 K.

with activation energy 100 meV. In the subsequent part of this work, both morphology V_2O_5 micro flowers and nanoparticles morphologies were compared in terms of their crystal structure, surface area, activation energy and further for NO_2 gas sensing applications. The sensing measurements were performed for 10 ppm NO_2 gas at 50 to 250 °C for 5 minute gas exposure time. The result showed the better response for micro flowers morphology as compared to nanoparticles and the reason was explained on the basis of morphology, the high surface area of V_2O_5 micro flowers (9.12 m^2/g) as compared to nanoparticles (2.19 m^2/g), calculated by BET analysis. Hence, our finding suggest that the V_2O_5 micro flowers is the promising candidate for high response NO_2 gas sensing application.

सार

वैनेडियम पेंटोक्साइड (V_2O_5), एक संक्रमण धातु ऑक्साइड ने अपने विशिष्ट भौतिक और रासायनिक गुणों के कारण अपनी महान क्षमता दिखाई है। इसकी अर्धचालक प्रकृति, धर्मल और रासायनिक स्थिरता के साथ ऑप्टिकल, विद्युत और चुंबकीय गुणों के कारण यह एक आशाजनक सामग्री या धातु ऑक्साइड है। सभी संक्रमण धातु ऑक्साइडों में, V_2O_5 ने अपनी स्तरित संरचना, पर्यावरण के अनुकूल प्रकृति और लागत प्रभावशीलता के कारण भारी शोध रुचि प्राप्त की है। वर्तमान समय में, वायु प्रदूषण अपने विशाल औद्योगीकरण और ईंधन वाहन परिवहन के कारण अचानक वृद्धि के कारण मानव के लिए वैश्विक समस्या है। वायु प्रदूषकों को नियंत्रित करने या निगरानी करने के लिए कम लागत और स्मार्ट सेंसर की आवश्यकता होती है, साथ ही कम काम करने वाले तापमान पर भी संचालित किया जा सकता है। इस प्रयोजन के लिए, V_2O_5 गैस संवेदन के क्षेत्र में दिलचस्प परिणाम दिखाता है। हमने अपना पहला लक्ष्य हाइड्रोथर्मली संश्लेषित माइक्रोस्ट्रक्चर के विद्युत और चुंबकीय गुणों जैसे भौतिक विशेषताओं का पता लगाने के लिए निर्धारित किया है ताकि इसके चार्ज वाहक गतिशीलता को निर्धारित किया जा सके। इस कार्य के बाद, हमने सूक्ष्म फूलों जैसे निर्दिष्ट आकारिकी के साथ V_2O_5 धातु ऑक्साइड के गैस सेंसिंग अनुप्रयोग की जांच करने के लिए अपना दूसरा उद्देश्य निर्धारित किया। इसके बाद, हम NO_2 गैस सेंसिंग अनुप्रयोगों के लिए सर्वोत्तम प्रतिक्रिया का पता लगाने के लिए विभिन्न आकारिकी, सूक्ष्म फूलों और नैनोकणों की तुलना करते हैं।

काम की शुरुआत में, वैनेडियम पेंटोक्साइड V_2O_5 को हाइड्रोथर्मल विधि द्वारा सफलतापूर्वक संश्लेषित किया जाता है। संरचनात्मक अध्ययन ने ऑर्थोरोम्बिक चरण में तैयार V_2O_5 प्रतिरूप की उत्पत्ति की व्याख्या की। रमन अध्ययन ने V_2O_5 प्रतिरूप की स्तरित संरचना का खुलासा किया। इसके अलावा, तैयार किए गए V_2O_5 प्रतिरूप के रूपात्मक विश्लेषण ने $\sim 3\mu m$ व्यास के साथ नैनोरोड्स द्वारा स्व-इकट्टे किए गए माइक्रोस्ट्रक्चर जैसे फूल दिखाए। माइक्रोस्ट्रक्चर की क्रिस्टलीय प्रकृति की पुष्टि SAED पैटर्न द्वारा की गई थी। विद्युत परिवहन माप ने उच्च तापमान रेंज 285 K से 380 K में सक्रियण ऊर्जा 185 meV के साथ V_2O_5 माइक्रोस्ट्रक्चर की अर्धचालक प्रकृति की पुष्टि की। जबकि, वेरिबल रेंज होपिंग चालन कम तापमान रेंज 200-285 K में पाया जाता है। इस कार्य में V_2O_5 माइक्रोस्ट्रक्चर के प्रयोगात्मक रूप से देखे गए व्यवहार पर चर्चा की गई है। एम एच प्लॉट से यह स्पष्ट है कि सिस्टम वैनेडियम के $V + 4$ ऑक्सीकरण अवस्था की उपस्थिति के कारण फेरोमैग्नेटिक व्यवहार के छोटे घटक के साथ-साथ पैरामैग्नेटिक व्यवहार को दर्शाता है। V_2O_5 प्रणाली की चुंबकीय विशेषताओं ने संकीर्ण तापमान रेंज 45-65 K में भी अपना विषम व्यवहार दिखाया, जहां चुंबकीय ग्राउंड की स्थिति एंटीफेरोमैग्नेटिक है।

इसके बाद, हमने गैस सेंसिंग एप्लिकेशन के लिए विशिष्ट आकारिकी के साथ हाइड्रोथर्मल विधि द्वारा V_2O_5 धातु ऑक्साइड प्रतिरूप तैयार किया, जो हाल के समय में अनुसंधान के क्षेत्र में प्रसिद्ध विषय है। रूपात्मक विश्लेषण ने फूल जैसी संरचनाओं (सूक्ष्म फूल स्व-इकट्टे नैनोप्लेट्स द्वारा) की पुष्टि की। संरचनात्मक अध्ययन ने V_2O_5 सूक्ष्म फूलों के ऑर्थोरोम्बिक चरण और स्तरित संरचना को दिखाया। इसके अलावा, SAED पैटर्न सूक्ष्म फूलों की पॉली क्रिस्टलीय प्रकृति की पुष्टि करता है। सूक्ष्म फूलों के विद्युत परिवहन विश्लेषण ने 103 meV सक्रियण ऊर्जा के साथ पूरे तापमान रेंज (170 - 380 K) में अर्धचालक

प्रकृति को दिखाया। V_2O_5 सूक्ष्म फूलों के गैस सेंसिंग एप्लिकेशन भाग में, यह प्रतिरोध में तेज वृद्धि को दर्शाता है, जब गैस प्रवाह चालू होता है और 10 मिनट NO_2 गैस एक्सपोजर समय से अधिक संतृप्ति मूल्य तक पहुंच जाता है। संवेदन परिणाम अनुकूलतम ऑपरेटिंग तापमान पर NO_2 गैस की ट्रेस मात्रा के लिए बेहतर चयनात्मकता और संवेदनशीलता को भी दर्शाता है।

काम के अगले भाग में, पहले हमने V_2O_5 के नैनोपार्टिकल आकारिकी को प्राप्त करने के लिए 150, 170, 190 °C (समान संश्लेषण अग्रदूतों का उपयोग करके) जैसे विभिन्न संश्लेषण तापमानों पर हाइड्रोथर्मल संश्लेषण विधि द्वारा V_2O_5 धातु ऑक्साइड की आकृति विज्ञान को अनुकूलित किया। सभी अनुकूलित नमूनों के संरचनात्मक लक्षण वर्णन ने स्तरित संरचना के साथ V_2O_5 नमूने के ऑर्थोरोम्बिक चरण गठन को दिखाया। रूपात्मक अध्ययन ने 190 °C तापमान पर ~ 100 nm के आयाम के साथ नैनोकणों के गठन की पुष्टि की। विद्युत परिवहन अध्ययन ने 170 - 380 K . के तापमान रेंज में नैनोकणों की अर्धचालक प्रकृति की पुष्टि की सक्रियण ऊर्जा के साथ 100 meV। इस कार्य के बाद के भाग में, दोनों आकारिकी V_2O_5 सूक्ष्म फूल और नैनोकणों आकारिकी की तुलना उनके क्रिस्टल संरचना, सतह क्षेत्र, सक्रियण ऊर्जा और NO_2 गैस संवेदन अनुप्रयोगों के लिए की गई थी। 5 मिनट के गैस एक्सपोजर समय के लिए 50 से 250 °C पर 10 पीपीएम NO_2 गैस के लिए सेंसिंग मापन किया गया था। परिणाम ने नैनोकणों की तुलना में सूक्ष्म फूलों के आकारिकी के लिए बेहतर प्रतिक्रिया दिखाई और इसका कारण आकारिकी के आधार पर समझाया गया था, नैनोकणों (2.19 मी²/ग्राम) की तुलना में V_2O_5 सूक्ष्म फूलों (9.12 मी²/ग्राम) का उच्च सतह क्षेत्र, BET विश्लेषण द्वारा परिकल्पित है। इसलिए, हमारी खोज से पता चलता है कि V_2O_5 सूक्ष्म फूल उच्च प्रतिक्रिया NO_2 गैस सेंसिंग एप्लिकेशन के लिए आशाजनक उम्मीदवार हैं।

Table of Content

Certificate	i
Acknowledgements	ii
Abstract	iv
Table of Contents	vii
List of Figures	xiii
List of Tables	xvii
List of Symbols and Abbreviations	xviii
Chapter 1: Introduction.....	1
1.1 Introduction.....	1
1.2 Metal Oxides: An overview	1
1.3 Electrical materials.....	3
1.3.1 Thermally activated band conduction.....	3
1.3.2 Variable range hopping conduction	5
1.4 Magnetic materials.....	6
1.4.1 Classification of magnetic materials.....	7
1.5 Nanomaterials: An overview.....	8
1.6 Vanadium Pentoxide.....	10
1.7 Synthesis methods of V ₂ O ₅	12
1.7.1 Chemical vapor deposition method.....	12
1.7.2 Physical vapor deposition method.....	13
1.7.3 Solution based method.....	14
1.8 Properties and applications of V ₂ O ₅	15

1.9 Gas sensor: An overview.....	16
1.10 Definitions.....	17
1.10.1 Sensitivity.....	18
1.10.2 Selectivity.....	18
1.10.3 Operating temperature.....	19
1.10.4 Response and recovery time.....	19
1.10.5 Repeatability.....	19
1.11 Selection of V ₂ O ₅ as a gas sensing material.....	19
1.12 Sensing mechanism.....	20
1.13 Influencing factor for gas sensing characteristic	22
1.13.1 Additional/ Supported material.....	22
1.13.2 Composite materials.....	24
1.13.3 Crystallite size, Surface area and Crystallographic shape	25
1.13.4 Operating temperature.....	25
1.14 Motivation.....	26
1.15 Objectives of the Thesis.....	27
1.16 Organization of the Thesis.....	28
1.17 References.....	31
2 Chapter 2: Synthesis and Characterizations Techniques	46
2.1 Introduction.....	46
2.2 Material preparation.....	46
2.2.1 Hydrothermal method.....	47
2.2.2 Chemical reagents utilized.....	48

2.2.3	Optimization of different morphology.....	49
2.2.4	Pellet formation.....	50
2.3	Characterization techniques.....	50
2.3.1	X-ray diffraction.....	50
2.3.2	Raman spectroscopy.....	53
2.3.3	Brunauer-Emmett-Teller.....	54
2.3.4	X-ray photoelectron spectroscopy.....	55
2.3.5	Scanning electron microscopy.....	57
2.3.6	Transmission electron microscopy.....	59
2.3.6.1	Imaging mode.....	60
2.3.6.2	Diffraction mode.....	60
2.3.7	Physical properties measurement system.....	62
2.3.8	Gas sensing set-up.....	64
2.4	References.....	65

Chapter 3: Physical properties of V₂O₅ microstructure formed by self-assembled nanorods
.....**68**

3.1	Introduction.....	68
3.2	Experimental section.....	70
3.2.1	Synthesis method.....	70
3.3	Results and discussion.....	70
3.3.1	X-ray diffraction and Raman analysis.....	70
3.3.2	X-ray photoelectron spectroscopy analysis.....	72
3.3.3	Morphological analysis.....	73

3.3.4	Growth mechanism of V ₂ O ₅ microstructure.....	74
3.3.5	Electrical transport analysis.....	75
3.3.6	Magnetic analysis.....	78
3.4	Conclusion.....	80
3.5	References.....	81
Chapter 4: Development of selective NO₂ gas sensor based on V₂O₅ micro flowers.....		86
4.1	Introduction.....	86
4.2	Experimental section.....	87
4.2.1	Synthesis method.....	87
4.2.2	Gas sensing characterizations.....	88
4.3	Results and discussion.....	88
4.3.1	X-ray diffraction and Raman analysis.....	88
4.3.2	Morphological analysis.....	90
4.3.3	Growth mechanism of V ₂ O ₅ micro flowers.....	91
4.3.4	Electrical transport analysis.....	91
4.3.5	Gas sensing results.....	92
4.3.6	NO ₂ gas sensing mechanism of V ₂ O ₅ micro flowers.....	96
4.4	Conclusion.....	98
4.5	References.....	98
Chapter 5: Synthesis and characterization of V₂O₅ nanoparticles and study the effect of morphology of V₂O₅ (nanoparticles and micro flowers) on NO₂ gas sensing.....		103

Part I: Optimization of V₂O₅ nanoparticles morphology and electrical transport properties of V₂O₅ nanoparticles.....	103
5.1 Introduction.....	103
5.2 Experimental section.....	104
5.2.1 Synthesis method.....	104
5.3 Results and discussion.....	105
5.3.1 X-ray diffraction and Raman analysis.....	105
5.3.2 Morphological analysis.....	107
5.3.3 Electrical transport analysis.....	108
5.4 Conclusion.....	109
Part II: Effect of morphology of V₂O₅ (nanoparticle and micro flower) for NO₂ gas sensing applications.....	109
5.5 Introduction.....	109
5.6 Experimental section.....	110
5.6.1 Synthesis method.....	110
5.6.2 Gas sensing characterization.....	111
5.7 Results and discussion.....	111
5.7.1 Morphological analysis.....	111
5.7.2 Brurauer-Emmett-Teller analysis.....	112
5.7.3 Gas sensing results.....	113
5.8 Conclusion.....	116
5.9 References.....	117

Chapter 6: Summary and future scope.....	120
6.1 Summary.....	120
6.2 Future scope.....	122
List of publications.....	123
List of Participation in Workshop and Paper Presented in National/ International Conferences.....	123
Author’s Bio-data.....	126

List of figures

Chapter 1

Figure 1.1	(a) Energy band diagram for n-type and (b) p-type semiconductor.....	4
Figure 1.2	Schematic diagram for Mott's variable range hopping conduction mechanism.....	6
Figure 1.3	Applications of metal oxides in different domains.....	8
Figure 1.4	Schematic Diagram of density of state versus Energy for 3D, 2D, 1D and 0D systems.....	10
Figure 1.5	Schematic representation of V ₂ O ₅ crystal structure.....	11
Figure 1.6	Process of chemical vapor deposition.....	13
Figure 1.7	Schematic diagram for physical vapor deposition method.....	14
Figure 1.8	Schematic band diagram for sensing mechanism on the surface of semiconducting metal oxide.....	22

Chapter 2

Figure 2.1	Schematic representation of the hydrothermal method.....	47
Figure 2.2	(a) Schematic illustration of X-ray diffraction from the parallel atomic planes, (b) schematic demonstration of X'Pert Pro X-ray diffractometer...	52
Figure 2.3	The transition diagram for Stokes and anti-Stokes Raman lines.....	54
Figure 2.4	BELSORP-max II BET surface area analyzer system installed at Central Research Facility at IIT Delhi (Sonipat Campus).....	55
Figure 2.5	Schematic diagram for generation of photoelectron.....	56
Figure 2.6	Schematic diagram of X-ray photoelectron spectrometer with various components.....	57

Figure 2.7	Schematic diagram of Scanning electron microscopy with the various component.....	58
Figure 2.8	Schematic diagram of Transmission electron microscopy (a) imaging mode and (b) diffraction mode.....	61
Figure 2.9	Photo of physical property measurement system at IIT Delhi (left), sample puck for transport measurement, VSM coil and VSM sample holder (right)	63
Figure 2.10	Linear contact for resistivity measurement.....	63
Figure 2.11	Schematic diagram of the experimental set up employed to study the gas sensing performance of V ₂ O ₅ metal oxides.....	65

Chapter 3

Figure 3.1	(a) XRD pattern of V ₂ O ₅ microstructure self-assembled by Nano rods and (b) Raman spectrum of V ₂ O ₅ microstructure.....	71
Figure 3.2	(a) Room temperature XPS spectra of O1s and V2p of V ₂ O ₅ microstructure (b) core-level spectrum of V2p of vanadium oxide microstructure, (c) O1s spectra of vanadium oxide microstructure.....	73
Figure 3.3	(a) SEM image, (b) TEM image and (c) SAED pattern of V ₂ O ₅ microstructure.....	74
Figure 3.4	(a) Resistivity versus temperature (T) plot, (b) Arrhenius plot for resistivity of V ₂ O ₅ microstructures.....	76
Figure 3.5	Plot for dc conductivity versus temperature (a) T ^{-1/2} , (b) T ^{-1/3} and (c) T ^{-1/4} with temperature range 200 -285K.....	77
Figure 3.6	(a) The magnetic field hysteresis (M-H) curve for V ₂ O ₅ at the various temperature range 5 K-350 K, the inset shows an enlarged view of M-H curve and (b) temperature dependence of the zero field cooled (ZFC) and the field cooled (FC) magnetization at an applied field of 1T in the V ₂ O ₅ microstructure.....	79

Chapter 4

Figure 4.1	Room temperature (a) XRD spectra of V ₂ O ₅ micro-flower annealed at 350 °C and (b) Raman spectra of V ₂ O ₅ micro flowers.....	89
Figure 4.2	(a) & (b) are the FESEM images of the V ₂ O ₅ micro-flower on different scale. (c) corresponding TEM image and (d) SAED pattern of V ₂ O ₅ micro-flower annealed at 350 °C.....	90
Figure 4.3	(a) Temperature dependent resistivity plot (b) Arrhenius plot for resistivity of V ₂ O ₅ micro flowers.....	92
Figure 4.4	Gas sensing characterization at the pellet surface of V ₂ O ₅ micro-flower towards NO ₂ gas (a) response of 10 ppm NO ₂ gas at 50 to 250 °C operating temperature, (b) response on exposure to 10 ppm NO ₂ gas at 200 °C, (c) Five different cycles of almost equal response for 10 ppm NO ₂ gas at 200 °C, (d) limit of Detection of micro-flower for NO ₂ gas...	94
Figure 4.5	(a) Selective response of V ₂ O ₅ micro-flowers for NO ₂ gas at 200 °C, (b) gas response for oxidizing (NO ₂) and reducing (H ₂) gases with n-type V ₂ O ₅ gas sensor at 200°C.....	95
Figure 4.6	Gas sensing chamber with heater sensor pellet assembly and electrical connections. (a) base current in zero air, (b) current in NO ₂ oxidizing gas, (c) current in H ₂ reducing gas.....	96

Chapter 5

Figure 5.1	(a) XRD pattern of V ₂ O ₅ nanostructure synthesized at different synthesis temperatures, (b) Raman spectra of V ₂ O ₅ nanostructure synthesized at different synthesis temperatures.....	106
Figure 5.2	(a-c) shows the SEM image of V ₂ O ₅ nanostructure synthesized at temperature 150 °C, 170 °C and 190 °C respectively. (d-e) shows the TEM image of V ₂ O ₅ nanoparticles and (f) shows the SAED pattern of the V ₂ O ₅ nanoparticles.....	107

Figure 5.3	(a) Resistivity versus temperature plot (b) Arrhenius plot for resistivity of V_2O_5 nanoparticles.....	108
Figure 5.4	FESEM images of the V_2O_5 structures on different scales, (a) and (b) are the nanoparticles and (c) and (d) are the micro-flowers.....	112
Figure 5.5	Nitrogen adsorption-desorption isotherm (a & b) and pore size distribution plot inset (a & b) of hydrothermally synthesized micro flowers and nanoparticles, respectively.....	113
Figure 5.6	(a) Response of 10 ppm NO_2 gas at 50 to 250 °C operating temperature for V_2O_5 nanoparticles and micro flowers, (b) response of 10 ppm NO_2 gas for both morphologies of V_2O_5 at 150 °C, (c) and (d) four different cycle of response for 10 ppm NO_2 gas at 150 °C for V_2O_5 nanoparticles and micro flowers respectively.....	115

List of Tables

Chapter 1

Table 1.1	Different types of magnetic material and their characteristic.....	7
Table 1.2	Typically used additional material in V ₂ O ₅ for detecting the various gas/ vapor.....	23
Table 1.3	Various composites of V ₂ O ₅ for detecting/sensing various gases at different operating temperatures.....	24

Chapter 2

Table 2.1	The information regarding the chemical reagents used in this thesis....	48
Table 2.2	Hydrothermally synthesized V ₂ O ₅ sample of different morphologies and corresponding experimental environment	49

Chapter 3

Table 3.1	Raman spectra with assignments of bands in V ₂ O ₅ sample.....	72
------------------	--	----

Chapter 5

Table 5.1	Structural parameter for V15, V17 and V19 samples of V ₂ O ₅	105
Table 5.2	Comparative Study of V ₂ O ₅ micro flowers and Nanoparticles synthesized by hydrothermal method and its NO ₂ (10 ppm) gas sensing response at optimum temperature 150 °C.....	114

List of Symbols and Abbreviations

Symbols

ΔE	Activation energy
a, b, c	Lattice parameters
d	Interplaner spacing
D	Crystallite size
e^-	Electron charge
E_a	Acceptor impurity ion energy level
E_B	Binding energy of electron
E_c	Energy of conduction band
E_d	Donor impurity ion energy level
E_F	Fermi energy
E_g	Band gap
E_k	Kinetic energy of electron
E_v	Energy of valance band
eV_{surface}	Potential barrier
H	Magnetic Field
h, k, l	Miller indices
k_B	Boltzmann constant
M	Magnetic moment
n	Integer
$N(E_F)$	Density of the state
R_a	Resistance of sensor in air
R_g	Resistance of sensor in target gas
S	Sensitivity
T	Temperature
W	Work function
α^{-1}	Localized length
β	Full width at half maximum

γ	Dimensionality factor
θ	angle
λ	wavelength
Λ_{air}	Thickness of surface charge layer
ρ	resistivity
σ	Conductivity
χ	Susceptibility

Abbreviations

0D	Zero dimensional
1D	One dimensional
2D	Two dimensional
3D	Three dimensional
BET	Brunauer-Emmett-Teller
BSE	Backscattered electron
CB	Conduction band
CVD	Chemical vapor deposition
DI	De-ionized
e-beam	Electron beam
FC	Field cooling
FEG	Field emission gun
FESEM	Field emission scanning electron microscopy
HRTEM	High resolution transmission electron microscopy
i-t	Current-time
JCPDS	Joint committee on powder diffraction
LOD	Limit of detection
MIS	Metal to semiconductor
MIT	Metal to insulator
MO	Metal oxide
MOCVD	Metal organic chemical vapor deposition

NMs	Nanomaterials
PLD	Pulse laser deposition
ppm	Part per million
PPMS	Physical property measurement system
PVD	Physical vapor deposition
RT	Room temperature
SAED	Selected area electron diffraction
SE	Scattered electron
SEM	Scanning electron microscopy
slpm	Standard liter per minute
TE	Thermal evaporation
TEM	Transmission electron microscopy
TMOs	Transition metal oxides
VB	Valance band
V-O	Vanadium oxide
VRH	Variable range hopping
VSM	Vibrating sample magnetometer
WCR	Wet chemical reaction
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction
ZFC	Zero field cooling