Synthesis, Characterization and Antimicrobial Study of Vanadium (V) oxide using Alkoxide Precursor

Adam Hassan Elhaj Yousif *
Associate Professor, Department of Chemistry, Faculty of Education, University of ALFashir, Sudan.

Nosaiba Abdalgadir Nusr Mohamed
Chemistry Lab, Department of Chemistry, Faculty of Education, University of ALFashir, Sudan.
*Corresponding author: Adamhassan43@gmail.com

DOI: https://doi.org/10.47285/isr.v2i1.61

Citation: Yousif, A. H. E., & Mohamed, N. A. N. (2021). Synthesis, Characterization and Antimicrobial Study of Vanadium (V) oxide using Alkoxide Precursor, International Science Review, 2(1), 1-6. DOI: https://doi.org/10.47285/isr.v2i1.61

Research Article

Abstract
The present study is aimed to synthesize V₂O₅ using V(OR)₃ precursor and study its anti-microbial activity. The V(OR)₃ was prepared by reaction of VCl₃ and isopropanol alcohol. The synthesized V(OR)₃ was hydrolyzed to yield black V(OH)₃ gel using concentrated HCl as gelling agent. Yellow V₂O₅ obtained by thermal treatment of V(OH)₃ gel at 110°C and 380°C. All products were characterized using FT-IR Spectroscopy method. IR spectral revealed to found V(OR)₃, V(OH)₃ gel and V₂O₅ were formed. The V₂O₅ has been tested in vitro against numbers of microorganisms, to assess their antimicrobial activity using disc diffusion method, according to the National Committee for Clinical Laboratory Standards Guidelines. The results showed that V₂O₅ is active against Staphylococcus aureus and Pseudomonas aeruginosa and partial active against Bacillus subtilis, Escherichia coli and Candida albicans. Antimicrobial Study of VO(OR)₃ is required.

Keywords: V₂O₅, antimicrobial activity, FT-IR Spectroscopy, V(OR)₃, Disc diffusion method.

1. Introduction
Vanadium is an essential trace element for different organisms. The increasing interest in coordination chemistry of Vanadium is derived from its well established chemical and biological function [1]. Medicinal chemistry has great important interest in design of metal complexes as potential diagnostic and therapeutic agent [2]. Popular drug design strategy involves transition metal ions incorporated in to compounds of known therapeutic value [3]. Vanadium complexes with organic ligands used in treatment many cases such as diabetes, tumors and cardiovascular dysfunction [4]. Vanadium dioxide already made from alkoxide V(OR)₄. However, these alkoxides rather difficult to synthesize and highly reactive to hydrolysis and oxidation. Vanadium (V) oxo alkoxides VO(OR)₃ would be much more convenient precursors. Vanadium pentaoxide (V₂O₅) obtained in air whereas reduced phases formed in the presence of hydrogen. The pure VO₂ phase is obtained when the film is heated for two hours at 5.
2. Materials and Methods

2.1 Preparation of vanadium isopropoxide:
0.25 g of vanadium trichloride and 25ml of isopropanol were placed put into 500ml round bottom flask fitted with reflux condenser. The content of the flask was heated and refluxed for six hours at 82°C with continuous stirring.

2.2. Preparation of vanadium hydroxide gel:
Prepared vanadium isopropoxide was transfer into clean 100ml beaker flask. Three drops of concentrated HCl were added and 10ml of deionized water 1ml over each 30 mints. The mixture was stirred at room temperature for seven 7 hours until the solution being gel. The gel was dried in oven at 110°C for five 5 hours.

2.3. Preparation of vanadium (V) Oxide:
The dried vanadium hydroxide gel was calcined at 380°C. The yellow film was obtained.

2.4. Testing of antimicrobial activity of vanadium (V) oxide:
The paper disc diffusion method was used to screen the antibacterial activity of vanadium (V) oxide and performed by using Mueller Hinton Agar (MHA). The experiment was carried out according to the National Committee for Clinical Laboratory Standards Guidelines (NCCLS, 1999). Bacterial suspension was diluted with sterile physiological solution to 10^8 cfu/ ml (turbidity = McFarland standard 0.5). One hundred micro liters of bacterial suspension were swabbed uniformly on surface of MHA and the inoculums was allowed to dry for 5 minutes. Sterilized filter paper discs (Whiteman No.1, 6 mm in diameter) were placed on the surface of the MHA and soaked with 20 µl of a solution of vanadium oxide. The inoculated plates were incubated at 37 °C for 24 h in the inverted position. The diameters mm of the inhibition zones were measured. The same steps followed for antifungal activity.

3. Results and Discussion:

3.1 Characterization of vanadium isopropoxide Using FT-IR Spectroscopy:
Vanadium (isopropoxide have been prepared by reaction of Vanadium trichloride and isopropanol. The result of FT-IR analysis was shown in figures 1 and table 1 respectively. The IR spectra for Vanadium isopropoxide show bands at 1161.07, 1128.28, 999.06, 950.84, 815.83 cm⁻¹. These results confirmed the formation of vanadium isopropoxide.

3.2 Characterization of vanadium hydroxide gel:
Vanadium hydroxide gel was prepared by hydrolysis of vanadium isopropoxide in presence of isopropanol as solvent. Hydrochloric acid was used as gelling agent. The gel was formed after seven 7 hours. The resulting gel was analyzed using FT-IR techniques. The result show in figure (2) and table (2). FT-IR showed bands at 891.05, 991.34 cm⁻¹ of (V-O) and 3379.05 cm⁻¹ of (O-H).

3.3 Synthesis of vanadium oxide from vanadium hydroxide gel by thermal treatments:
Vanadium oxide was obtained by thermal treatments of vanadium hydroxide gel. Thermal treatment involves drying and calcination. Then calcination converts the dried gel in to yellow oxide material. The obtained oxide material analyzed using FT-IR spectroscopy. The results confirmed the formation of vanadium oxide in which showed band at 1041 cm⁻¹.
Fig. 1. IR spectrum of the V(OR)₃ obtained by reacted VCl₃ with isopropanol at 82 °C.

Fig. 2. IR spectrum of V(OH)₃ gel obtained from hydrolysis of V(OR)₃ using HCl as gelling agent.
Table 1: Results of FTIR analysis of V(OR')₃ obtained by reaction of VCl₃ and isopropanol.

| Wave number cm⁻¹ | Chemical bond       | References |
|------------------|---------------------|------------|
| 3352             | O-H (stretching)    | [9]        |
| 2970             | C-H (stretching)    | [9]        |
| 1469             | C-H(bending)        | [9]        |
| 1379             | C-H (bending)       | [9]        |
| 1128-1161        | V-O (bending)       | [10]       |
| 815              | V-O (stretch)       | [10]       |

Table 2: Results of FTIR analysis of V(OH)₃ obtained by hydrolysis of V(OR')₃ using hydrochloric acid as gelling agent.

| Wave number cm⁻¹ | Chemical bond       | References |
|------------------|---------------------|------------|
| 3379             | O-H (stretching)    | [11]       |
| 1620             | C-H (bending)       | [9]        |
| 991              | V-O(bending)        | [10]       |
| 891              | V-O(stretching)     | [10]       |

Table 3: Results of FTIR analysis of V₂O₅ obtained by thermal treatment of V(OH)₃ gel.

| Wave number cm⁻¹ | Chemical bond        | References |
|------------------|----------------------|------------|
| 3433             | O-H (stretching)     | [9], [12]  |
| 1631             | O-H(deformation)     | [12]       |
| 1014             | V-O(bending)         | [10]       |
| 840              | V-O (stretch)        | [10]       |

3.4 Antimicrobial activity of vanadium (v) oxide:
Invitro antimicrobial activities of vanadium oxide was carried out using culture of four bacteria types, Bacillus subtilis and Staphylococcus aureus as gram positive. Escherichia coli and Pseudomonas aeruginosa as gram negative, one fungal Candida albicans. The test was using
The susceptibility of bacterial and fungal to test sample was determined by formation of zone of inhibition mm produced arrange of environmental anclinically pathogenic bacteria using (0.1mg/ml) concentration of test sample. The pictures and table (4) shows the inhibition zone (mm) of each bacterial and fungal.

![Fig.4. Qualitative assessment antimicrobial activity of vanadium oxide against pathogenic gram positive bacteria (A, B), gram negative bacteria (C, D) and Fungal (E).](image)

Note: (A) for Bacillus subtilis; (B) for Staphlyococcus aureus. (C) for Escherichia coli, (D) for Pseudomonas aeruginosa; (E) for Candida albicans

| Gram positive bacteria | Gram negative bacteria | fungal |
|------------------------|------------------------|--------|
| Bacillus subtilis      | Staphylococcus aureus  | Escherichia coli | Pseudomonas aeruginosa | Candida albicans |
| Inhibition Zone diameter(mm/mg sample) | 10 | 13 | 12 | 14 | 10 |

According to literature when the measuring zone < 9 mm, the tasted sample inactive; 9-12 mm, partially active; 13-18 mm, active; >18 mm, very active. The results from the table (4) show the vanadium (V) oxide is active with Pseudomonas aeruginosa and Staphylococcus aureus, and partially active with Bacillus subtilis, Escherichia coli and Candida albicans. These results were in good agreement with relevant literature [8], vanadium oxide was synthesized by direct reaction of vanadium metal and oxide target.

4. Conclusion
Vanadium (V) oxide (V$_2$O$_5$) was successfully prepared using vanadium isopropoxide precursor as indicated by results of FT-IR analyses. Its antimicrobial activity was investigated. The results show that vanadium (V) oxide is active against Staphylococcus aureus and Pseudomonas aeruginosa and partially active against Bacillus subtilis, Escherichia coli and Candida albicans.
Acknowledgements: We like to thanks the technicians of the laboratories of the Chemistry and microbiology department of plants at the National Research Center (Khartoum). We also thank the staff of Leather Technology Center at the Ministry of Industry, Industrial Consulting Division (Khartoum) for their help in experimental work.

Author Contributions: Both authors equally contributed to the study.

Conflict of Interest: The authors declare no conflict of interest.

REFERENCES
1. Qian S.S, Zhao X., Wang J., You Z., (2015). Synthesis and Crystal Structures of Benzohydroamate- Coordinated Vanadium(V) Oxo Complexes With Aroylhydrazone Ligands. Acta Chim. Slov., 62 (4) Pp:828-833.
2. Karabasannavar S.S., Aliollli P.R., Kalshetty B.M., (2017) Synthesis, Characterization, Antimicrobial, DNA cleavage, and Cytotoxicity studies of some Metal(II) complexes of tridentate Schiff Base ligand: 2- hydroxyl-3-(4-phenylthiazol-2-yl) semicarbazide methyl Benzoic Acid. Indian Journal of Pharmaceutical Education and Research, 51(4) Pp: 748-757.
3. Muruganandam L., Kumar K.K. and Balasubramanian K. (2013). Synthesis, Characterization, Antibacterial, Antifungal and Anticancer Studies of new Antimatabolite :N-[di(phenylamino)methyl]acetamide and Some of Its Inner Transition Metal Chelates. Chem. Sci. Trans. 2, 379-384.http://doi.org/10.7598/Cst 2013.
4. Rehder D. (2016). Implications of Vanadium in technical Applications and pharmaceutical Issues. Iorganica Chimica Acta , 455 Pp: 378-389
5. Livage J. (1998). Optical and electrical properties of vanadium oxide films deposited from alkoxides. Bol. Soc. Esp. ceram-vidrio. 37(2-3) Pp:8
6. Livage J., Guzman G., Beteille F. and Davidson P. (1997). Optical properties of sol-gel Derived Vanadium oxide films. Journal of sol-gel science and technology. (8)Pp: 857-865.
7. Michael E.A. Warwick and Binions R. (2014). Advances in thermo chromic Vanadium Dioxide films. Journal of Materials Chemistry A; 2 (10) Pp:3275-3292.
8. Wang J., Zhou H., Guo G., Cheng T., Mao X., Li J., and Zhang X. (2017). A functionalized surface modification with Vanadium nanoparticles of Various valences against implant-associated bloodstream infection. International Journal of Nano medicine. 12 Pp:3121-3136.
9. Hashem Y.G.A. (2017). Synthesis and characyerization of Cobalt Oxides using sol-gel process. M.Sc Thesis. ALfahir University. Pp:26-27.
10. Chen W., Mai L.Q., Peng J.F., and Xu Q. (2004). FTIR study of Vanadium oxide nanotubes from Lamellar structure. Journal materials science.(39)Pp:2625-2627.
11. Yosif M.M. (2017). Synthesis and characterization of Nickel alkoxides using sol-gel process. M.Sc Thesis; Alfaishir University. Pp:20-21.
12. Yosif A.H.E., and Eltoum M.S.A. (2020). Mechanistic Investigation of Sol-Gel Reactions Using Alkoxyilsilane Precursor.Chemical Science International Journal.29 (5); Pp: 25-31.