

SYNTHESIS AND CHARACTERIZATION OF VANADIUM BASED ELECTRODE FOR APPLICATIONS IN METAL-ION BATTERIES

Submitted in partial fulfillment of the requirements for the award of
Master of Science in Chemistry

by

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SATHYABAMA
INSTITUTE OF SCIENCE AND TECHNOLOGY
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DEPARTMENT OF CHEMISTRY

BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **GOMATHI V(39910006)** who carried out the project entitled **“SYNTHESIS AND CHARACTERIZATION OF VANADIUM BASED ELECTRODE FOR THE APPLICATIONS IN META-ION BATTERIES”** under my supervision from 01/11/2019 to 15/03/2020.

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DECLARATION

I, **GOMATHI V** hereby declare that the Project Report entitled “**SYNTHESIS AND CHARACTERIZATION OF VANADIUM BASED ELECTRODE FOR APPLICATIONS IN METAL-ION BATTERIES**” done by me under the guidance of **Dr.A. SHEIK MIDEEN, M.Sc., Ph.D.** Professor, Department of Chemistry, Sathyabama Institute of Science and Technology, Chennai – 600119, India is submitted in partial fulfillment of the requirements for the award of **Master of Science in Chemistry** in April 2021.

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ABSTRACT

Modern cities demand a reliable offer of electricity once and wherever it's needed, thus a key style for power generators feeding on noncontiguous, nonfuel renewable is putting in auxiliary battery-type energy storage systems (BESS). Batteries victimization zinc element ions in binary compound electrolytes square measure highly enticing because of low price and elemental abundance concerns, but attaining changeability at a high energy density and long operation is challenging. Layered vanadium oxides have been considered as a promising alternative for various battery systems owing to their large interlayer distance, high theoretical specific capacity, abundant distributions, and low cost.

Vanadium oxide (V_2O_5) is gaining popularity as a potential cathode material for aqueous zinc-ion batteries. As a result, this review has summarised traditional methods for the generation of V_2O_5 nanoparticles, such as the simple chemical method, Hummer's method for the synthesis of Graphene and the simple mechanical method for the synthesis of V_2O_5 /Graphene nanocomposites. The cathode V_2O_5 /Graphene nanocomposite is made by mechanically doping Graphene nanoparticles into V_2O_5 nanoparticles.

Characterization using various spectroscopic techniques is also discussed. XRD spectra values and graphs, FTIR peaks and graphs, EDX images and composition, and FESEM morphology are compared and examined for graphene nanoparticles, V_2O_5 nanoparticles, and V_2O_5 /Graphene nanocomposite. In the FESEM characterization of V_2O_5 nanoparticles, nanospheres are obtained.

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CHAPTER 1

INTRODUCTION

The value of grid-scale energy storage systems has been highlighted by the recent drive for renewable energy integration (ESSs). Frequency control and peak shaving operations can be used by ESSs to help balance the power grid. In order to optimise productivity, power supply should ideally balance demand. In fact, however, this is rarely the case because supply and demand fluctuates. For example, the intermittent existence of renewable energy sources like wind also causes power supply oscillations, even though some periods of the day necessitate high levels of power. ESSs can help to correct this disparity. ESSs, on the other hand, must be extremely sensitive to perturbations in order to handle such real-time shifts. To put it another way, high rate capability is essential for such responsiveness, as ESSs must be able to store extra power and discharge it quickly when needed. Furthermore, since ESSs are made up of several battery modules, a small fire that starts as a minor blaze can end up costing a lot of money while also wreaking havoc on the environment and/or human lives. As a result, batteries built for ESS applications must meet two main criteria: high power density and protection.

In this regard, aqueous batteries hold a lot of promise. To begin, substituting aqueous electrolytes for traditional organic electrolytes reduces the risk of fire. In the unfortunate event that a battery short circuits due to physical/electrical/chemical factors, the sudden exothermic reactions can result in a fire. This problem can be avoided by using aqueous electrolytes. Second, water has a much higher ionic conductivity than other organic solvents, typically by a factor of two to three. As a result, aqueous batteries are much more likely to be able to work at high rates.

While Na, K, Ca, and Mg-based batteries have been investigated, their volumetric capacities are inferior to Zn's, especially when comparing their metallic forms as battery anodes. Zn ions can potentially pass multiple charges per ion unit

because they are multivalent (MV). As a result, we will only address Zn ion–based chemistries in this study. Despite the fact that few reviews document electrode materials and their outputs for rechargeable aqueous Zn-ion batteries, a detailed understanding of ion storage mechanisms is still missing. We assume that a simultaneous understanding of various elemental chemistries in context with one another is necessary for effective implementation of MV-ion batteries. We present a review of electrochemical reactions for Zn-ion electrodes that have been published. We compare activation mechanisms for ion transport and the location of water in various crystal lattices to investigate specific challenges for electrode materials such as reduced cation mobility, spontaneous dissolution, weak cycling, O₂ interaction, unintended proton/hydronium co-insertion, ineffective electrode–electrolyte interface formation, and current-collector corrosion. We demonstrate how certain battery design parameters affect these challenges, with the aim of seeking optimized solutions or alternative strategies to boost the viability of Zn-ion aqueous batteries (ZIABs) BESS. [Huang et al., 2019]

Aqueous zinc (Zn) ion batteries (AZIBs) have recently emerged as a promising device that can reach today's challenging output standards, among a variety of candidates for aqueous batteries. Zn metal (anode), aqueous electrolyte (mildly acidic pH), a separator, and a (in)organic cathode material are the components of a standard AZIB. Aside from the aqueous electrolyte, the use of Zn metal as the anode is a special function of this device. In aqueous electrochemical systems, Zn has many advantages. To begin with, it is a resource that is not only readily available and relatively inexpensive, but it is also known for its non-toxicity and chemical stability in aqueous media as compared to alkali metals like lithium or sodium. Second, Zn is oxidized to Zn²⁺ without forming intermediate phases under mildly acidic conditions (pH 4–6) and exhibits a high over potential for the hydrogen evolution reaction (HER). Third, given the relatively small operating window beyond which gas (H₂ and O₂) evolution occurs in water, Zn has a redox potential of 0.76 V (in contrast to the typical hydrogen electrode) that is ideal for battery operation. Finally, in its metallic state, Zn has a high theoretical potential (820 mA h g⁻¹, 5854 mA h L⁻¹).

Aqueous zinc-ion batteries (AZIBs) have high protection, low cost and good rate capabilities and they have been gaining growing interest for large-scale energy storage applications. These characteristics make zinc a promising anode material.

Zn-ion intercalation batteries, one of many multivalent batteries, have gained a lot of attention as a potential replacement for Li-ion batteries in large-scale applications. The electrodes used in Zn-ion batteries are both environmentally friendly and cost-effective to produce. The chemistry of the electrolytes is also very basic. Efforts to develop new technologies have expanded in recent years. [Guduru et al., 2016]

When we discuss about Zn^{2+} ion-injecting rechargeable electrodes we will not address these systems because the electrodes for zinc–nickel batteries, alkaline Zn– MnO_2 batteries, or hybrid zinc aqueous batteries store other ions (rather than Zn^{2+}). The majority of the ZIAB electrode literature covers different polymorphs of manganese dioxide, hexacyanoferrates and vanadium compounds and for these electrodes, we discuss a few Zn^{2+} storage mechanisms. We also discuss key similarities, missing links in the reaction mechanism, and the role of water in the process. [Wang et al., 2019]

CHAPTER 2

LITTERATURE SURVEY

2.1 CATHODE MATERIAL:

Vanadium is found in small amounts in the earth's crust. It is found at an average concentration of 150 mg kg^{-1} in mineral ores and in the range of $3\text{--}300 \text{ mg kg}^{-1}$ in soil. For more than a century, vanadium pentoxide, the highest oxidation state 5+ of vanadium, has been identified. Sir Henry Roscoe, a British chemist, described the various oxidation states of binary oxides of vanadium and set the end-member “vanadic acid” V_2O_5 in his Bakerian lecture to the Royal Society in December 1867. Ditte, a French chemist, identified the first V_2O_5 gel in 1885. V_2O_5 is present naturally (50 percent) in flue-gas deposits from oil-fired furnaces and residues. Shcherbinaite V_2O_5 (orthorhombic crystal system) is located on the walls of volcanic fissures and navajoite is a mineral trihydrate $\text{V}_2\text{O}_5 \cdot 3\text{H}_2\text{O}$. (monoclinic structure). In lump shape, titaniferous magnetite ore contains around 1.5–1.7 percent vanadium pentoxide. [Fang et al., 2019]

2.2 ELECTROLYTE:

The aqueous zinc battery's electrolyte is neutral (or mild acid) aqueous solution and the majority of them contain soluble zinc salts like zinc sulphate, zinc nitrate, zinc acetate, or other organic acid salts. In reality, the mild electrolyte is already optimised compared to the alkaline electrolyte because the zinc anode has much better reversibility and a smaller dendrite. The decrease in pH value, on the other hand, will trigger hydrogen evolution during the charge phase, posing a challenge to improving columbic performance. In general, mild electrolytes, such as the most commonly used ZnSO_4 solution, have poor Zn stripping/plating columbic efficiency due to inherently reduced solubility. When compared to ZnSO_4 , Chen and colleagues found that 3 m $\text{Zn}(\text{CF}_3\text{SO}_3)_2$ electrolyte has lower polarization and higher columbic efficiency of Zn stripping/ plating. The bulky CF_3SO_3 anions are responsible for the superior properties. It reduces the number of water molecules surrounding Zn^{2+} cations and thus reduces

the redemption effect, making Zn^{2+} transportation, charge transfer and storage simpler. [Guduru et al., 2016]

2.3 GRAPHENE:

Nanoscience and nanotechnology are primarily concerned with the development, characterization, discovery, and use of nanomaterials. Carbon, one of the most abundant atoms on the planet, exists naturally in a variety of ways and as a part in a wide range of substances known as allotropes of carbon. The world's thinnest, fastest, and stiffest substance is graphene, a "wonder material." As well as being an outstanding heat and energy conductor. It is the fundamental constituent of all other essential allotropes. Because of its low cost, ease of access, and widespread ability to convert to graphene, graphene oxide (GO) is a hot topic. Another function that is highly desired is scalability. [Paulchamy et al., 2015]

2.4 OTHER CATHODES:

2.4.1 $\alpha\text{-MnO}_2$:

Despite the fact that some forms of MnO_2 are currently used in commercial alkaline batteries, they are gaining popularity as potential electrodes for Zn-ion intercalation batteries. The crystal structure of $\alpha\text{-MnO}_2$ has a tunnel structure (cryptomelane), and putting alkali, alkaline earth, and post-transition metals into these tunnel structures has long been a research subject. High discharge capacities with a realistic discharge potential of 1.3 V (vs. Zn/Zn^{2+}) at moderate currents and an energy density of 225 Wh/kg in 1 M aqueous ZnSO_4 electrolyte are two of the most important properties of $\alpha\text{-MnO}_2$ in intercalation batteries. However, long-term cycling causes power fading in these intercalation batteries, resulting in poor output at high current rates. Xu et al. recorded reversible intercalation of Zn^{2+} into the tunnel structures of $\alpha\text{-MnO}_2$, resulting in a specific capacity of 210 mAh/g, which is significantly higher than the 125 mAh/g capacity of Zn/MnO_2 alkaline batteries. They had a power retention of up to 30

cycles with a very high columbic efficiency at a slow current rate of C/20. The use of non-toxic, non-corrosive, and low-cost mild electrolytes with ample raw materials is one of the most appealing features of these batteries. Lee et al. looked into the process of Zn^{2+} intercalation into $\alpha\text{-MnO}_2$ nanorods in order to find out why their intercalation efficiency was degrading. This work discovered a reversible phase transition of $\alpha\text{-MnO}_2$ structures into a layered triclinic Zn-birnessite. The interaction of Zn^{2+} with the tunnel structures of $\alpha\text{-MnO}_2$ was found to be quite strong because of large electrostatic forces compared to Li^+ and Na^+ , and it has been observed that when Mn is intercalated into tunnel structures, it changes its oxidation state. The dissolution of unstable Mn^{3+} from the tunnel walls into the electrolyte was discovered through X-ray near edge absorption structure (XANES) tests, resulting in a phase transition from tunnelled to layered structure. The deposition of Mn on the Zn anode was not observed, but the Mn ions were seen to reversibly inject back into the layer structures of Zn-birnessite during the charging process. Due to excessive stresses produced during the phase transitions of Nanomaterials 2016, 6, 41 7 of 19 $\alpha\text{-MnO}_2$ and Zn-birnessite, electrodes became amorphous as the number of cycles increased, causing the capacity to fade. Lee et al., on the other hand, did not give any suggestions for improving the cycling behaviour of Nanomaterials 2016, 6, $\alpha\text{-MnO}_2$.

In addition to Ni-ion batteries, Xu et al. focused on creating Zn-ion intercalation batteries in 2015. They used supercapacitor research to perform both theoretical and experimental work with the goal of improving the capacitance and charge rate capacity of batteries while increasing the valence of ions in the electrolyte. With a binding energy ranging from 1.05 to 2.09 eV, their theoretical calculations predicted a thermodynamically desirable injection of Zn^{2+} into various lattice sites of the tunnel structure of $\alpha\text{-MnO}_2$. Their experiments backed up the theoretical modelling studies, resulting in a basic ability of around 220 mAh/g. However, unlike previous work on $\alpha\text{-MnO}_2$, they used 20 wt percent graphene as an active ingredient in their electrodes, along with 70 wt percent $\alpha\text{-MnO}_2$, which helped them achieve not only a slightly higher ability but also a higher energy density of 320 Wh/kg.

2.4.2 λ -MnO₂ :

Yuan et al. published an important paper in which they synthesised λ -MnO₂ by selectively leaching Li from LiMn₂O₄. They looked into the electrochemical efficiency of an aqueous ZnSO₄ electrolyte for use in Zn-ion intercalation battery electrodes. The oxidation and reduction reactions were determined to lead to the transformations of Mn³⁺/Mn⁴⁺ and Mn⁴⁺/Mn³⁺, respectively, and they showed an initial discharge capacity of 442.6 mAh/g with a coulombic efficiency of nearly 100 percent, but the capacity fell to 33.8 mAh/g at a current density of 408 mA/g.

2.4.3 Zinc Hexacyanoferrate (ZnHCF) :

The oxidation and reduction reactions were determined to lead to the transformations of Mn³⁺/Mn⁴⁺ and Mn⁴⁺/Mn³⁺, respectively, and they showed an initial discharge capacity of 442.6 mAh/g with a coulombic efficiency of nearly 100 percent, but the capacity fell to 33.8 mAh/g at a current density of 408 mA/g. A full cell configuration of Zn anode against ZnHCF, on the other hand, intercalated at 1.7 V (vs. Zn/Zn²⁺) with a capacity of 65.4 mAh/g, and this intercalation voltage was stated to be one of the highest for aqueous Zn-ion batteries. Based on the total mass of the active electrode materials, the specific energy density of these complete cells was estimated to be 100 Wh/kg. The cyclability studies also showed strong results, with up to 76 percent of initial capacity retained after 100 cycles, demonstrating the benefit of ZnHCF electrodes with high voltage for Zn-ion battery applications. The authors did not, however, include information on the pH of the electrolyte or potential explanations for the high voltage of the complete cell configuration.

2.4.4 Todorokite:

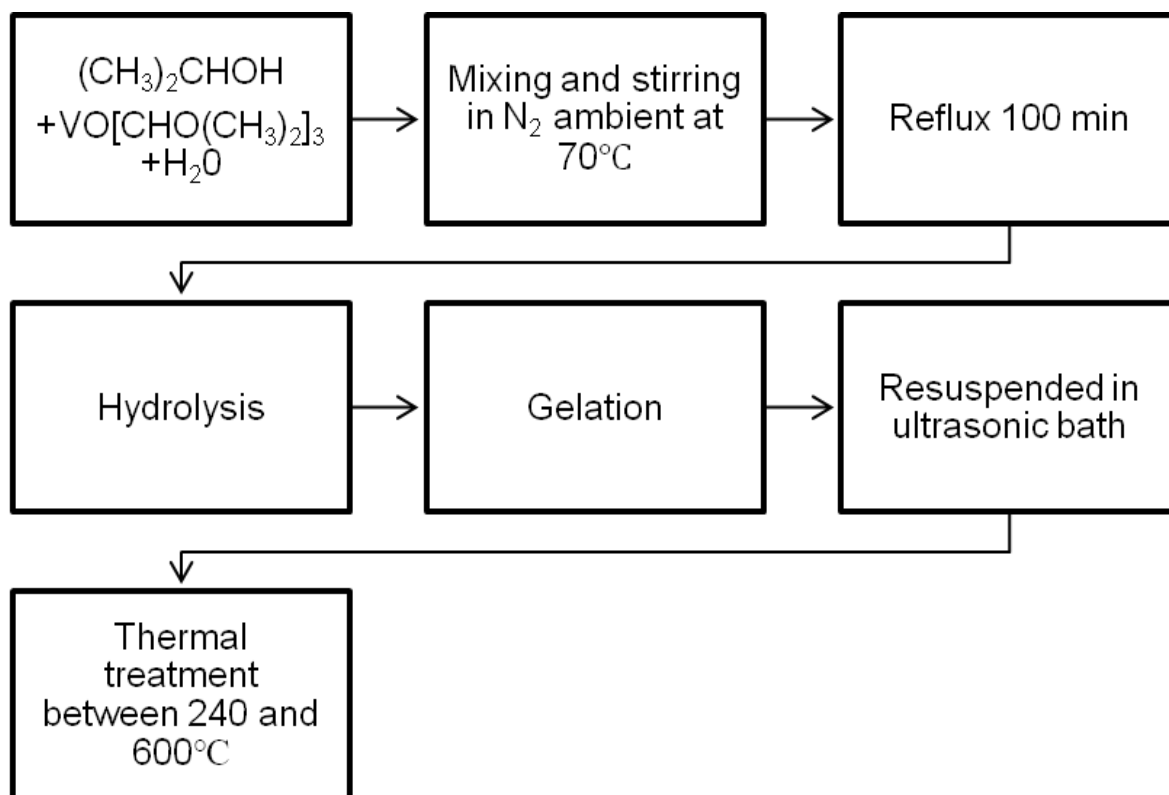
Todorokite is a manganese oxide (MnO_2) mineral with the chemical formula $\text{Mg}_{1.8}\text{Mn}_6\text{O}_{12} \cdot 4.8\text{H}_2\text{O}$ and a one-dimensional 33 tunnel structure. Many cations, including alkali, alkaline-earth, transition-metal ions, post-transition metals, and water molecules, will intercalate into this huge tunnel and stabilise the entire structure. Todorokite's tunnel structures are larger than those of -MnO_2 , allowing for easier Zn^{2+} diffusion. Furthermore, the crystallographic water within the tunnels is supposed to have a partial shielding effect on Zn^{2+} from the tunnel walls, reducing electrostatic interactions and Zn^{2+} bonding. A 2013, Lee et al. used a 1 M ZnSO_4 aqueous electrolyte with a Zn metal anode to investigate the intercalation of Zn^{2+} into a synthetic Todorokite. The electrochemical characterization revealed a basic capacity of 99 mAh/g, with cycle stability of 50 cycles. Todorokite, on the other hand, had a potential of less than half that of -MnO_2 (210 mAh/g), which was due to the presence of Mg^{2+} in the tunnel structures. Pre-intercalated Mg^{2+} is predicted to lower Mn's average oxidation state (AOS) to 3.4, as opposed to 3.67 in -MnO_2 . As a result, when compared to -MnO_2 , a lower AOS of Mn results in a lower amount of Zn^{2+} intercalation into Todorokite, resulting in a lower specific potential. Because of partial removal of pre-intercalated Mg^{2+} , the same Todorokite showed a higher capacity of 108 mAh/g in the second discharge period. Todorokite had a lower discharge capacity than -MnO_2 , but its capacity retention at high current rates was higher, due to the large 33 tunnel structures and partial shielding effect of crystallographic water in Todorokite tunnels. [Guduru et al., 2016]

2.5 METHODS:

2.5.1 SYNTHESIS OF V_2O_5 :

2.5.1.1 SOL-GEL SYNTHESIS:

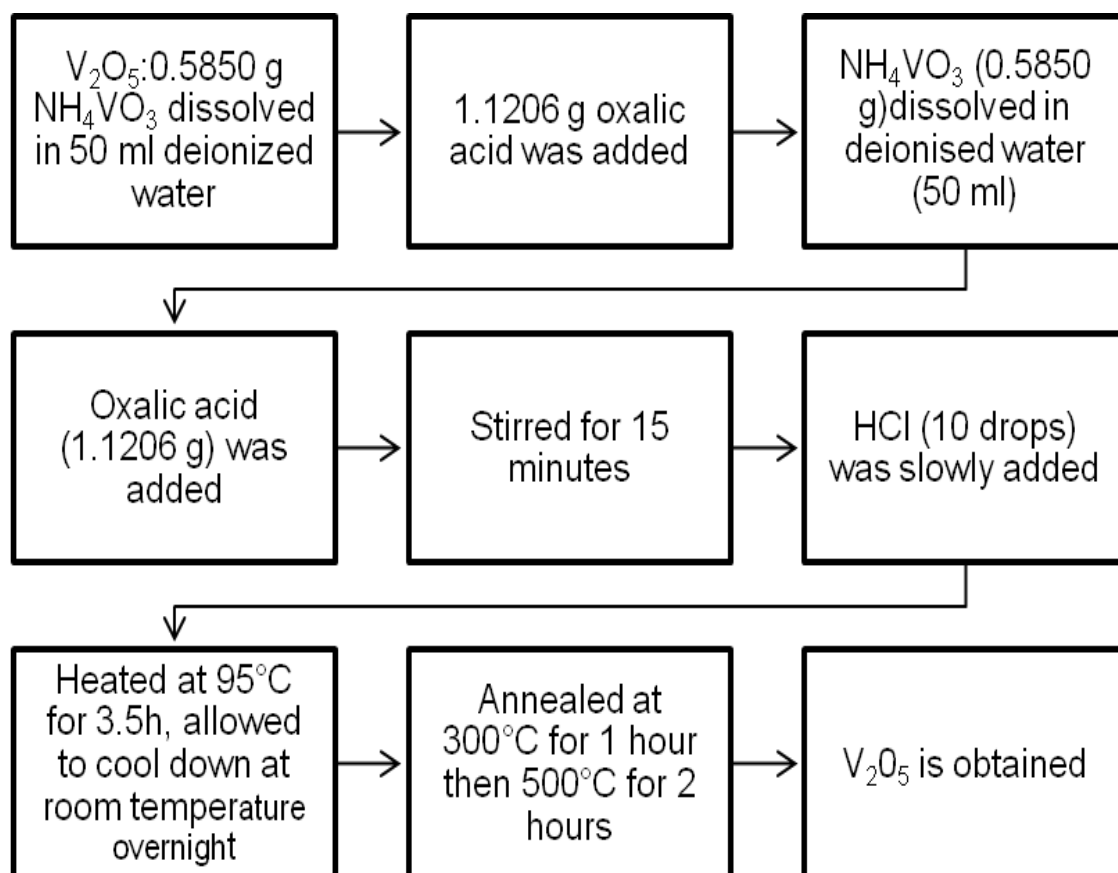
The synthesis was carried out in an oil bath with an all-glass assembly that included a round-bottomed boiling glass flask with four necks, a glass mixer, a water condenser, an inlet glass tube for N_2 bubbling, and one ground outer glass joint for reagent addition. The glass mixer was attached to the reaction flask's outer centre neck joint; it was made up of a specially built glass adapter that was mounted to the mixer and filled with silicon oil to avoid any interaction between air moisture and the reaction flask. Extra pure N_2 was purified using a pyrogallol trap, a concentrated H_2SO_4 bubbler, and two silica gel tubes before being bubbled into the reaction vessel. The experiment began with the addition of isopropanol (187 mL), vanadium (V)-triisopropoxide (25 g), and water in the following order: (25 ml). In a nitrogen atmosphere, the solution in the reaction vessel was refluxed and stirred continuously at 500–1000 rpm. The “frozen” gel was resuspended in an ultrasonic bath for 10 minutes after 100 minutes of hydrolysis at 70 °C to release trapped water. The resulting “liquefied gel” was poured into a Petri dish and dried at 60 degrees Celsius. [Liu et al., 2015]



2.5.1.2 HYDROTHERMAL METHOD:

A hydrothermal method was used to grow V₂O₅ nanoflowers on a glass substrate. The substrates were thoroughly washed with acetone and ethanol. The substrates were washed, then rinsed with deionized water and dried at room temperature. For V₂O₅, hydrothermal synthesis was used: 0.5850 g NH₄VO₃ dissolved in 50 ml deionized water, followed by 1.1206 g oxalic acid to form an orange solution. In deionized water, ammonium metavanadate, NH₄VO₃ (0.5850 g) was dissolved (50 ml). Oxalic acid (1.1206 g) was directly applied to the solution. Before adding 50 mL deionized water, the resulting solution was stirred for 15 minutes. After that, 10 drops of HCl were slowly applied to the resulting solution. The solution was put in a Pyrex glass bottle with a substrate at the bottom and heated to 95°C for 3.5 hours before being allowed to cool at room temperature overnight. V₂O₅ nanoflowers were observed uniformly deposited on the glass substrate after being rinsed extensively with deionized

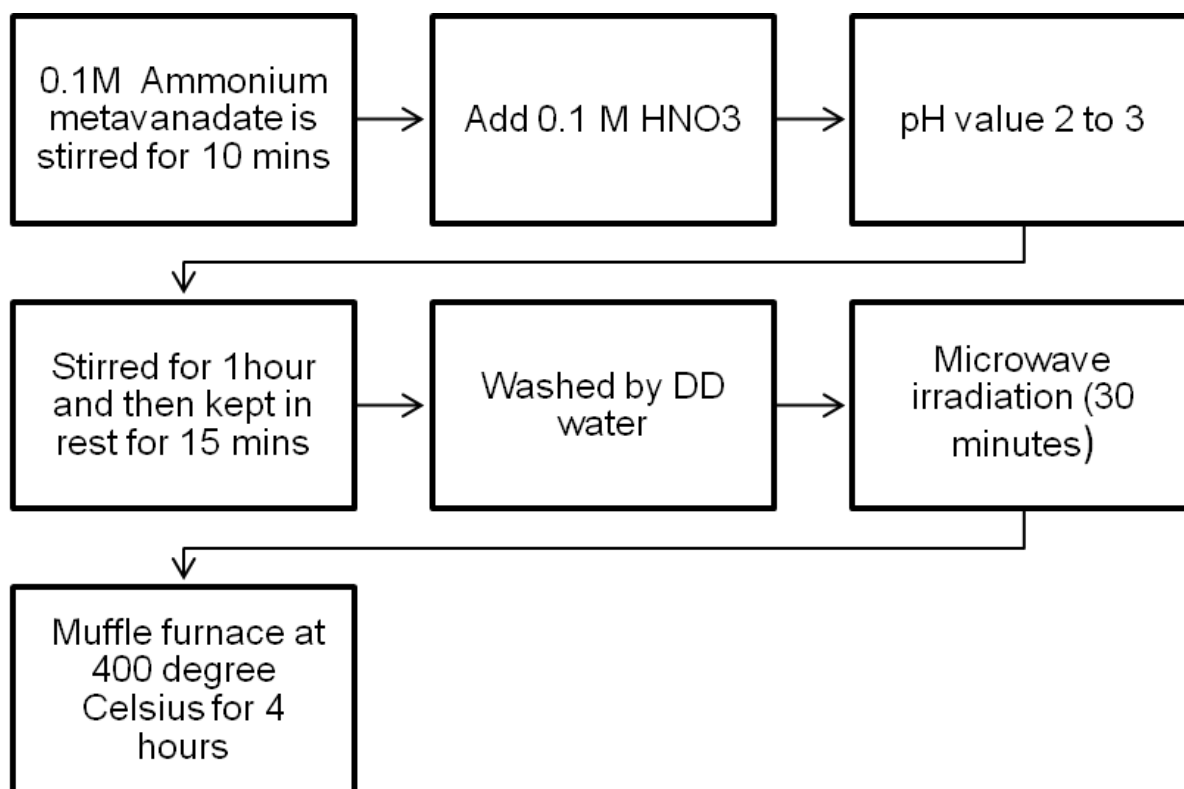
water and dried in ambient air. To build a V₂O₅ sheet, the samples were annealed at 300°C for 1 hour and then 500°C for 2 hours. [Abd-Alghafour et al., 2017]



2.5.1.3 SIMPLE CHEMICAL METHOD:

A 0.1 M solution of Ammonium Meta vanadate solution was prepared in a room atmosphere to synthesise vanadium oxide nanoparticles. After 10 minutes of stirring, 0.1 M nitric acid (HNO₃) was slowly applied to the solution to raise the pH level up to 2-3. The colour of the solution shifted to a deep red. The solution was continuously stirred for 1 hour under these conditions. The solution was then permitted to rest for 15 minutes in order to form the precipitate. The precipitate was extracted by sucking the unwanted residue solution and then slowly washed with DD water to remove the residue. Finally, the precipitate was put in a microwave oven to be irradiated (30 minutes) continuously. It was then dried on a hot plate to remove any excess water in the precipitate. After full

drying, the product was placed in a muffle furnace and heated for 4 hours at 400 degrees Celsius. [Liu et al., 2019]



2.5.2 SYNTHESIS OF GRAPHENE:

2.5.2.1 Hummer's method of synthesis:

The Hummers method was used to create graphene oxide by oxidising graphite. The following is a step-by-step guide to the preparation: In a 1000 mL volumetric flask, graphite flakes (2 g) and NaNO₃ (2 g) were mixed in 50 mL of H₂SO₄ (98 percent) with continuous stirring in an ice bath (0-5°C). At this temperature, the mixture was stirred for 2 hours, and potassium permanganate (6 g) was slowly added to the suspension. To keep the reaction temperature below 15°C, the rate of addition was carefully monitored. The ice bath was then removed, and the mixture was stirred at 35°C for 2 days until it became a pasty brownish colour after that, 100 ml water is slowly added to

dilute it. With effervescence, the reaction temperature was easily increased to 98°C, and the colour changed to brown. This solution was further diluted by adding 200 mL of water, which was continuously stirred. Finally, 10 mL H₂O₂ is added to the solution to bring the reaction to a close with the appearance of a yellow hue. The mixture was purified by rinsing and centrifuging with 10% HCl, followed by multiple washes with deionized (DI) water. The graphene oxide (GO) powder was obtained after filtration and drying under vacuum at room temperature.

2.5.2.2 Modified Hummer's method of synthesis:

Because of the thermal treatment of the solution, this modified process of synthesis requires both oxidation and exfoliation of graphite sheets. The procedure for stepwise synthesis is as follows: In a 1000 mL volumetric flask, graphite flakes (2 g) and NaNO₃ (2 g) were mixed in 90 mL of H₂SO₄ (98 percent) with continuous stirring in an ice bath (0-5°C). At this temperature, the mixture was stirred for 4 hours, and potassium permanganate (12 g) was slowly added to the suspension. To hold the reaction temperature below 15°C, the rate of addition was carefully monitored. The mixture is diluted with 184 mL water, which is slowly added and stirred for 2 hours. After that, the ice bath was removed, and the mixture was stirred for 2 hours at 35°C. For 10-15 minutes, the above mixture is held in a reflux machine at 98°C. Adjust the temperature to 30°C after 10 minutes for a brown-colored solution. After 10 minutes, raise the temperature to 25°C and keep it there for 2 hours. The solution is then treated with 40 mL H₂O₂, resulting in a bright yellow colour change. The mixture is then left undisturbed for 3-4 hours, after which the particles settle to the bottom and the remaining water is poured through a filter. The resulting mixture is centrifuged several times, first with 10% HCl, then with deionized (DI) water, until it forms a gel-like material (pH- neutral). The gel-like material is vacuum dried at 60°C for more than 6 hours after centrifugation to produce GO powder. [Arthi et al., 2015]

OBJECTIVES AND METHODOLOGY

1. To synthesis Graphene nanoparticle by Hummer's method.
2. To synthesis V_2O_5 nanoparticle by Simple Chemical method.
3. To synthesis V_2O_5 /Graphene nanocomposite by mechanical method.
4. To characterise Graphene nanoparticle, V_2O_5 nanoparticle and V_2O_5 /graphene nanocomposite by powder X-ray diffraction, FTIR and FESEM..

CHAPTER 3

EXPERIMENTAL

3.1 SYNTHESIS OF GRAPHENE NANOPARTICLE:

1. The mixture of graphite fine powder and NaNO_3 was taken in the ratio of 2:1, to this mixture 50 ml of con. H_2SO_4 added in 500 ml volumetric flask.
2. 9 g of KMnO_4 is added and kept in the ice bath ($10\text{-}15^\circ\text{C}$) for 2 hours with continuous stirring, Greyish colour appears.
3. Now the temperature is increased to 35°C and kept for 90 minutes and then left in the room temperature for overnight (ageing), brown colour appears.
4. Temperature is increased to 98°C for 60 minutes, add 100 ml of distilled water gradually in the interval of 15 mins (effervescence will be seen) and stirred continuously.
5. Allow the solution to settle in the room temperature and to the solution add 10-15 ml of H_2O_2 .
6. Filter the solution with 5% HCl and distilled water.
7. Dry the filtrate in the room temperature.
8. Graphene nano particle is obtained.

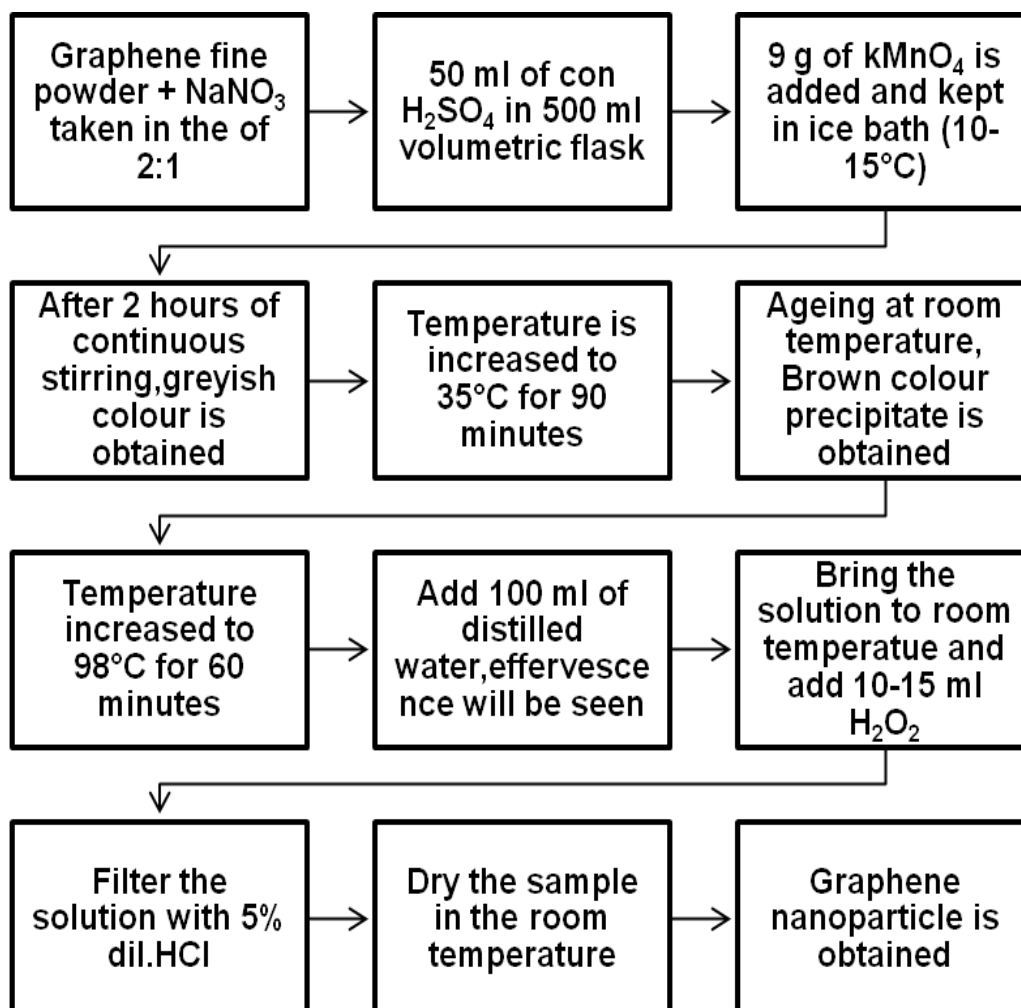


FIGURE 3.1 Synthesized graphene nanoparticle

3.2 SYNTHESIS OF V_2O_5 :

1. 1.2 g of ammonium vanadate mixed with ethanol and methanol was taken in 500 ml volumetric flask, several drops of acetic acid was added till the pH attained 3.5.
2. Stirred for 4 hours in 50°C .
3. Solution is evaporated at room temperature.
4. The solution is filtered in the filter paper and dried under the sunlight.
5. Calcined for 2 hours at 450°C .
6. V_2O_5 nanoparticle is obtained.

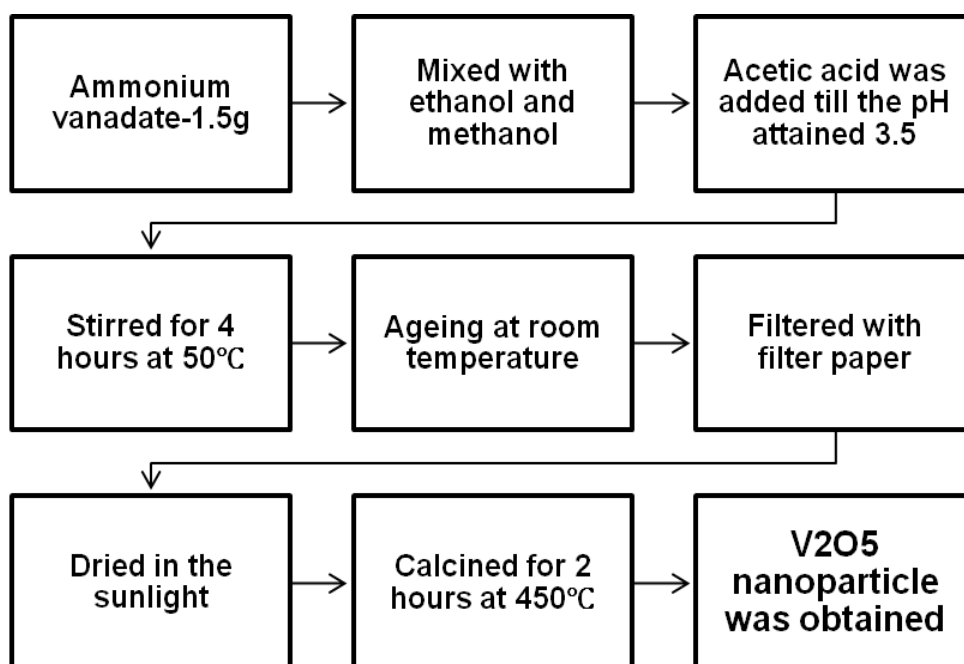




FIGURE 3.2 Synthesized V₂O₅ nanoparticle

3.3 SYNTHESIS OF V₂O₅/GO NANOCOMPOSITE:

1. Synthesized V₂O₅ and Synthesized Graphene were taken in 3:1 ratio.
2. In mortar and pestle the mixture is grinded well.
3. Mixture is kept in the muffle furnace for 4 hours at 500°C.
4. V₂O₅/Graphene nanocomposite was obtained.

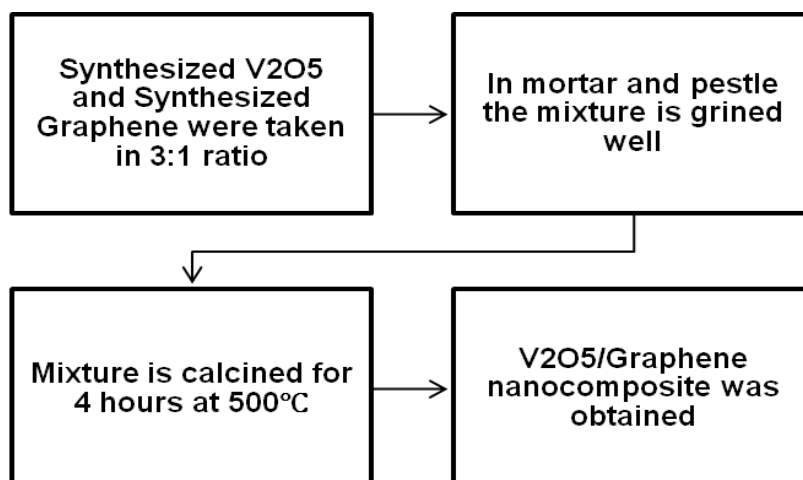


FIGURE 3.3: Synthesized V₂O₅/graphene nanocomposite

3.4 CHARACTERIZATION TECHNIQUES:

Different techniques can be used for the characterization of Graphene, V₂O₅ and the nano composites. In these studies, crystallographic information and purity of the phase is obtained by X-ray diffraction (XRD). Fourier transform infrared (FTIR) spectroscopy is used to identify the components that cannot be identified by XRD and to analyze the characteristic peaks. Field emission scanning electron microscope (FESEM) is used to study morphology of the nanoparticles.

CHAPTER 4

RESULT AND DISCUSSION

3.4 CHARACTERIZATION OF NANOPARTICLE:

4.1.1 *FOURIER TRANSFORM INFRARED SPECTROSCOPY(FTIR) ANALYSIS*

Fourier transform infrared (FTIR) spectroscopy analysis used to displays the FTIR transmittance spectra of V_2O_5 , GO, and VGO, as well as the vibration modes associated with each. Peaks about 3700 cm^{-1} in V_2O_5 , GO, and VRGO can be attributed to the bending vibration mode of H–O–H bonds caused by adsorbed water molecules during pellet handling. The O–H group vibrates in a stretching motion. Due to the intermediate product VGO, O–H and H–O–H motifs which appear in VGO. The FTIR spectrum of GO, which was used as a precursor in the experiment and exhibits frequency wise interrelated bands with VGO, was reported to confirm this. Variation in oxygen motif transmittance per centage confirms GO reduction. V=O stretching mode occurs around 1116 cm^{-1} in pure V_2O_5 . V–O–V stretching mode is represented by peaks at 843 cm^{-1} . In the case of V–O–V have typical peaks at 633 cm^{-1} , respectively. A strong interaction between V_2O_5 particles and the GO layer is indicated by the downshift in peaks. (Bhaskaram et al., 2016)

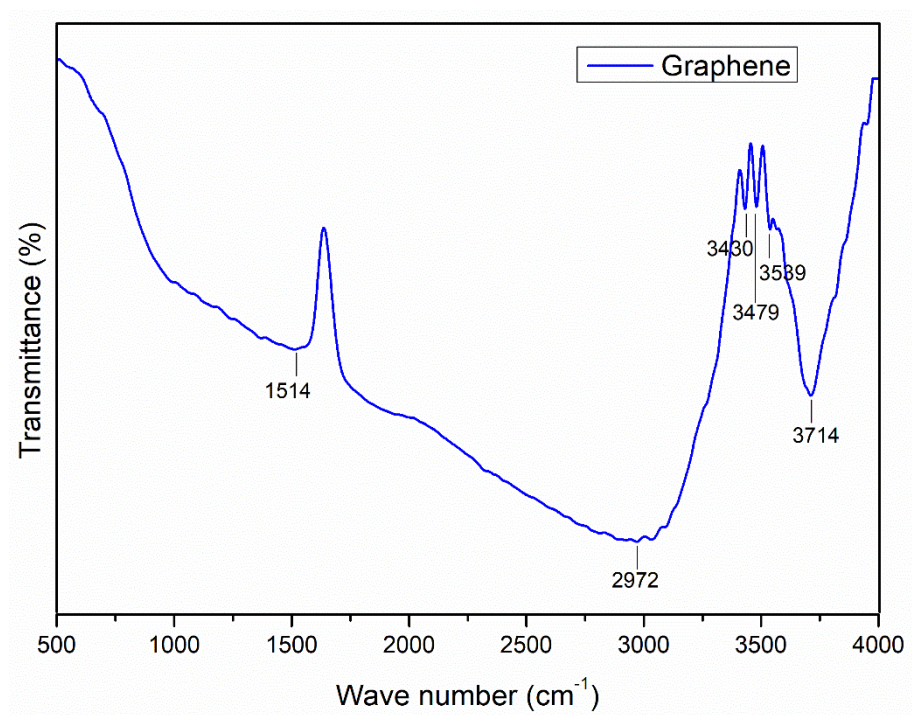


Figure 4.1: FTIR SPECTRUM OF GRAPHENE

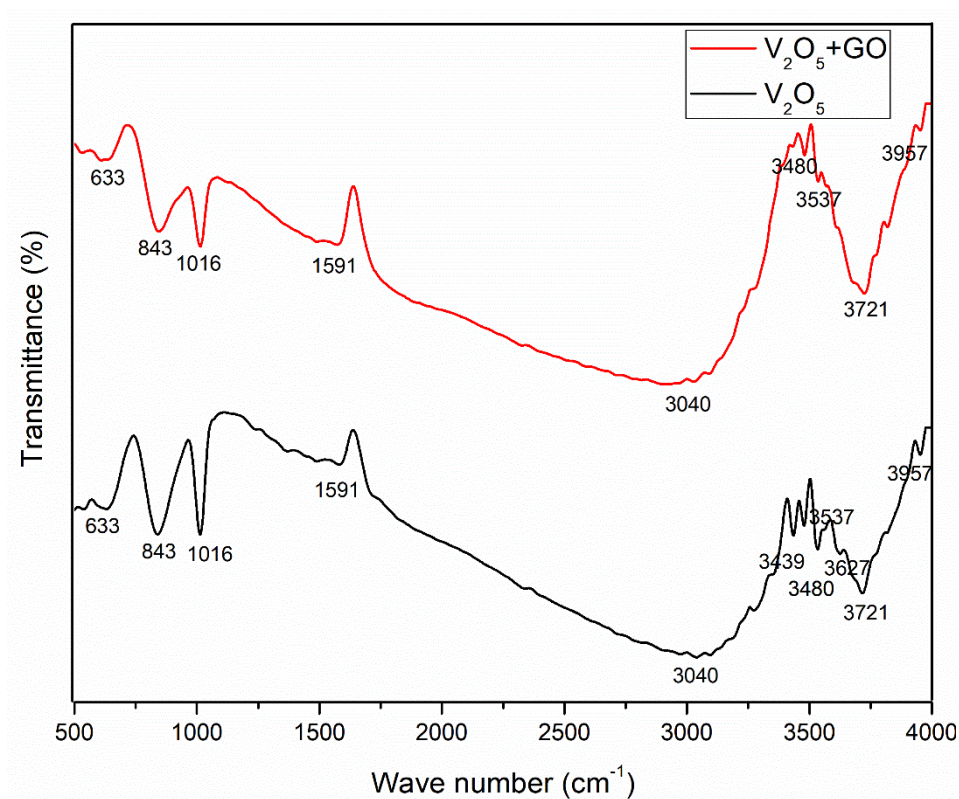


Figure 4.2: FTIR Spectra of V_2O_5 and V_2O_5+GO

VIBRATIONAL FREQUENCY	FUNCTIONAL GROUP
1016 cm^{-1}	V=O stretching
843 cm^{-1}	V-O-V stretching
633 cm^{-1}	V-O-V peak
1591 cm^{-1}	C=Cpeak
3040 cm^{-1}	C-H stretching
3040- 3957 cm^{-1}	Water -OH stretching

TABLE 4.1 VIBRATIONAL FREQUENCY IN FTIR SPECTRA V_2O_5 AND VGO

4.1.2 X-RAY DIFFRACTION ANALYSIS:

The XRD patterns of pure V_2O_5 nanoparticles as prepared, VGO after mechanical method, and GO are seen. The orthorhombic structure of V_2O_5 is responsible for the peaks. The diffraction angles and lattice planes obtained are shown in the table 4.2. The favoured growth direction was in the direction of intense and sharp peaks, indicating strong crystallinity. (Bhaskaram et al., 2016)

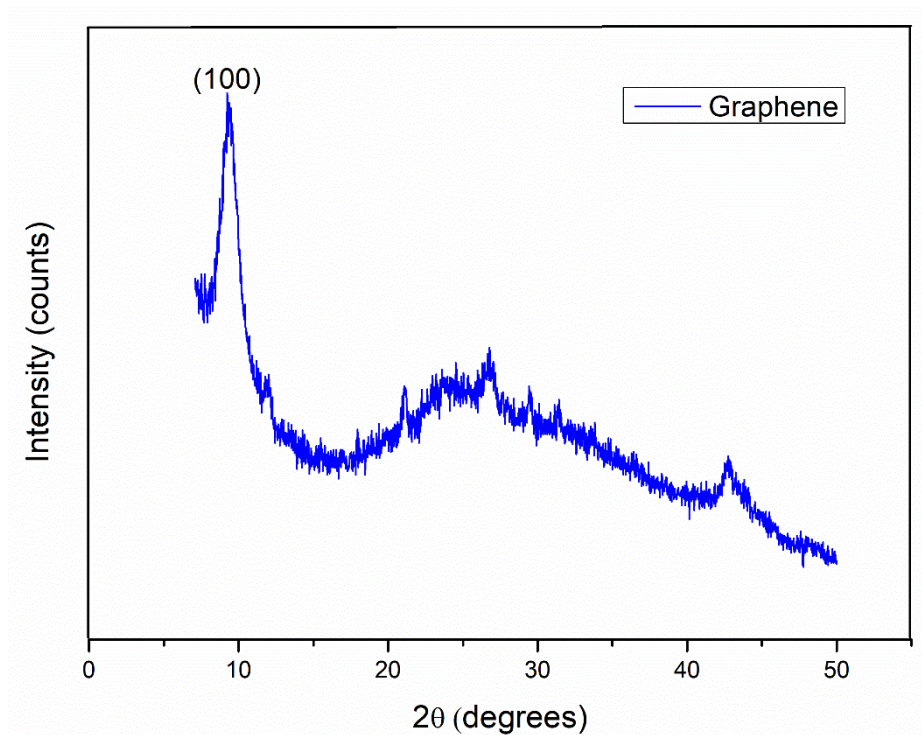


Figure 4.3: XRD Pattern of Graphene

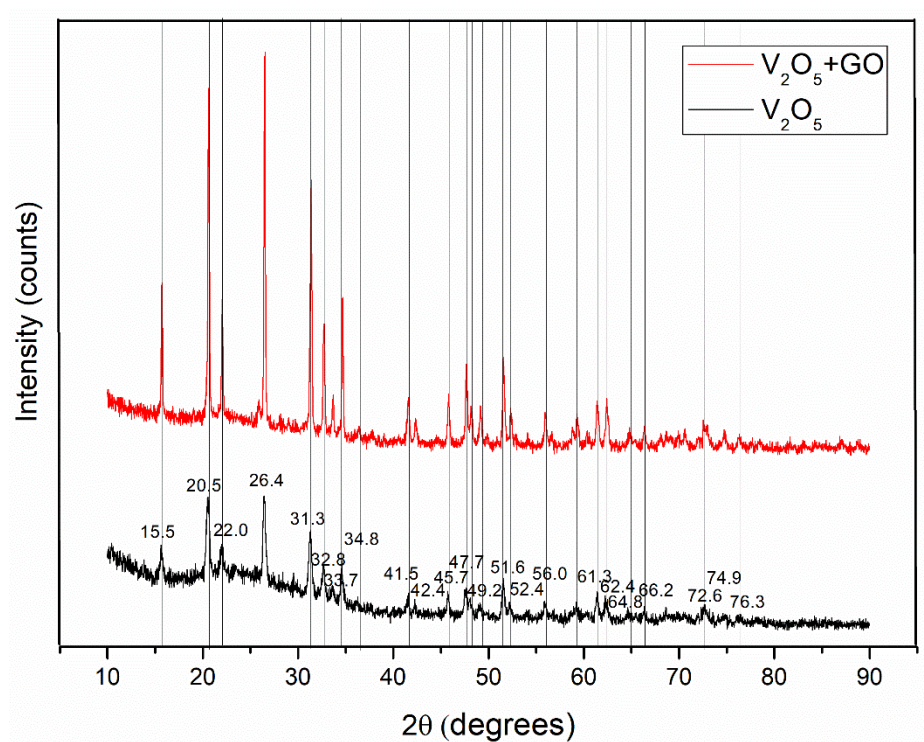


Figure 4.4: XRD Patterns of V_2O_5 and $V_2O_5 + GO$

OBTAINED DATA	REFERENCE	
2θ	2θ	LATTICE PLANE
15.5°	15.5°	(200)
20.5°	20.5°	(310)
26.4°	26.4°	(211)
31.3°	31.3°	(301)
41.5°	41.5°	(411)
51.6°	51.6°	(600)
66.3°	66.3°	(521)

TABLE4.2 X-Ray Diffraction analysis 2θ values obtained from graph and the reference values

4.1.3 Field emission scanning electron microscopy:

Scanning electron microscopy (SEM) has been carried out to study the morphology of V_2O_5 nanostructure and VGO. Microscope images of all the samples are shown. The V_2O_5 prepared by simple chemical method contains nearly spherical nanoparticles, with individual particle size not exceeding 200 nm. There is no agglomeration detected and a uniform repeated pattern indicates porosity. While wrapping V_2O_5 with GO using the mechanical method, the hydrophilic nature of GO allows homogeneous dispersion of V_2O_5 particles along GO layers. Smaller particles coalesce in order to reduce the surface energy, forming an elongated network of rodlike particles wrapped with GO. There exist a good contact between V_2O_5 particles and GO. This provides a conducting network for carrier transport and act as host for ions and electrolyte with large interface area. (Bhaskaram et al., 2016)

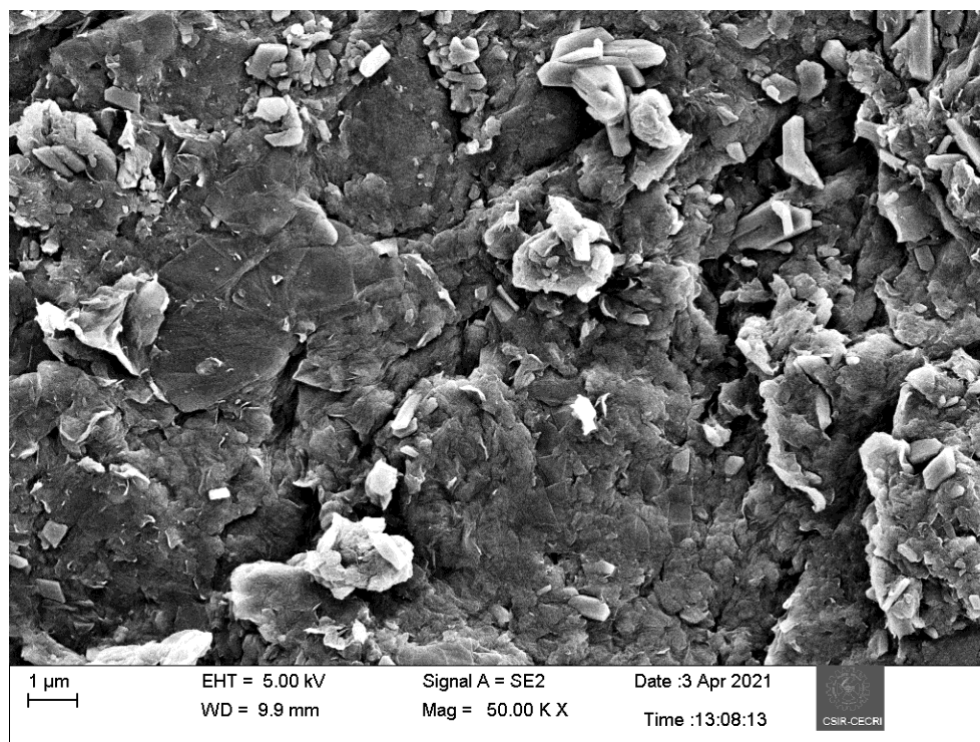


Figure 4.5: FESEM IMAGES OF GRAPHENE

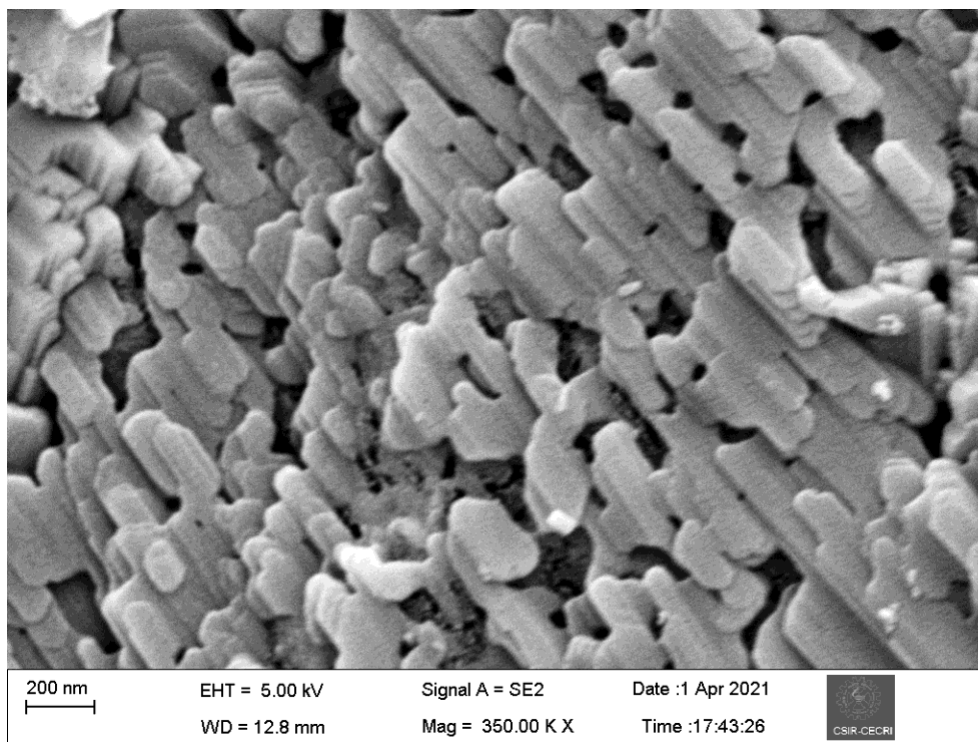


Figure 4.6: FESEM IMAGES OF V_2O_5

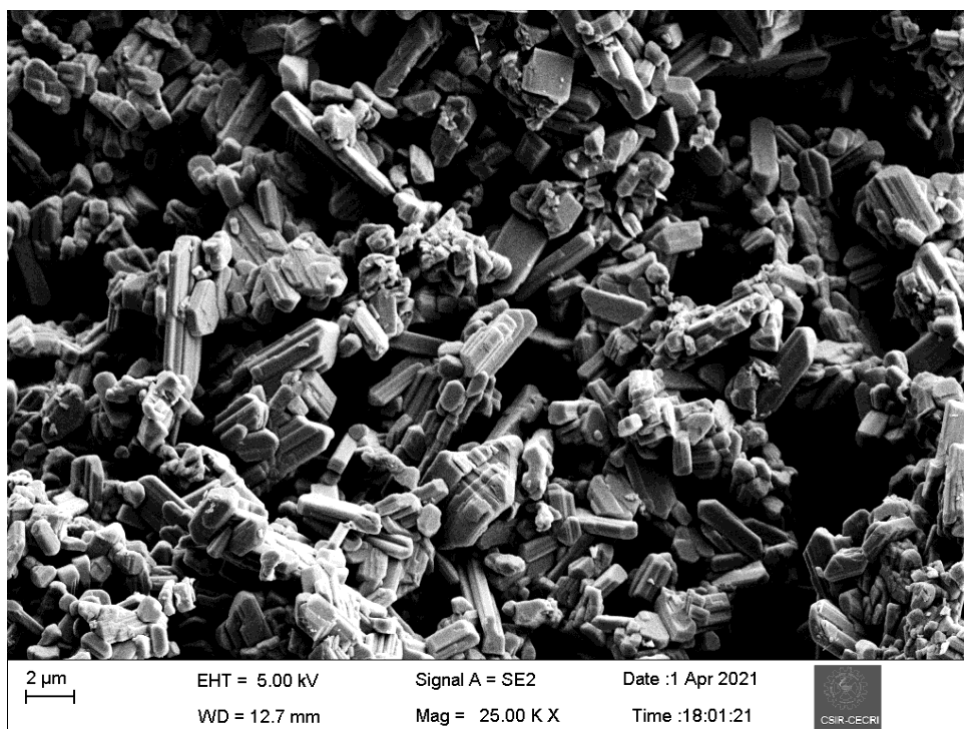
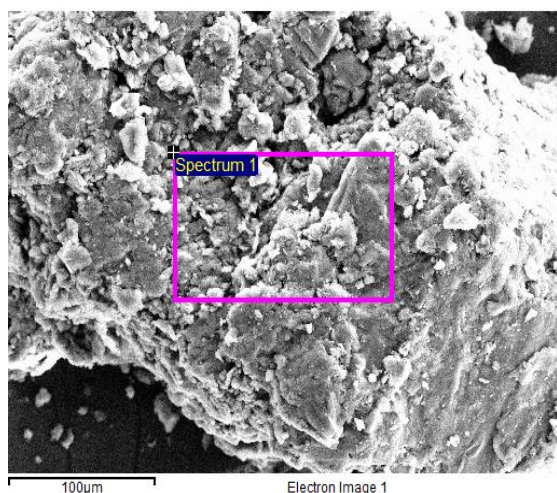


Figure 4.7: FESEM IMAGES OF V_2O_5 and VGO

4.1.4 ENERGY DISPERSIVE X-RAY(EDX) ANALYSIS:

4.1.4.1 GRAPHENE:

EDX analysis of the sample depicted in Figure 4.4.1.2 exhibits that the final product comprises of only C and O, suggesting that the product is Graphene



Element	Weight%	Atomic%
C K	59.41	66.09
O K	40.59	33.91
Totals	100.00	

Figure 4.8: EDAX IMAGES OF GO

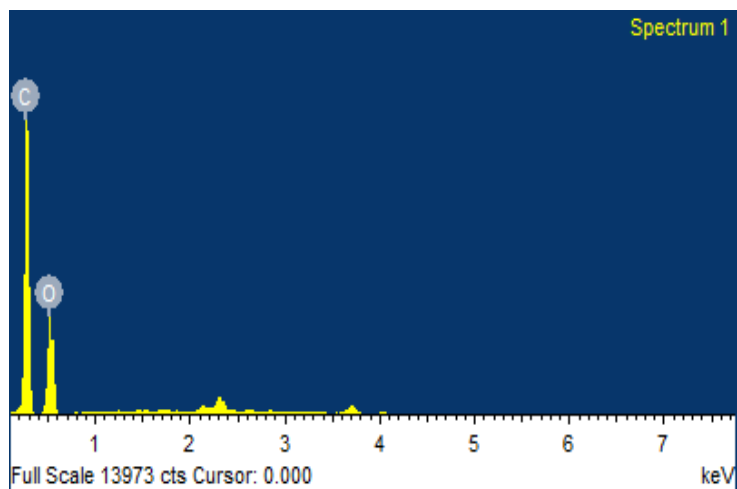
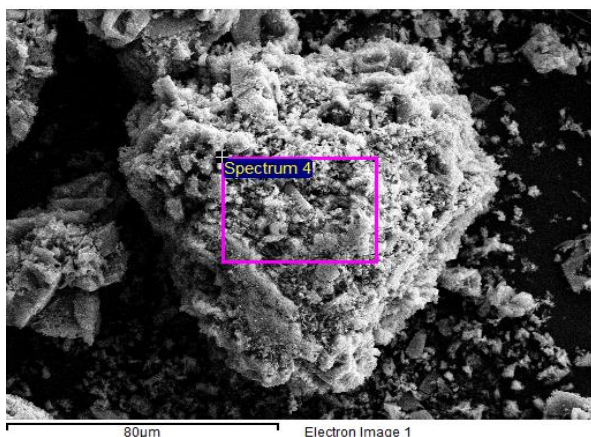


Figure 4.9: EDAX Spectra ofGO

4.1.4.2 V_2O_5 :

EDX analysis of the sample depicted in Figure 4.10 exhibits that the final product comprises of only V and O, suggesting that the nano spheres are made



Element	Weight%	Atomic%
O K	40.71	68.62
V K	59.29	31.38
Totals	100.00	

Figure 4.10: EDAX IMAGES OF V_2O_5

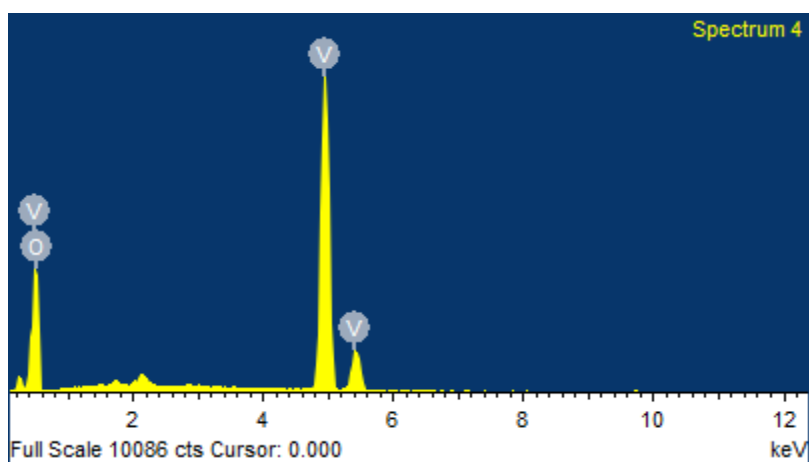
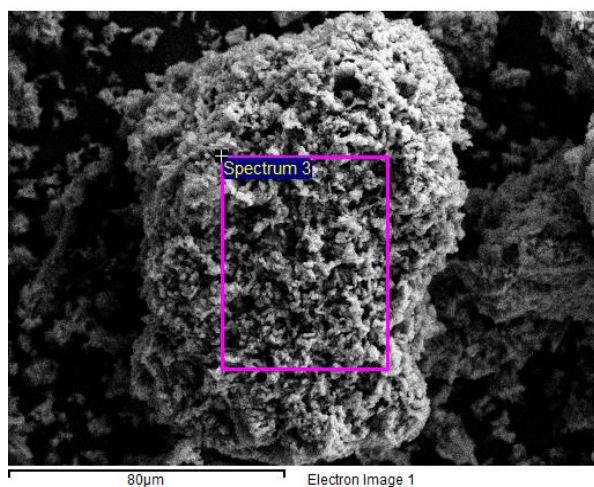


Figure 4.11: EDAX Spectra of V_2O_5

4.1.4.2 V_2O_5/GO :

EDX analysis of the sample depicted in Figure 4.12 exhibits that the final product comprises of C, O and V suggesting that there is carbon (C) peak in the EDX spectra which comes from Graphene. [Wu et al., 2014]



Element	Weight%	Atomic%
C K	4.43	16.44
V K	95.57	83.56
Totals	100.00	

Figure 4.12: EDAX IMAGES OF V_2O_5 and VGO

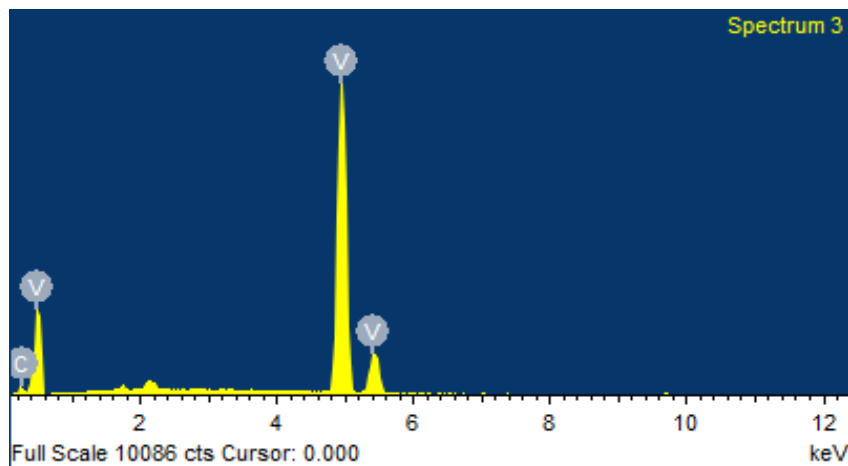


Figure 4.13: EDAX Spectra of V_2O_5 and V_2O_5+GO

CHAPTER 5

CONCLUSION

Based on the experimental results and discussions, we conclude that:

- ❖ To create an environmentally friendly electrode material, nanocomposite of Graphene and V_2O_5 nanoparticles were successfully synthesized by mechanical method.
- ❖ The V_2O_5 precursor was environmentally friendly, and the use of a toxic reducing agent was avoided.
- ❖ The XRD results, the sharp and intense peak shows the strong crystallinity.
- ❖ The presence of different molecular vibrational modes is confirmed by the FTIR spectroscopy.
- ❖ Morphology was established using SEM, and it is clear that Graphene had effectively wrapped V_2O_5 nanoparticles.
- ❖ EDAX results show the composition in graphene, V_2O_5 and nanocomposite V_2O_5 +GO.
- ❖ The above results show that, the eco-friendly electrode material for energy storage systems the commodity has a good chance of competing such as lithium ion batteries and super capacitors.

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