

INSIGHTS INTO THE STRUCTURAL, MORPHOLOGICAL, AND OPTICAL PROPERTIES OF V_2O_5 CATHODE MATERIAL SYNTHESIZED BY SELF-PROPAGATING COMBUSTION METHOD UNDER VARIED ANNEALING TEMPERATURES

(Pembelajaran ke atas Ciri-Ciri Struktur, Morfologi, dan Optik V_2O_5 yang Disintesis Melalui Kaedah Pembakaran yang Menyebar Sendiri di Bawah Pelbagai Suhu Pemanasan)

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Abstract

In this study, we report the novel synthesis of V_2O_5 cathode material utilizing the self-propagating combustion (SPC) method, marking the first-ever successful application of this technique for V_2O_5 production. Nonetheless, a thorough examination of the synthesis of V_2O_5 cathode material is required, with a special emphasis on the impact of annealing temperatures. The insufficient examination of the structural, morphological, and optical properties of V_2O_5 produced by the self-propagating combustion method in the current literature impedes the capacity to enhance its synthesis for application in energy storage devices, catalytic systems, and optoelectronic devices. In order to close this information gap, this study will carefully investigate how different annealing temperatures affect the final V_2O_5 material properties by providing comprehensive insights into the structural, morphological, and optical properties of V_2O_5 at varying annealing temperatures. The thermal profile was studied using Simultaneous Thermal Analysis (STA) providing measurement of multiple thermal properties of the sample as a function of time or temperature. The structural characterization was carried out using X-ray diffraction (XRD) analysis, revealing the crystal structure and phase purity of the synthesized V_2O_5 samples. Field emission scanning electron microscopy (FESEM) was employed to examine the morphological features, including particle size, shape, and surface morphology. Additionally, Energy Dispersive X-ray Spectroscopy (EDX) provides elemental composition to further substantiating the synthesis method's efficacy. The optical properties were investigated through UV-Visible (UV-Vis) spectroscopy, providing information on the bandgap energy and optical absorption behavior of the synthesized V_2O_5 . The results revealed that the annealing temperature significantly influenced the

structural, morphological, and optical properties of V_2O_5 . With increasing annealing temperature, the crystallinity and phase purity of V_2O_5 improved. The morphological analysis indicated variations in particle size, shape, and surface texture as a function of annealing temperature. Moreover, the optical characterization demonstrated that the annealing process influenced the bandgap energy and optical absorption properties of V_2O_5 . In this study, it was found that, the optimum annealing temperature is at 600°C, which gives a single-phase crystal structure with uniform thickness of nano-sheets like appearance.

Keywords: self-propagating combustion method, annealing temperatures, X-ray diffraction, field emission scanning electron microscopy, UV-Visible spectroscopy

Abstrak

Dalam kajian ini, kami melaporkan sintesis V_2O_5 sebagai bahan katod baru dengan menggunakan kaedah pembakaran penyebaran sendiri (SPC), menjadikan ianya laporan pertama penghasilan V_2O_5 menggunakan kaedah ini. Walau bagaimanapun, kajian menyeluruh sintesis bahan katod V_2O_5 diperlukan, dengan penekanan khusus kepada kesan suhu pemanasan. Penyelidikan yang tidak mencukupi mengenai sifat struktural, morfologi dan optik V_2O_5 yang dihasilkan oleh kaedah pembakaran yang menyebarkan diri dalam kajian semasa, menghalang keupayaan untuk meningkatkan sintesisnya untuk aplikasi dalam peranti penyimpanan tenaga, sistem katalis, dan peranti optoelektronik. Untuk menutup jurang maklumat ini, kajian ini akan mengkaji dengan teliti bagaimana suhu pemanasan yang berbeza mempengaruhi sifat bahan V_2O_5 dengan menyediakan konsepsi yang komprehensif ke atas sifat struktural, morfologi, dan optik V_2O_5 pada suhu pemanasan yang berlainan. Profil haba telah dianalisa menggunakan Analisis Terma Serentak (STA) yang menyediakan pengukuran pelbagai sifat haba sampel sebagai fungsi masa atau suhu. Bagi mengkaji struktur kristal dan fasa asli bahan, analisis pembelauan sinar-X (XRD) telah dijalankan ke atas sampel V_2O_5 yang telah disintesis. Mikroskopi Imbasan Elektron Pancaran Medan (FESEM) digunakan untuk mengkaji ciri-ciri morfologi, termasuk saiz partikel, bentuk, dan morfologi permukaan. Selain itu, Spektroskopi Sinar-X Tenaga Serakan (EDX) menyediakan komposisi elemental untuk lebih membuktikan keberkesanan kaedah sintesis. Ciri-ciri optik telah dipelajari melalui spektroskopi UV-Vis, yang menyediakan maklumat mengenai tenaga jurang dan tingkah laku penyerapan optik V_2O_5 yang disintesis. Hasilnya mendedahkan bahawa suhu pemanasan secara signifikan mempengaruhi sifat struktural, morfologi dan optik V_2O_5 . Dengan meningkatnya suhu pemanasan, kristaliniti dan kemurnian fasa V_2O_5 meningkat. Analisis morfologi juga menunjukkan perubahan dalam saiz partikel, bentuk, dan tekstur permukaan. Selain itu, sifat optik menunjukkan bahawa proses pemanasan mempengaruhi tenaga jurang dan sifat penyerapan optik V_2O_5 . Dalam kajian ini, ia telah ditemui bahawa, suhu pemanasan optimum adalah pada 600°C, yang memberikan struktur kristal satu fasa dan morfologi dengan permukaan yang sekata serta ketebalan berbentuk lapisan bersaiz nano yang seragam.

Kata kunci: kaedah pembakaran penyebaran sendiri, suhu pemanasan, pembelauan sinar-X, mikroskopi imbasan elektron pancaran medan, spektroskopi UV-cahaya nampak

Introduction

The need for effective electrical energy storage has arisen because of the increasing demand for cleaner energy and the swift expansion of the global economy [1, 2]. Among the various commercialized technologies, batteries have attracted enormous attention due to their relatively high energy density and long cycle life [3]. Rechargeable lithium-ion batteries (LIBs) have been widely used as the most popular energy storage technology in electronic devices and vehicles [4]. It is widely used in the field of portable energy storage due to its long life, without memory effect and environmental toxicity [5, 6]. Nevertheless, some problems including low safety, high cost and lithium resources shortage still hinder its development for large-

scale application [5]. Aluminum ion batteries are viewed as practical substitutes for large-scale energy storage devices due to their high theoretical capacity (2980 mAh g^{-1} , 8063 mAh cm^{-3}), abundance, and safety, with aluminum metal anodes being the primary contributing factor [7, 8]. On the other hand, developing cathode materials *via* intercalation reaction seems challenging due to the high charge density of trivalent aluminum ions. Therefore, in recent years, there have been many reports on the cathode materials of aluminum batteries, including carbon-based materials [9-11], oxides [12, 13], sulfides [14, 15], and selenides [16]. Among these cathode materials, vanadium (V) oxide, also known as V_2O_5 , is a widely recognized transitional metal oxide (TMO) due to its layered structure, low toxicity, low

cost, and ability to exhibit multiple oxidation valences (V^{2+} , V^{3+} , V^{4+} , and V^{5+}).

V_2O_5 stands as a prominent cathode material in advanced energy storage systems, due to its exceptional redox properties and high theoretical capacity [17]. Despite its appealing features, V_2O_5 has limitations that hinder its performance in certain applications. For example, the material has poor electronic and ionic conductivities, leading to lower reversible capacity and reduced rate capability [18]. To address these limitations and improve the performance of V_2O_5 , researchers are actively exploring various synthesis methods for V_2O_5 nanomaterials. Those methods are including the sol-gel method, hydrothermal synthesis, electro-spinning technique and dispersion method [19]. However, based on our current understanding, no prior attempts have been made to utilize the self-propagating combustion (SPC) method for the synthesis of V_2O_5 cathode material. SPC method has emerged as a good choice due to its simplicity, cost-effectiveness, and potential for precise control over material properties [17]. The low electrical and ionic conductivities of V_2O_5 can be ascribed to its crystal structure and morphology, which can be altered by annealing at various temperatures. By manipulating the annealing temperature, one can exert control over the crystal structure, phase purity, and morphology of V_2O_5 , hence influencing its electronic and ionic conductivities [20]. Therefore, in this study, we introduce the SPC method as a novel synthesis approach and emphasizes the importance of annealing temperature variation in tailoring material characteristics. The insights garnered herein not only contribute to the fundamental understanding of V_2O_5 , as a cathode material but also hold significant implications for the design and fabrication of high-performance energy storage devices. Simultaneous thermal analysis (STA), X-ray diffraction (XRD), field-emission scanning electron microscopy (FESEM), Energy Dispersive X-ray Spectroscopy (EDX) and Ultraviolet-Visible Spectroscopy (UV-Vis) were used to study the thermal, structural, morphological and optical characteristics of the obtained V_2O_5 cathode materials.

Materials and Methods

Preparation of V_2O_5 material

A self-propagating combustion method was used to prepare vanadium(V) oxide, V_2O_5 nanoparticles. The raw material and fuel are crucial components in combustion process. The weight of each materials used was calculated using stoichiometry method. Citric acid (fuel) and the starting material, ammonium metavanadate, NH_4VO_3 was stirred in 15 ml and 200 ml deionized water respectively before the fuel was poured into the NH_4VO_3 solution. The poured solution was vigorously agitated for 25 minutes on a magnetic stirrer to achieve a homogenous mixture. The solution is held on the hot plate at 300 °C until combustion occurs, and then the material is calcined in a prepared furnace at different temperatures of 500 °C, 600 °C and 700 °C for 6 hours. The materials were removed after the material is allowed to cool in the furnace in off mode resulting in the production of black powder product.

Thermal analysis

Thermal profile of the precursors was carried out using a simultaneous thermogravimetric analyzer (STA; SETARAM SETSYS Evolution 1750, Caluire, France). It means that the thermogravimetric analysis (TGA) and the differential scanning calorimetry (DSC) were measured simultaneously. In this study, about 10-15 mg of each precursor was heated from 30 °C to 700 °C with a heating rate of 10 °C per minute under air atmosphere. The precursors were placed in alumina crucibles which was suitable for heating to high temperatures.

Characterization

The XRD of the samples was obtained by a diffractometer (PANalytical X'pert PRO) with Cu α radiation. The morphology of cathodes was characterized *via* FESEM. The EDX was used to characterize the composition of the cathode materials. The optical band gap characteristics of the sample were obtained using UV-Vis spectroscopy.

Results and Discussion

Thermal profile

Figure 1 shows the decomposition behavior of the precursor. There are three weight loss occurring on the TGA profile marked as I, II and III. The first weight loss in region I is around 6.5% at the temperature range of 30 °C to 160 °C accompanied by an exothermic peak that

is attributed to the evaporation of water from the sample surface [21]. NH_4VO_3 began to transform into ammonium vanadate ($(NH_4)_2(V_6O_{16})$) with the escape of NH_3 [22]. The abrupt weight loss of 30.8% from ~ 200 °C to 430 °C period may correspond to the bound water in the structure [23] and it is related to the exothermic process occur at that point. The third region, between 420 °C and 560 °C relates to weight loss of 27.20%. It is correlated with the high and broad exothermic peak on DSC curve at ~ 540 °C which indicates the releasing of NH_3 , O_2 and H_2O [24]. The endothermic peak at 680 °C was attributed to the recrystallization of V_2O_5

materials physical process, which did not affect the vanadium valence of the final products, in facts it involves breaking and reforming intermolecular bonds [23]. This can be related to the TGA graph, where there is no weight loss in the compound at above 600°C, while the endothermic peak may be due to the structural phase transition of V_2O_5 crystals. Consequently, according to the acquired thermal analysis, all samples were annealed at 500 °C, 600 °C and 700 °C. From here, it was suggested that varying the annealing temperature may lead to distinct material characteristics.

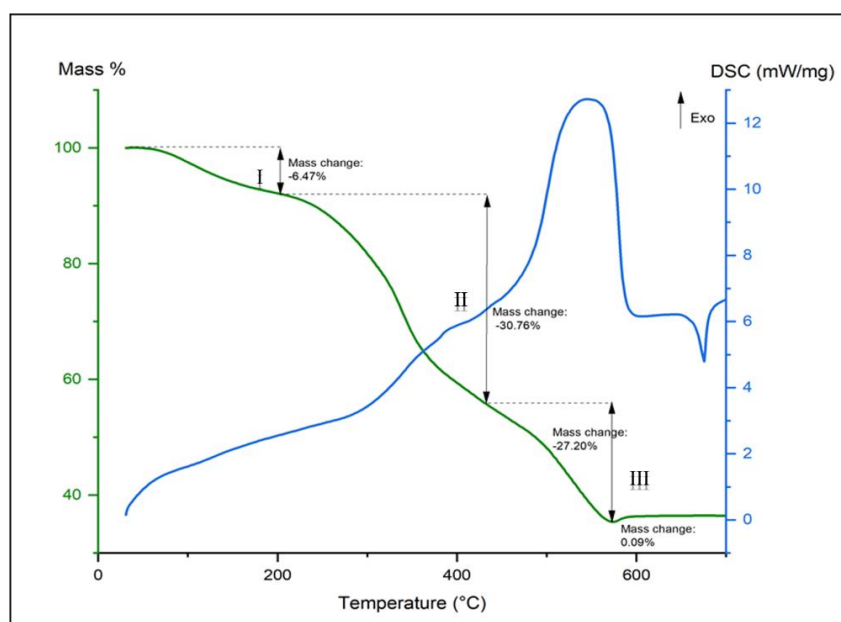


Figure 1. Simultaneous thermogravimetric analyzer (STA) of the V_2O_5 precursor

Structural characterization

Figure 2 displays the XRD patterns of various materials. The annealed sample at a temperature of 500 °C (as shown in Figure 2(a)) exhibits XRD peaks of low intensity, suggesting the existence of orthorhombic and monoclinic crystal structures attributed to vanadium (V) oxide and vanadium (IV) oxide, respectively. The orthorhombic crystal structure of V_2O_5 can be achieved through the process of annealing at temperatures of 600 °C and 700 °C, as depicted in Figure 2(b) and (c) correspondingly. Both samples have a high level of purity and possess an orthorhombic crystal structure system with the Pmmm space group, as validated by the JCPDS reference number 00-041-1426. The

augmentation of the annealing temperature results in a corresponding augmentation of the XRD peak intensities. This observation suggests enhancements in the crystallinity of the V_2O_5 material, as well as the production of well-organized crystals. Nevertheless, an abrupt increase in magnitude is noted in the specimen that underwent annealing at a temperature of 700 °C. This increase aligns with the recrystallization peak identified in the STA analysis, which occurs at around 680 °C during the thermogravimetric analysis's stable phase (refer to Figure 1). FESEM micrographs can provide additional evidence to corroborate this condition.

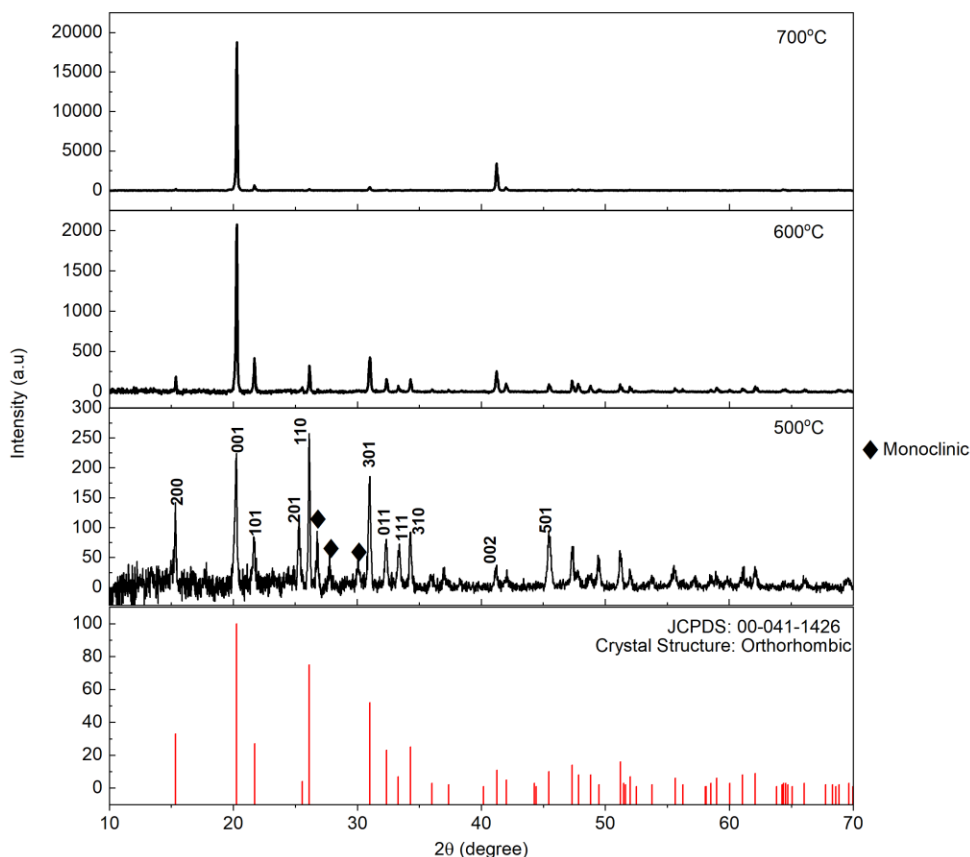


Figure 2 XRD patters of V_2O_5 samples annealed at 500 °C, 600 °C and 700 °C

Morphological characterization

The shape of V_2O_5 at various annealing temperatures is depicted in the FESEM pictures presented in Figure 3(i) and (ii). For the sample annealed at 500 °C, the observed structures exhibit a high degree of agglomeration, forming flake-sheet-like arrangements. These structures possess an average thickness of 196 nm. The flakes have a slender and flat configuration reminiscent of a two-dimensional plane, characterized by a sleek exterior and intermittent protrusions and boundaries. The nano-sheets depicted in sample annealed at 600 °C exhibit a homogeneous thickness spanning 48 nm, accompanied by a regular arrangement of layers while sample annealed at 700 °C shows that the compound displayed prominent bulky flakes with a sheet-like structure. This observation suggests that the compound maintained a constant flaky sheet shape as the annealing temperature was raised. Significantly, the flaky sheets exhibited an increase in size while retaining their characteristic

layered structure, as depicted in Figure 3(ii) under a magnification of 50,000x. The observed morphology aligns with the findings of the XRD investigation, which suggests an increase in crystallinity with elevated annealing temperatures. The structures' large aspect ratio is believed to contribute to their possibly improved characteristics in the context of battery applications. The reduced thickness of micro- and nano-sheets facilitates shorter diffusion paths inside the electrode material, leading to enhanced ion transport and improved utilization of the active material. Consequently, this enables faster charge and discharge cycles. The utilization of this data can facilitate the optimization of the annealing temperature to achieve the desired V_2O_5 compound, which serves as an optimal cathode material for usage in aluminum batteries. The compositional atomic percent of the elements obtained by EDX are shown in Table 1 with percentage difference less than ~4%.

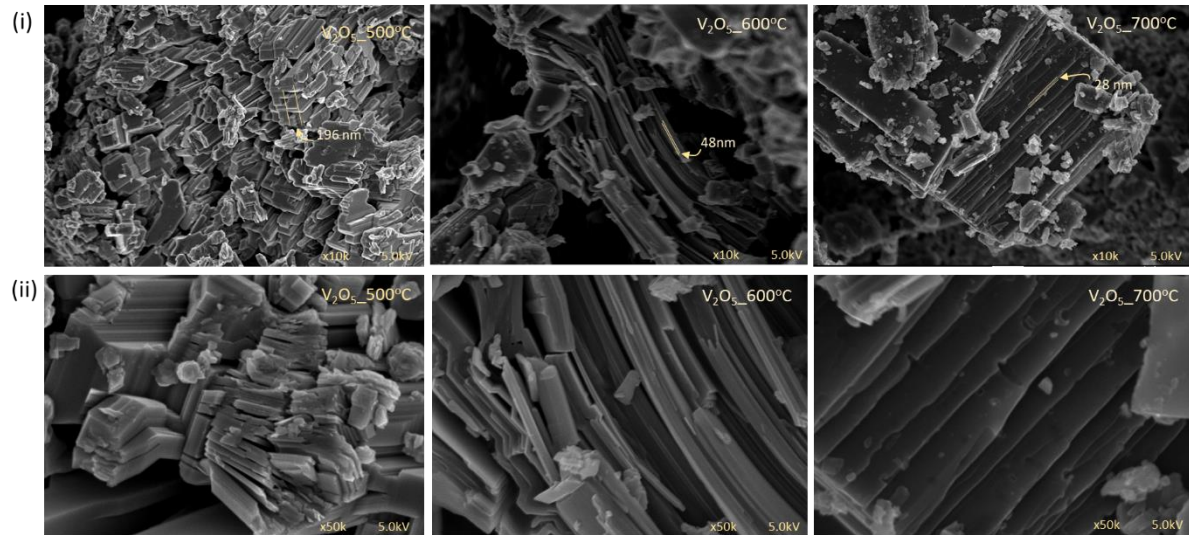


Figure 3. (i) FESEM images of pure V₂O₅ at 10K magnification with annealing temperature of 500°C, 600°C and 700°C and (ii) 50K magnification for each sample

Table 1 Compositional distribution from EDX of the constituent elements for the V₂O₅ at different annealing temperatures.

Elements	Theoretical Atomic %	Experimental Atomic %					
		V ₂ O ₅ _500°C	% Difference	V ₂ O ₅ _600°C	% Difference	V ₂ O ₅ _700°C	% Difference
Vanadium	28.6	27.5	3.9	29.7	3.8	29.0	1.4
Oxygen	71.4	72.5	1.5	70.3	1.6	71.0	0.6

Optical characterization

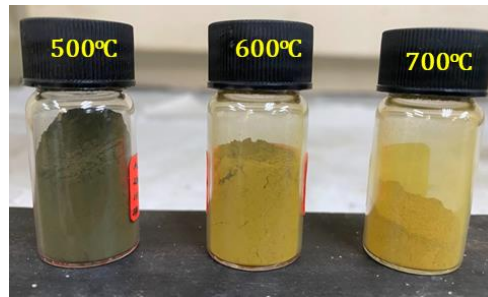


Figure 4. V₂O₅ samples at different annealing temperature

As in Figure 4, the samples of synthesized V₂O₅ were observed at different annealing temperatures. An interesting observation was made as the annealing temperature increased, the colour of the synthesized V₂O₅ became lighter. For optical band gap evaluation, reflectance spectra of all samples were obtained by UV-Vis spectroscopy and further analysed using the Tauc equation (equation (1) [25],

$$(\alpha h\nu) = A(h\nu - E_g)^x \quad (1)$$

where α is the absorption coefficient of the material at a certain value of the wavelength λ , h is the Planck's constant, A is a proportionality constant, ν is the frequency of light, E_g is the band gap energy and $x = 1/2$ (for direct transition mode materials). The absorption coefficient is evaluated using,

$$\alpha = k \ln \left(\frac{R_{max} - R_{min}}{R - R_{min}} \right) \quad (2)$$

where k is a constant, R_{max} is the maximum reflectance and R_{min} is the minimum reflectance.

Consideration of Eqs. (1) and (2) gives,

$$(ah\nu)^2 = A'(h\nu - E_g) \quad (3)$$

where A' is a constant. From Eq. (3), a Tauc plot can be drawn of $(ah\nu)^2$ versus $h\nu$. The band gap energy is obtained by extrapolation of the linear part and the point that meets the abscissa will give the value of the band gap energy of the material. Upon careful analysis using Tauc plots (Figure 5), it was evident that the band gap values experienced a significant reduction as the annealing temperature increased. Specifically, the band gap values were measured to be 3.896 eV, 3.878 eV, and 3.870 eV for annealing temperatures of 500°C, 600°C, and 700°C respectively. These changes indicate the alterations in the electronic structure of the material.

Specifically, it points towards a reduction in the band gap [26]. This correlation between the colour of the sample and the band gap is in line with expectations. Lighter colours are associated with lower band gap values. This is because a smaller band gap implies that less energy is required to promote an electron from the valence band to the conduction band. As a result, the material absorbs and reflects light differently, giving rise to the observed colour change. In battery operation, band gap governs charge transfer mechanisms, dictating the efficiency with which charge carriers the electrons and holes are generated and facilitating smooth charge transfer during electrochemical reactions. Moreover, the band gap significantly influences the voltage and energy density of the battery, ensuring compatibility with other components for optimal performance. Furthermore, identifying a suitable band gap contributes to cycling stability, offering insights into the material's durability over numerous charge-discharge cycles, a pivotal factor for the battery's long-term reliability [27].

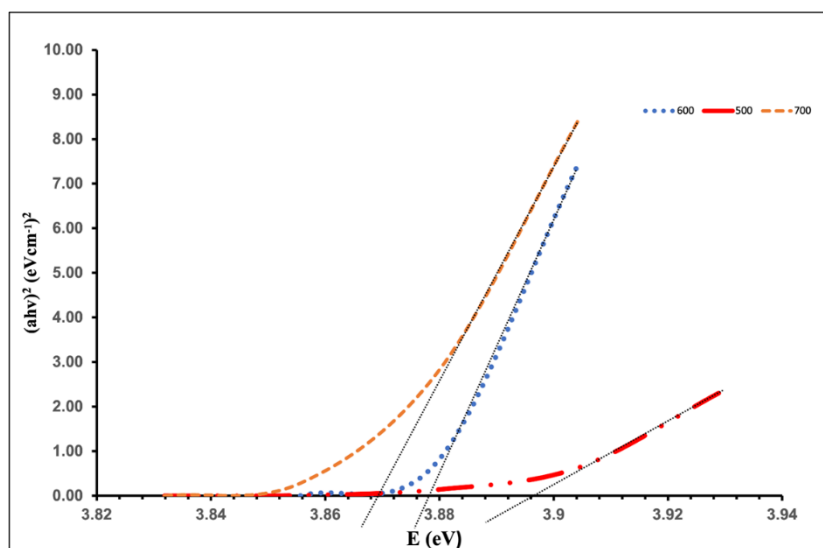


Figure 5. Tauc plots for V_2O_5 powder annealed at 500 °C, 600 °C and 700 °C

Conclusion

Structural, morphological and optical properties of single-phase V_2O_5 powder are synthesized by self-propagating combustion method. XRD results show that the compound is polycrystalline with orthorhombic symmetry structure. After the annealing process, the crystallization was improved by increasing temperature. FESEM images show a highly agglomerated flakes-

sheet-like structure for sample annealed at 500 °C. All flakes exhibited thin and planar structure that resemble a two-dimensional sheet. The surface of the flakes was found to be predominantly smooth, with occasional ridges and edge. Upon increased annealing temperature, the compound exhibited consistent flaky sheet morphology. The flaky sheets grew in dimensions while maintaining their distinct layered appearance as well as

the ridges and edges. This is consistent with the XRD analysis where it is profound that the crystallinity was enhanced at higher annealing temperature. Sample anneals at 600 °C gives a single-phase orthorhombic crystal structure with uniform thickness of nano-sheets like appearance and smooth surface suggesting that the optimum annealing temperature was at 600 °C. Optically, it was found that lighter colors of V₂O₅ powder associated with lower band gap values. The observed changes in color and band gap values provide valuable insights into how annealing temperatures influence the electronic properties of the synthesized V₂O₅, which in turn can have significant implications for its performance as a cathode material in energy storage applications.

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