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Synthesis of Vanadium Pentoxide using Hydrothermal Autoclave Approach

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ABSTRACT

The simple hydrothermal autoclave approach was successfully used to synthesize V_2O_5 nanoparticles from NH_4VO_3. The synthesized nanoparticles were characterized using various techniques that indicating the presence of V_2O_5 nanoparticles. The synthesized V_2O_5 nanoparticles characterized using X-Ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM) and atomic force microscopy (AFM). The result confirms forming of V_2O_5 nanoparticles and the diameter was around 61- 167 nm.

1. INTRODUCTION

Metal oxides (MOs) are versatile materials with a wide range of properties and applications [1]. Many MOs (semi)conductors are used as transparent conductive coatings in electronic devices such as solar cells and touch screens because they have energy gaps higher than 3 eV. The properties of metal oxides can be tailored by controlling their composition, structure, and morphology. This allows for the development of materials with specific electrical, optical, and catalytic properties for different applications [2]. The ionic compositions, known as metal oxides, are composed of cation metal and anion oxygen [3] and they can have ability to undergo redox reactions makes them useful in energy storage and conversion devices. [4]. Despite the fact that positive metallic ions' s-shells are always completely filled with electrons in metal oxides, the d-shells may not always be [5].

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The element Vanadium (V) was initially named "panchromium" in 1801 by del R´ıo, but it was later rediscovered in 1830 by the Swedish chemist Nils Gabriel Sefstr¨om and renamed vanadium [6]. Overall, vanadium is a versatile element with several oxidation states, with the +4 and +5 states being the most common. There are many types geometrical V-O coordination of vanadium oxides, including V-O, V_2-O_3, V-O_2 and V_2-O_5 [7].

Vanadium pentoxide (V_2O_5) is the form of vanadium that is both most common and useful. Other widely used vanadium salts are sodium orthovanadate (Na_3VO_4), ammonium metavanadate (NH_4VO_3) and sodium metavanadate ($NaVO_3$). While vanadium has numerous industrial and technological applications, it is important to handle and use it with caution due to its potential toxicity. The most hazardous form is vanadium pentoxide, inhalation of vanadium compounds can lead to respiratory issues, while ingestion can cause gastrointestinal irritation [8]. Three polymorphs of V_2O_5 exist: α-V_2O_5 (orthorhombic), β-V_2O_5 (monoclinic) and γ- V_2O_5 (orthorhombic) [9]. The other phases were changed into α -V_2O_5, since it is the most stable phase at high pressure and temperature. [10]. Vanadium pentoxide (V_2O_5) has received intense attention because of its interesting electro-chemical properties, thermos-chromic, and electro-chromic advantages, a wide band gap and n-type semiconductor material employed in electrocatalytic applications. Interestingly, the change from p- to n-type conduction was found to be controlled by orthorombic V_2O_5 doping. [11]. There are five bonds between oxygen and vanadium atoms, with lengths ranging from 1.585 to 2.021. As shown in Fig. 1, there is one link involves O (1) atoms, one link involves O (2) atoms and three links include O (3) atoms [12].

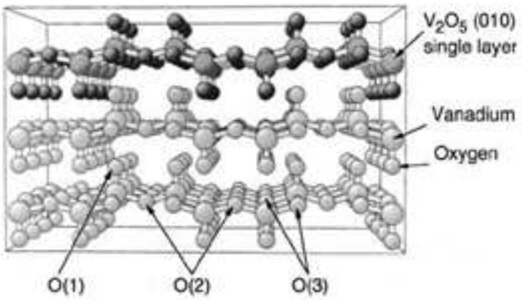

Fig.1: V_2O_5 crystal section [12].

2. Experimental

2.1. Chemical Materials

NH_4VO_3 from Sigma-Aldrich Co. (99.0%), Ethanol (100%) from HaymanKymia.

2.2. Synthesis of V_2O_5

The V_2O_5 NPs were fabricated using hydrothermal methods. The overall reaction for the thermal decomposition of NH_4VO_3 to yield V_2O_5 can be represented as:

$$2\ NH_4VO_3 \rightarrow V_2O_5 + 2\ H_2O + 2\ NH_3 \qquad (1)$$

This reaction involves the decomposition of (5 gm) of ammonium metavanadate (NH_4VO_3) kept in stainless steel reactor at high temperatures (500-550 ºC) for 2 hours, to produce vanadium pentoxide (V_2O_5) with the orange color, water (H_2O), and ammonia (NH_3). [13]. (Fig.2) shows the hydrothermal prosses.

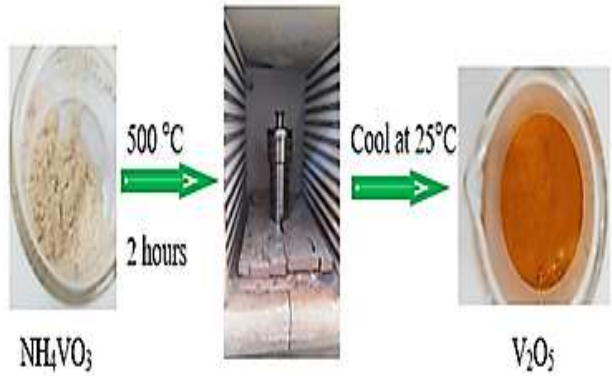

Fig.2: shown the hydrothermal presses.

The synthesis of V_2O_5 nanoparticles through a series of steps including weight of micro-size V_2O_5 (5 gm) powder and placing it in a reactor, and heating it to 850 ºC in a muffle furnace to melt it. After that, it is cooled it by cold deionized water until reached to room temperature, while being continuously stirred for 8 hours. Finally, aging of V_2O_5 solution by kept overnight in a sealed airtight bottle.

After that, the solution was poured into a stainless-steel hydrothermal vessel lined with Teflon. The vessel was then heated to 180 ºC for 15 hours, and it was left to cool naturally through convection at room temperature. The resulting yellow residue is then collected and rinsed numerous times with deionized water before being cleaned with ethanol to obtain pure V_2O_5 nanoparticles [14].

3. Result and discussion

3.1. X-Ray diffraction (XRD)

From the XRD measurements that are shown in Fig. (3) and table (1) the main diffraction peaks of V_2O_5 2θ =15.62°, 20.63°, 22.0°, 26.4°, 31.3°, 32.7°, 34.65°, 41.62°, 45.8°, 47.66°, 49.1° 51.5° 52.3°, 55.9°, 58.9°, 61.43° and 62.4° respectively, correspond to the characteristic diffraction of the (200), (001), (101), (110), (301), (011), (310), (002) (102), (411), (012), (600), (020), (021), (412), (321) and (710) planes of the V2O5 as indexed in the JCPDS card No. 00-041-1426 [15].

Table (1): The crystalline size (D) of the V_2O_5:

2θ (degree)	FWHM (degree)	Crystal Size (nm)	Average (nm)
15.68	0.191	42	
20.63	0.246	33	
22.06	0.232	35	
26.49	0.220	37	
31.36	0.246	33	
32.71	0.25	33	
34.65	0.206	40	
41.62	0.203	42	
45.82	0.175	50	40
47.66	0.231	38	
49.15	0.288	30	
51.55	0.195	45	
52.3	0.181	49	
55.96	0.195	46	
58.90	0.23	40	
61.43	0.216	43	
62.44	0.229	41	

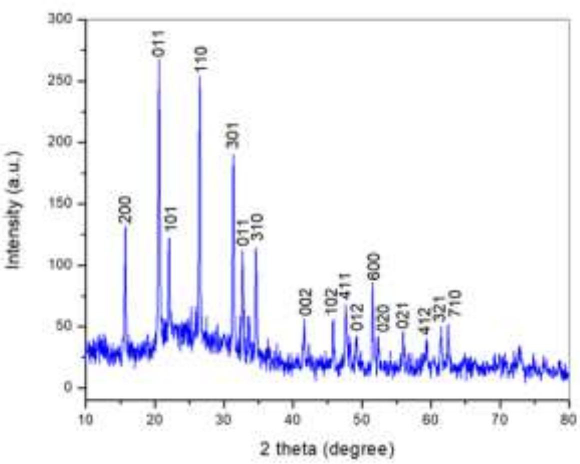

Fig. 3: XRD of vanadium pentoxide nanoparticles.

3.2. FE-SEM of vanadium pentoxide

The FE-SEM analysis is a useful tool to study the surface morphology of nanoparticle. Fig. 4 exhibits FE-SEM images of V_2O_5 nanoparticles that shows the particle of V_2O_5. The lateral size of nano particle was found in the range 61- 167 nm.

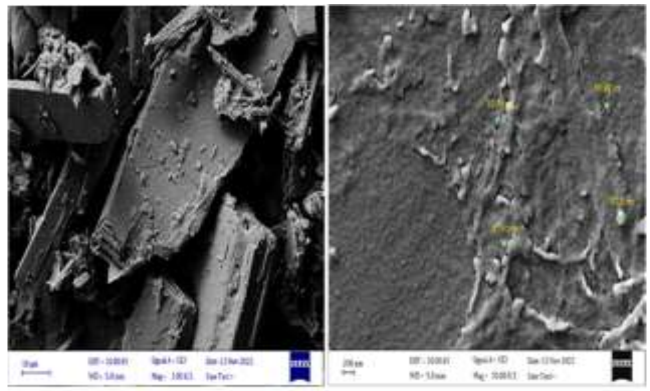

Fig. 4: FE-SEM image of vanadium pentoxide nanofibers.

3.3. AFM of vanadium pentoxide

AFM was used to assess and analyze the surface morphology and roughness of the V_2O_5 nanoparticles. Fig. 5 displays the AFM image of the V_2O_5, which shows how grain-like tubes are deposited to create a thin layer. These findings are consistent with the SEM images, which also displayed similar morphologies. Table 2 displays the surface roughness of the V_2O_5 nanoparticle films, which was found to be 8.713 nm.

Table (2): AFM information of V_2O_5.

Roughness Average Sa (nm)	Root Mean Square Sq (nm)	Ten Point High Sz (nm)	Average Diameter D (nm)
8.713	10.983	69.14	56.99

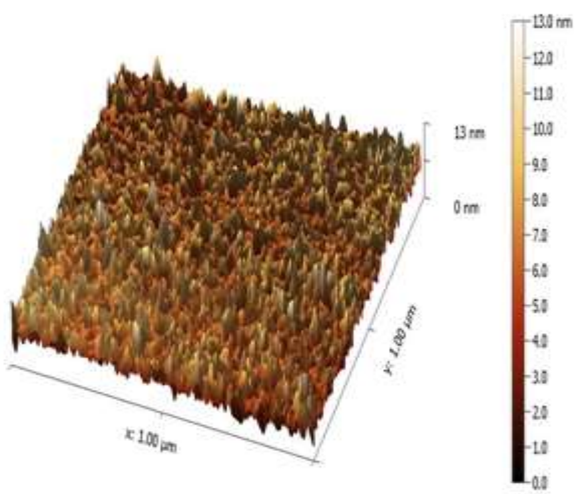

Fig. 5: AFM image of V_2O_5 nanoparticle.

4. Conclusion

We conclude from this study that the using a hydrothermal autoclave is simple, no expensive and friendly to the environment method. The temperature at 180 ° C for about 15 hours is led to formation of nanoparticles of vanadium pentoxide with the diameter of 61- 167 nm and average of crystalline size (D) was 40 nm.

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Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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تحضير خامس أوكسيد الفناديوم باستخدام طريقة الأوتوكلاف المائي الحراري

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الخلاصة:

معدن الفناديوم الانتقالي يمكنه بسهولة أن يتحول بين حالات الأكسدة +5، +4، +3، ويوسع مجاله الى ما بعد التنسيق رباعي السطوح، يتأكسد الى ثلاثي أوكسيد الأسود البني، رباعي الأوكسيد الأسود المزرق، وخامس الأوكسيد البرتقالي المحمر عند تسخينه في الهواء بدرجات حرارية مختلفة. تم استخدام طريقة الأوتوكلاف المائي الحراري البسيطة لتخليق دقائق خامس أوكسيد الفناديوم النانوية من ميتا فانادات أمونيوم. العينات التي تم تحضيرها شخصت بواسطة حيود الأشعة السينية والمجهر الإلكتروني الماسح ومجهر القوة الذرية. النتائج أكدت تكون الدقائق النانوية لخامس أوكسيد الفناديوم والقطر كان حوالي 61–167 نانومتر.

الكلمات المفتاحية: حراري مائي، أكسيد المعدن، الدقائق النانوية، خامس أوكسيد الفناديوم.