Facile synthesis of high yield two dimensional zinc vanadate nanoflakes

Sawsan A. Mahmoud¹ · Samar H. Bendary¹ · A. A. Salem¹ · Osama A. Fouad²

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Abstract

Zinc vanadate nanoflakes, with highly porous structure in the range of 50 nm wall thickness, were successfully prepared from ion solutions of the corresponding precursors by a simple co-precipitation method. The prepared zinc vanadate nanoflakes were inspected by X-ray diffraction (XRD), Field emission scanning electron microscope (FE-SEM), High Resolution transmission electron microscope (HRTEM), fourier transform infrared (FTIR), Raman spectroscopy and X-ray fluorescence (XRF) techniques. The formed nanoflakes phase, after drying at 60 °C, is zinc hydroxide vanadium oxide hydrate. After calcination at 400 °C zinc vanadium oxide phases were formed as confirmed from XRD analysis. FE-SEM shows the formation of two dimensional (2D) zinc vanadate nanoflakes which caused by familiarizing shielding of $V(OH)_4^-$ ions on Zn^{2+} surface. The absence of ZnO Raman peaks indicates that the bulk phase was a pure zinc vanadate phase. Schematic mechanism of the surface morphology of the zinc vanadate formation was suggested. The high yield of the unique structure obtained by this simple synthesis method might open the door for vital industrial applications of the prepared material and synthesis of other nanostructured materials.

Keywords Zinc vanadate nanoflakes · Co-precipitation · Porous structure

1 Introduction

Zinc Oxide has several advantages including its abundance in nature, nontoxic, high chemical stability, easy to be fabricated and to be doped [2, 26, 28, 56]. Recently, as reported by Gowrishankar, significant awareness has been concentrated on the development of ZnO for different applications, such as electrochromic and optoelectronic devices [11]. The band gap energy of ZnO is 3.3 eV at room temperature and an excitonic binding energy is 60 meV, so the improvement of the band gap is one of the prime requirements in designing optoelectronic devices. Literature reported that ZnO band gap could become narrow and could be engineered by alloying this material with three major elements: Mg [11, 27], Vanadium [11] and cadmium [15, 27]. Magnesium and Vanadium are known to broaden the band gap, whereas Cd is known to narrow it.

Metal vanadates are considered to be a vital candidates of inorganic nanomaterials that have considerable attention as complex oxides owing to their possible application in different areas as; catalysis [25], photoluminescence property [31, 42], antibacterial agent (Holtz et al. [13], photocatalytic activities [48], multiferroic behavior [55], nanotribology [47], cathode electrode in batteries for energy storage [24], implantable cardiac defibrillators (ICDs) [7] and low-temperature magnetic devices [21].

Most of the reported synthesis methods mainly carried out by the hydrothermal method for example; researchers have focused on the synthesis of metal vanadates of sodium [1]. Song et al. [50] have synthesized super long β -AgVO₃ nanoribbons. Dey et al. [8] have synthesized a Cu(II) connected V₄O₁₆ cubane based metal organic framework. Pei et al. [33, 34] represent a controlled synthesis of calcium vanadate. Recently,

✉ Sawsan A. Mahmoud, sawsanhassan2003@yahoo.com | ¹Egyptian Petroleum Research Institute, EPRI, P.O. Box: 11727, Nasr City, Cairo, Egypt. ²Central Metallurgical Research and Development Institute, CMRDI, P.O. Box: 87, Helwan, Cairo 11421, Egypt.

few reported researches have been focused on facile synthesis of metal vanadate by different methods for example; Khan and Qurashi [18] reported a facile synthesis of copper vanadate nanostructures via sonication assisted sol gel method. Mohammad Reza Mosleh has synthesized FeVO_4 by a facile morphology control in presence of different surfactants [30]. Lin et al. [20] has reported the Synthesized Cu vanadate nanorods using polyvinyl pyrrolidone polymer by hydrothermal method for photocatalytic degradation of gentian violet under visible-light. Copper vanadate nanobelts have been successfully synthesized by a facile hydrothermal process using sodium vanadate and copper acetate as raw materials, and the polymer polyvinyl pyrrolidone (PVP) as a surfactant by adjusting the pH value as reported by Pei et al. [39, 40]. Manganese vanadate nanobelts have been synthesized by a simple hydrothermal process using polymer polyvinyl pyrrolidone as a photocatalyst in methylene blue as reported by Pei et al. [41]. Manganese vanadate nanorods with a single crystalline triclinic $\text{Mn}_2\text{V}_2\text{O}_7$ phase have been synthesized through a hydrothermal process using sodium lauryl sulfonate (SDS) as a surfactant [35–37]. Mn vanadate nanosheets and visible-light photocatalytic performance for the degradation of methyl blue was reported by Pei et al. [35–37]. Formation mechanism of manganese vanadate microtubes and their electrochemical sensing properties was reported by Pei et al. [35–37].

Ternary vanadate one-dimensional nanomaterials exhibit wide application potential in the fields of lithium ion batteries, photocatalysis and electrochemical sensors due to their good electrochemical and photocatalytic properties [38].

However, the synthesis of zinc vanadate is infrequently described. Several articles have shown that zinc vanadate has been synthesized by hydrothermal method. Natarajan described the crystal structure of $[(\text{NH}_3(\text{CH}_2)_3\text{NH})\text{Zn}]_2^{3+}[\text{V}_4\text{O}_{13}]$ by hydrothermal synthesis [32]. Saldarriaga et al. have synthesized a porous zinc vanadate with molecular formula $\text{Zn}_3(\text{VO}_4)_2 \cdot 3\text{H}_2\text{O}$ under hydrothermal conditions [14]. Shi et al. [44] reported the synthesis of $\text{Zn}_3\text{V}_2\text{O}_8$ under hydrothermal route. Wang et al. [53] reported the photophysical and photocatalytic properties of $\text{Zn}_3\text{V}_2\text{O}_8$. Sun et al. [51] improved a methodology for the synthesis of ultra-long monoclinic ZnV_2O_6 nanowires. Xiao et al. [58] reported the non-aqueous sol–gel synthesis of clew like ZnV_2O_4 hollow spheres under hydrothermal conditions and their storage performance. Shi et al. [44] studied the synthesis of $\text{Zn}_3\text{V}_2\text{O}_7(\text{OH})_2(\text{H}_2\text{O})_2$ and $\text{Zn}_3\text{V}_2\text{O}_8$ nanostructures by hydrothermal route and their photocatalytic performance. Pei et al. [39, 40] reported the synthesis of Zinc vanadate nanorods by a simple hydrothermal process

using zinc acetate and sodium vanadate as the raw materials.

Medjnoun et al. [26] reported the synthesis of nanostructured $\text{Zn}_{1-x}\text{V}_x\text{O}$ thin films with high vanadium content elaborated by rf-magnetron sputtering. [3] have discussed the synthesis of zinc vanadate by controlled co-precipitation of $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ using NaVO_3 as precipitant in presence of non-ionic Polysorbate 80 surfactant in thin film mixed flow glass reactor. Recently, Mondal et al. [29] have reported the synthesis of robust cubooctahedron $\text{Zn}_3\text{V}_2\text{O}_8$ using simple method and have studied its efficiency for photocatalytic dye degradation in water. Some researchers reported facile sol–gel synthesis and photocatalytic activity of the V_2O_5 – ZnO nanoflakes [46].

Herein, for the first time, we have synthesized hierarchical $\text{Zn}_3(\text{OH})_2(\text{V}_2\text{O}_7)(\text{H}_2\text{O})_2$ nanostructure via a template free-simple co-precipitation approach in high yield under mild condition. Then $\text{Zn}_2\text{V}_2\text{O}_7$, with similar nanostructure, was produced by heat treatment of $\text{Zn}_3(\text{OH})_2(\text{V}_2\text{O}_7)(\text{H}_2\text{O})_2$ nanostructures.

2 Experimental

2.1 Materials

Zinc acetate dihydrate $(\text{CH}_3\text{COO})_2\text{Zn} \cdot 2\text{H}_2\text{O}$; 99.5%, Merck), ammonia solution (NH_4OH ; 30%, Adwic) and ammonium meta vanadate (NH_4VO_3 ; 98%, Adwic) were used as received without further treatment. Bi-distilled water was used as a dissolving media for the experimental reactions and as a washing agent for the produced materials.

2.2 Synthesis of zinc vanadate

Zinc vanadate was synthesized by a simple precipitation method. Briefly, 0.40 mol of zinc acetate dihydrate and 0.089 mol of ammonium metavanadate were dissolved separately in bi-distilled water and assigned as solution (1) and (2), respectively. Then solution (2) was added to solution (1) and the pH was adjusted using ammonia solution at 9 with continuous stirring at 80 °C for 6 h. Then the solution was left overnight at room temperature without stirring. A yellow precipitate was formed and separated from the residual solution by centrifuging. Then the separated powders were dried at 60 °C and then calcined at 400 °C and kept for further investigation and studies.

2.3 Characterization techniques

X-ray diffraction patterns were recorded using a Pan Analytical Model X'Pert Pro system, which was equipped with Cu-K α radiation ($\lambda = 0.1542$ nm). Particle size distribution

in the powder samples are measured by Scherrer's equation. The crystallite size of the prepared zinc vanadate samples was calculated from the most intense peak plane that is obtained from the X-ray diffraction data using the Debye–Scherrer's formula as follows:

$$d_{RX} = k\lambda/\beta \cos \theta \quad (1)$$

where d_{RX} is the crystallite size, $k=0.9$ is a correction factor accounted for particle shapes, β is the full width at half maximum (FWHM) of the most intense diffraction peak plane, λ is the wavelength of Cu target = 0.15406 nm, and θ is the Bragg angle. The peak width in XRD spectra was measured with accuracy of $<0.05^\circ$. The oxide components in the prepared material are determined by X-Ray Fluorescence Spectrometry (XRF-1800, Shimadzu Corporation, Japan), the sample is grinded, compressed, and sintered at high temperature before analysis. The dynamic light scattering technique using Malvern Zetasizer ver. 6.32 instruments was used to measure the size distribution profile. Morphology of the prepared materials was investigated using high field emission scanning electron microscopy (HFSEM) JEOL, JEM 3500 electron microscope and high resolution transmission electron microscopy (HRTEM) using a JEOL, JEM-1230 electron microscope operating at 120 kV. The samples were sonicated in methanol for 20 min before measurement. Fourier Transform Infrared Spectroscopy (FTIR) was recorded on Nicolet i 50 Thermo Fisher Scientific FT-IR spectrophotometer. A Raman spectrum was recorded for the calcined sample using Senterra BRUKER with excitation laser wavelength at 532 nm.

3 Results and discussion

3.1 Crystal structure and phase identification

Zinc vanadate yellow powder precipitated upon addition of zinc ion and vanadium ion solutions in presence of ammonia solution under the previously mentioned controlled experimental conditions. Figure 1a, b shows the X-ray diffraction patterns of the dried and calcined samples. In general the diffraction peaks are relatively sharp with low intensities, Fig. 2a. It is clear that most of the diffraction peaks in Fig. 1a can be assigned to hexagonal $\text{Zn}_3(\text{OH})_2\text{V}_2\text{O}_7(\text{H}_2\text{O})_2$ phase according to JCPDS card # 01-087-0417. This indicates the good incorporation of vanadate ions into zinc lattice. Whereas the diffraction peaks shown in Fig. 1b can be assigned to orthorhombic $\text{Zn}_3\text{V}_2\text{O}_8$ phase in agreement to JCPDS card # 00-034-0378 and monoclinic $\text{Zn}_2\text{V}_2\text{O}_7$ phase matching the JCPDS card# 01-070-1532 nanocrystals. Quantitative calculation of the phase formation from XRD data shows that $\text{Zn}_3\text{V}_2\text{O}_8$ phase represents 94.1% while $\text{Zn}_2\text{V}_2\text{O}_7$ phase represents 5.9% of

Fig. 1 **a, b** XRD patterns of the produced zinc vanadate nanopowder samples and stick patterns of the corresponding phases. **a** dried at 60 °C and **b** calcined at 400 °C. The right insets are the particle size distribution histograms

the precipitated nanopowders. The mean crystallite size of the dried sample at 60 °C and the calcined sample at 400 °C is in the range of 26.3 and 58 nm, respectively. The diffraction data revealed that the materials transformed from monoclinic to orthorhombic system upon heat treatment which resulted in the formation of larger grains and hence larger particles. The large size grains of the calcined sample at 400 °C might be due to long-range order, and consequently high crystallinity [49].

The chemical composition of the as-prepared sample expressed as oxide components is given in Table 1. XRF result revealed that the as-prepared material is composed mainly of 46.63% ZnO and 37.11% V_2O_5 . The compounds of ZnO and V_2O_5 represent 83.75% of total weight. Whereas weight loss upon heating is 15.2% which could be attributed to the water contents in the sample. This in addition to some other traces oxides ($\approx 1\%$) which might be due to the impurities present in the precursor materials.

Particle size distribution profile was confirmed by dynamic light scattering (DLS) in the bulk powder of the calcined sample. DLS was shown in the insets of Fig. 1. In general, the sample shows a wide-sized range.

Fig. 2 **a, b** SEM and **c, d** HRTEM images of the obtained zinc vanadate nanopowders **a, c** dried at 60 °C and **b, d** calcined at 400 °C

3.2 Morphology

Table 1 Chemical composition of the prepared material

Element	Compound	Concentration, %
Mg	MgO	0.202
Al	Al ₂ O ₃	0.076
Si	SiO ₂	0.335
P	P ₂ O ₃	0.004
S	SO ₂	0.118
K	K ₂ O ₃	0.005
Ca	CaO	0.047
Ti	TiO ₂	0.010
V	V ₂ O ₅	37.112
Fe	Fe ₂ O ₃	0.036
Zn	ZnO	46.634

The distribution of the particles has a broad bimodal particle distribution with a major size of 519 nm with intensity of 87.8%, whereas a minor particle size distribution of 131.6 nm with 12.2% of the sample.

The amount of the produced OH⁻ ions in the solution acts as a controlling factor for the growth direction. The formed V(OH)⁴⁻ ions in the solution react mostly with the zinc active polar positively charged Zn²⁺ surface rather than the other nonpolar surfaces. This resulted in partially blocking the growth along c-axis [4, 12, 45]. The lateral growth in vanadium zinc oxide Nano flakes (NFs) might be caused by presenting shielding of V(OH)⁴⁻ ions on Zn²⁺ surface. Figure 2a–d shows the SEM and HRTEM images of the dried and calcined samples at 60 and 400 °C, respectively. Figures 2a–b show the SEM images of the zinc vanadate samples dried at 60 °C and calcined at 400 °C, respectively. It is clear that zinc vanadate NFs at low and high temperatures reveals the formation of two-dimensional NFs-like morphologies (Fig. 2a). Its thickness lies in the range of about 50 nm. Upon calcination at 400 °C, the morphology of the produced zinc vanadate powder did not show a significant change (Fig. 2b). However, the flake-like structures showed the formation of small spherical nanoparticles of about 50 nm in size. Figure 2c, d show the HRTEM images of the obtained zinc vanadate nanopowders that dried at 60 and that calcined at 400 °C,

respectively. The dried sample shows the formation of the nanoflakes whereas the formation of quasi-spherical like structures was observed for the calcined sample. Its size in the range of about 30–50 nm which is in agreement with that obtained from XRD and SEM investigations. It could be recognized that the particles are fused together and bottlenecks between the adjacent attachments is also established along the *c* axis and lateral oriented attachment parallel to the *c*-axis is also evident. The formation of nanoflakes could be attributed to the interior layered crystal structure.

Figure 3 shows schematic mechanism of the $\text{Zn}_3(\text{OH})_2\text{V}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ nanoflakes formation, a likely growth mechanism based on self-assembly and Ostwald-ripening processes is suggested. The VO^{3-} ions produced from ammonium meta vanadate react with the Zn^{2+} ions (produced from zinc acetate) to get $\text{Zn}_3(\text{OH})_2\text{V}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$. A defined crystallographic planes are self-assemble jointly in layer-by-layer style by the action of the electrostatic effects [5]. As a final point, the hierarchical $\text{Zn}_3(\text{OH})_2\text{V}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ nanoflakes are constructed as shown in Fig. 3. Increasing the temperature is an important parameter affecting the morphology of samples. Although the surface morphology of the dried sample at 60 °C is similar to the surface morphology of the produced sample after calcination at 400 °C, the composed flakes became thinner with the formation of Nano spheres. As the temperature increases, the removing of the residual nominal amount of H_2O in the hydroxide form occurs resulting in creation of voids (Fig. 3).

3.3 Porous structure evaluation

The pore volume and pore diameters were calculated from surface area analysis (not shown here). The pore volume was found to be 0.1 for the dried sample and 0.08 cc/g for the calcined one. The pore diameter was found to be 10.92 and 12.2 Å for the dried and calcined sample.

3.4 FTIR spectroscopy

The FTIR spectra (Fig. 4a) of the dried and calcined zinc vanadate samples show absorption band at 3486 cm^{-1} due to $-\text{OH}$ stretching frequency of hydroxyl group. The absorption band at 1621 cm^{-1} is assigned to the vibration of water molecule. The absorption band at 1400 cm^{-1} is assigned to the stretching vibration $\text{Zn}-\text{O}$ ($\nu\text{-ZnO}$) bands of tetrahedron unit of vanadate species which confirms the formation of ZnO phase [23]. These peaks diminish after calcination at 400 °C. The absorption bands in the range of $400\text{--}700\text{ cm}^{-1}$ could be attributed to the ZnO stretching modes [3]. The observed peak at 645 cm^{-1} for the calcined sample could be attributed to the ZnO [9]. The bands at $500\text{--}800\text{ cm}^{-1}$ are characteristic of $\text{V}-\text{O}-\text{V}$ vibrations [10]. The absorption bands appear at 771 and 757 cm^{-1} for the dried sample is assigned to $\text{V}-\text{O}$ and $\text{V}-\text{O}-\text{V}$ bonds, respectively [19, 22]. The band for $\text{V}-\text{O}-\text{V}$ is shifted to 792 cm^{-1} in the calcined sample at 400 °C.

Fig. 3 Schematic mechanism of the 2D $\text{Zn}_3(\text{OH})_2\text{V}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ and formation of spherical particles after calcination at 400 °C [54]

Fig. 4 **a** FTIR and **b** Raman spectrum of dried and calcined zinc vanadate nanoflakes

3.5 Raman spectroscopy

Figure 4b shows the Raman spectra in the range of 50–1000 cm^{-1} for the obtained zinc vanadium oxide material. Several peaks were observed in the sample at 68, 96, 148, 260, 314, 370, 801, 849, and 903 cm^{-1} . The peaks in the range of 100–314 cm^{-1} are related to the oxides of V modes and are mostly from the bending and lattice modes of V–O bonds. In lower wave number region, the peak at 320 cm^{-1} corresponds to second order scattering Raman mode ($E_2^{\text{high}}-E_2^{\text{low}}$) where E_2 is non-polar and includes two frequency modes E_2^{high} and E_2^{low} which are associated with the motion of oxygen atoms and zinc sub-lattice in ZnO. The band around 370 cm^{-1} is first-order A1 (LO) mode due to some defects like oxygen vacancies [43, 57]. A1 or E1 modes in Raman spectra indicate the lattice vibrations in crystal lattice or in sample are parallel or perpendicular to the c-axis, respectively. It is also supported by the longitudinal and transverse optical emission of phonons. The significant shift in peak position might be due to E1 (TO). These modes are characteristic of V position. Some Raman peaks are also observed towards lower frequencies at 148 cm^{-1} due to the stretching mode of $(\text{V}_2\text{O}_2)_n$ which corresponds to the chain translation [4]. The peak at 255 cm^{-1} is broad in nature and may be due to V–O–V bending mode [17]. The peak at 315 cm^{-1} could be attributed to triply coordinated oxygen ($\text{V}_3\text{--O}$) bond and stretching band [52]. There are many modes in the wave number range of 750–1000 cm^{-1} arises due to different oxidation states of vanadium [6, 16].

4 Conclusion

In conclusion, zinc vanadate nanoflake composites have been synthesized successfully in one-step simple co-precipitation method. This synthesis method of hierarchical zinc vanadate in high yield levels is possible at mild condition, cost effective direct method could initiate the way for upscale industrial production. The size of the prepared zinc vanadate was in the range of about 30–50 nm as confirmed from FESEM and TEM images for either dried or calcined samples. Upon heating of the hydrated zinc vanadate nanostructures in air at 400 °C, the nanoflakes are formed mainly of nanoparticles.

Funding This study was funded by Egyptian Petroleum Research Institute.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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