Synthesis and properties of $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$

P. Tabero

Received: 15 October 2012 / Accepted: 18 December 2012 / Published online: 22 January 2013
© The Author(s) 2013. This article is published with open access at Springerlink.com

Abstract New phase of a formula $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ ($\text{Fe}_{1-x}\text{V}_{1+x}\text{O}_4$, $x = 0.17$) has been obtained by quenching molten mixture comprising 75 mol% of V_2O_5 and 25 mol% of $\alpha\text{-Fe}_2\text{O}_3$ and subsequent leaching the excessive solid solution of Fe_2O_3 in V_2O_5 with the help of 6 mol solution of NaOH. New phase crystallizes in a monoclinic system with unit cell parameters: $a = 0.9914(3)$ nm, $b = 0.8967(2)$ nm, $c = 0.6852(2)$ nm, $\beta = 107.69(4)^\circ$. It is isostructural with $\alpha\text{-MnMoO}_4$. It was found that at 670°C this phase undergoes a transition to phase of FeVO_4 -I structure. IR spectrum of the $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ has been recorded.

Keywords $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ · DTA · TG · IR · XRD · SEM · EDX

Introduction

Transition metal orthovanadates of general formula MVO_4 , where $M = \text{Cr, Fe, In}$, have been extensively studied because of their potential use as electrode materials in lithium secondary batteries [1–4]. One of the most attractive properties of orthovanadates during the insertion of lithium ions is their high charge insertion capacity. The subject of investigations of numerous works are thin films containing amorphous, nanocrystalline, or well-crystallized orthovanadates [2, 4]. Among others, iron(III) orthovanadate(V) is one of the most frequently investigated [1–4].

The survey of publications has shown that $\text{Fe}_2\text{V}_4\text{O}_{13}$, FeVO_4 , and solid solution of the Fe_2O_3 in V_2O_5 , with solubility limit reaching 3 mol% of Fe_2O_3 , are formed in the two-component system of $\text{Fe}_2\text{O}_3\text{--V}_2\text{O}_5$ [5–13]. The basic properties of $\text{Fe}_2\text{V}_4\text{O}_{13}$ and FeVO_4 are known. The phase $\text{Fe}_2\text{V}_4\text{O}_{13}$ crystallizes in a monoclinic system, space group P21/C [5]. It melts incongruently at 665°C with deposition of FeVO_4 [6]. On the other hand, there are known four polymorphs of iron(III) orthovanadate(V) [5–12]. FeVO_4 -I, crystallizes in a triclinic system (S.G. P-1) [7, 8] and is stable at ambient conditions [6–11]. FeVO_4 -I melts incongruently at 850°C with deposition of $\alpha\text{-Fe}_2\text{O}_3$ as a solid product of melting [6]. Three other metastable polymorphs, the orthorhombic FeVO_4 -II, isostructural with CrVO_4 -III [9], the orthorhombic FeVO_4 -III adopting $\alpha\text{-PbO}_2$ structure [10] and monoclinic, wolframite structure FeVO_4 -IV [11] are normally formed under high pressure and high temperature conditions [6–12]. According to Muller and Joubert [11], FeVO_4 -III and FeVO_4 -IV were obtained directly by the high pressure, high temperature synthesis. On the other hand, orthorhombic FeVO_4 -II was obtained from FeVO_4 -III around 540°C , and has narrow range of stability, reaching 570°C [11]. The metastable FeVO_4 -II can be obtained also by hydrothermal method [9]. Muller and Joubert [11] suggest additionally that FeVO_4 -III is a reduced form of composition $\text{Fe}_{1-x}\text{V}_{1+x}\text{O}_4$ containing some tetravalent vanadium. The results of thermogravimetric (TG) investigations have shown that FeVO_4 prepared using solution method corresponds to amorphous phase of the $\text{FeVO}_4 \cdot 2.7\text{H}_2\text{O}$ formula [3]. The X-ray data of the amorphous phase annealed at 500°C show the beginning of crystallization, yielding well-crystallized triclinic FeVO_4 -I after annealing at 600°C . On the other hand, the studies conducted with the help of transmission electron microscopy and focused on preparation of the FeVO_4 thin films

P. Tabero (✉)
Department of Inorganic and Analytical Chemistry, Faculty
of Chemical Engineering, West Pomeranian University of
Technology, Szczecin, Al. Piastow 42, 71-065 Szczecin, Poland
e-mail: ptab@zut.edu.pl

have revealed that amorphous iron(III) orthovanadate(V) after thermal treatment at 300 °C transforms to orthorhombic FeVO₄-II, and next after sintering at 500 °C it transforms to triclinic FeVO₄-I [2].

The literature survey has shown as well that in the FeVO₄-CrVO₄ system solid solution of general formula Fe_{1-x}Cr_xVO₄, for 0.1 < *x* < 0.85, forms, which adopts α-MnMoO₄ structure [13, 14]. Thus, the structure of the Fe_{1-x}Cr_xVO₄ phase differs essentially from the stable at ambient conditions structures of triclinic FeVO₄-I and orthorhombic CrVO₄-III. On the other hand, soft chemistry synthesis methods lead to formation of CrVO₄-I and InVO₄-I isostructural with α-MnMoO₄ [15, 16].

Despite numerous works concerning the Fe₂O₃-V₂O₅ system, the investigations conducted lately in Department of Inorganic and Analytical Chemistry and focused on verification of the literature data concerning properties of FeVO₄ have revealed the presence of new phase in its melting products. Powder diffraction patterns of molten and quenched samples are frequently very complex and contain diffraction lines which are shifted, broaden, overlapped, and weak. It makes identification of single phases in such samples very difficult. Despite potential problems, the aim of this study was to obtain this new phase, denoted as X phase, and to investigate its properties with the help of XRD, DTA/TG, SEM/EDX, and IR methods. Results of investigations by XRD, DTA/TG, SEM/EDX, and IR methods allow a determination of composition of samples and establishing the range of their thermal stability [17–20].

Experimental

The materials used in the research were V₂O₅, p.a. (POCh, Poland), α-Fe₂O₃ p.a. (POCh, Poland), NaOH p.a. (POCh, Poland), and made in-house FeVO₄-I. FeVO₄-I was synthesized from stoichiometric mixture of oxides in the following stages: 560 °C (20 h) + 590 °C (20 h) × 2 [17, 18].

The DTA/TG examinations were made using an apparatus of Paulik–Paulik–Erdey type (MOM, Hungary). Samples of 500 mg were investigated in air up to the 1,000 °C in quartz crucibles and at the heating rate of 10 °C min⁻¹.

X-ray phase analysis (XRD) of the samples was performed using a HZG-4 diffractometer (Carl Zeiss, Germany) applying the Co Kα/Fe radiation (step 0.02° 2θ, time 1 s). The identification of the individual phases was performed on the ground of their characteristics contained in PDF cards. The powder diffraction pattern of the new phase was indexed with the help of the Refinement program of DHN/PDS package, using α-Al₂O₃ as the internal standard.

The density of the new phase was determined in argon with the help of the Ultrapytc 1200e ultrapycnometer (Quantachrome Instruments, USA).

The investigations by SEM/EDX methods were carried out by using of an electron scanning microscope (JSM-1600, Jeol, Japan) matched with an X-ray energy dispersive spectrometer (ISIS-300, Oxford Instruments).

The investigations with made in-house FeVO₄-I were conducted using fine ground samples subjected to heat treatment in different conditions. For the further experiments were selected five samples containing 10, 25, 33, 67, and 75 mol% of Fe₂O₃ in mixture with V₂O₅. The oxides weighted in an appropriate proportions after grinding in an agate mortar were heated at 900 °C for 3 h, quenched to room temperature, and subjected to X-ray phase analysis (XRD).

The single phase sample of the new phase X was obtained using mixture of oxides containing 25 mol% of α-Fe₂O₃ and 75 mol% of V₂O₅. A mixture of 7.736 g of V₂O₅ and 2.264 g of α-Fe₂O₃ was heated in crucible at 900 °C for 3 h and quenched to room temperature yielding a brown solid. After fine grinding in agate mortar, the excessive amounts of V₂O₅ were removed from solidified melt by leaching with 100 ml of 6 mol solution of NaOH. Next, the obtained solid was filtered using paper filter, washed several times with distilled water and dried at 110 °C for 4 h in drying oven. The phase composition of prepared phase was controlled with the help of XRD method. The dark brown solid obtained according to this procedure, the phase X, was subjected to further investigations.

Results and discussion

In order to obtain new phase, denoted as X phase, two series of experiments were conducted. In the first stage of research, experiments were concerning the heat treatment of FeVO₄ in different conditions. At the beginning, the sample of FeVO₄ was subjected to investigation with the help of DTA method. On the DTA curve of FeVO₄ one endothermic effect was recorded, with onset temperature at 850 °C, and maximum temperature at 900 °C, attributed to melting of this phase. These temperatures are in accord with the literature data [6]. In order to know the products of FeVO₄ melting, sample of FeVO₄ was heated for 3 h at 900 °C that is at temperature of maximum of endothermic effect attributed to its melting, and quenched to room temperature. The results of the X-ray phase analysis of this sample after heat treatment (Table 1, no. 2) have shown that besides V₂O₅ and α-Fe₂O₃ this sample contains phase X. The diffraction lines characteristic for V₂O₅ recorded on powder diffraction pattern of this sample were shifted

Table 1 Results of X-ray phase analysis of FeVO_4 samples heat treated in different conditions

No.	Temperature of heating/°C	Time of heating/h	Treatment after heating	Phases identified after heat treatment
1	1,000	3	Quenched	X, $\text{V}_2\text{O}_{5(\text{ss})}$, $\alpha\text{-Fe}_2\text{O}_3$
2	900	3	Quenched	X, $\text{V}_2\text{O}_{5(\text{ss})}$, $\alpha\text{-Fe}_2\text{O}_3$
3	900	3	Cooled slowly in furnace	$\text{FeVO}_4\text{-I}$, $\text{V}_2\text{O}_{5(\text{ss})}$, $\alpha\text{-Fe}_2\text{O}_3$
4	800	3	Quenched	$\text{FeVO}_4\text{-I}$

Table 2 Results of X-ray phase analysis of samples containing mixture of oxides of different compositions heated at 900 °C for 3 h and quenched to room temperature

No.	Composition of initial mixtures/mol%		Phases identified after quenching
	V_2O_5	$\alpha\text{-Fe}_2\text{O}_3$	
1	25	75	X, $\text{V}_2\text{O}_{5(\text{ss})}$, $\alpha\text{-Fe}_2\text{O}_3$
2	33	67	X, $\text{V}_2\text{O}_{5(\text{ss})}$, $\alpha\text{-Fe}_2\text{O}_3$
3	50	50	X, $\text{V}_2\text{O}_{5(\text{ss})}$, $\alpha\text{-Fe}_2\text{O}_3$
4	67	33	X, $\text{V}_2\text{O}_{5(\text{ss})}$, $\alpha\text{-Fe}_2\text{O}_3$
5	75	25	X, $\text{V}_2\text{O}_{5(\text{ss})}$
6	90	10	$\text{Fe}_2\text{V}_4\text{O}_{13}$, $\text{V}_2\text{O}_{5(\text{ss})}$

slightly towards higher 2θ angles. As described in the literature, it indicated formation of solid solution of Fe_2O_3 in V_2O_5 [6], denoted in this work as a $\text{V}_2\text{O}_{5(\text{ss})}$. Moreover, at 900 °C V_2O_5 does not exist as a solid, thus it crystallizes from liquid, and cannot be treated as a solid product of melting.

The results of a X-ray qualitative phase analysis of FeVO_4 samples heat treated in different conditions are given in Table 1. Analysis of these data permits advancing suggestion, in order to obtain phase X, samples have to be heated at temperatures higher than the temperature of melting of FeVO_4 (at 900 or 1,000 °C) and quenched to room temperature. Based on these conclusions, in the second stage of investigations samples were heated at 900 °C for 3 h and quenched to room temperature.

In the second stage of research, several mixtures comprising V_2O_5 and $\alpha\text{-Fe}_2\text{O}_3$ were heated at 900 °C for 3 h and quenched to room temperature. The compositions of samples and results of X-ray qualitative phase analysis after heat treatment are given in Table 2.

The measurement results shown in Table 2 have given rise to statement that only sample of initial composition of 75 mol% of V_2O_5 and 25 mol% of Fe_2O_3 after quenching

Table 3 The result of indexing of X-ray powder diffraction pattern of the $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$

No.	d_{exp}/nm	$d_{\text{calc}}/\text{nm}$	hkl	100 I
1	2	3	4	5
1	0.6533	0.6528	001	3
2	0.6492	0.6503	110	10
3	0.3691	0.3696	021	7
4	0.3369	0.3371	201	6
5	0.3261	0.3264	002	76
6	0.3250	0.3251	220	100
7	0.3198	0.3198	−112	32
8	0.3171	0.3174	−202	18
9	0.3053	0.3054	−311	20
10	0.2700	0.2699	112	30
11	0.2639	0.2639	022	7
12	0.2592	0.2590	−222	19
13	0.2361	0.2361	400	13
14	0.2242	0.2242	040	9
15	0.2209	0.2207	−113	6
16	0.2174	0.2176	003	26
17	0.2168	0.2168	330	42
18	0.2096	0.2095	222	7
19	0.2025	0.2025	240	14
20	0.2013	0.2014	−223	7

contains a new phase X and solid solution of Fe_2O_3 in V_2O_5 . This solid solution was removed from sample by leaching with 6 mol. NaOH solution yielding pure phase X. The powder diffraction pattern of the X phase was subjected to indexing of which results are given in Table 3. Phase X crystallizes in the monoclinic system and its end-centered (C) unit cell parameters are given in Table 4. The literature scan has shown that angular positions of diffraction reflection, their relative intensities and unit cell parameter of phase X are similar to these ones of $\text{Fe}_{1-x}\text{Cr}_x\text{VO}_4$ solid solution phase, $\text{CrVO}_4\text{-I}$ and $\text{InVO}_4\text{-I}$, which are isostructural with $\alpha\text{-MnMoO}_4$ [13–16]. It permits advancing suggestion, that phase X belongs to a group of orthovanadates, MVO_4 , isostructural with $\alpha\text{-MnMoO}_4$.

The sample of initial composition of 75 mol% of V_2O_5 and 25 mol% of Fe_2O_3 heated at 900 °C for 3 h and quenched to room temperature and containing a mixture of a new phase X and solid solution of Fe_2O_3 in V_2O_5 was subjected to investigations with the help of an electron scanning microscope matched with an X-ray energy dispersive spectrometer. A SEM image has revealed that in the surface layer of this sample needle-like crystals are prevailing, with their lengths confined in the range of 0.2–1 mm (Fig. 1). The crystallites from deeper layers were smaller and more irregular in shape (Fig. 1). The results of experimental determination of composition by

Table 4 Unit cell parameters and volumes of $\text{InVO}_4\text{-I}$ [16], $\text{Fe}_{0.75}\text{Cr}_{0.25}\text{VO}_4$ [13], $\text{Fe}_{0.2}\text{Cr}_{0.8}\text{VO}_4$ [13], $\text{CrVO}_4\text{-I}$ [15], and $\text{Fe}_{1-x}\text{V}_{1-x}\text{O}_4$

No.	Formula	a/nm	b/nm	c/nm	$\beta/^\circ$	V/nm^3
1	$\text{InVO}_4\text{-I}$	1.0271(1)	0.9403(1)	0.7038(1)	105.08(1)	0.6563(1)
2	$\text{Fe}_{1-x}\text{V}_{1-x}\text{O}_4$	0.9914(3)	0.8967(2)	0.6852(2)	107.69(4)	0.5803(3)
3	$\text{Fe}_{0.75}\text{Cr}_{0.25}\text{VO}_4$	0.9892(3)	0.8947(2)	0.6853(2)	107.61(4)	0.5781(2)
4	$\text{Fe}_{0.2}\text{Cr}_{0.8}\text{VO}_4$	0.9812(5)	0.8857(4)	0.6839(4)	107.75(6)	0.5660(4)
5	$\text{CrVO}_4\text{-I}$	0.9765(1)	0.8818(3)	0.68224(9)	107.62(1)	0.5599(1)

EDX analysis of needle-like crystals showed that the Fe:V ratio is near to 10:14 (on average: 41.52 at.% Fe and 58.48 at.% V) and it corresponds to phase X. Taking under consideration orthovanadate formula MVO_4 , it gives $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ ($\text{Fe}_{1-x}\text{V}_{1+x}\text{O}_4$, for $x = 0.17$). It resembles the case of $\text{FeVO}_4\text{-III}$, where the partial reduction of vanadium ions leads to formula $\text{Fe}_{1-x}\text{V}_{1+x}\text{O}_4$ [11].

On the other hand, the results of experimental determination of composition of crystallites from deeper layers have revealed the presence of two phases: phase X and solid solution of Fe_2O_3 in V_2O_5 . The results of experimental determination of composition of solid solution showed that the V:Fe ratio is close to 24:1 (on average 96.16 at.% V and 3.84 at.% Fe) and is in accord with the literature data [6].

The measured density of $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ amounts to $d_{\text{exp}} = 3.94(5) \text{ g cm}^{-3}$, and is in accord with XRD density $d_c = 3.89 \text{ g cm}^{-3}$ calculated for eight asymmetric units in the unit cell ($Z = 8$, characteristic for $\alpha\text{-MnMoO}_4$ structure).

The sample of pure phase $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ was additionally examined by DTA and TG methods. Figure 2 shows DTA curve of the $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ phase, which contains two effects up to 1,000 °C. The first effect recorded on DTA

curve is weak and exothermic, with onset temperature at 670 °C. This effect is accompanied by very weak, equal to 0.4 %, mass gain recorded on TG curve. The second effect registered on DTA curve is endothermic, with onset temperature at 850 °C.

In order to define the nature of the process connected with the exothermic effect recorded on DTA curve sample of $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ phase was heated for 1 h at 700 °C that is at temperature of maximum of the exothermic effect, and quenched to room temperature. Figure 3 shows fragments of powder diffraction patterns of phase $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ (curve a), phase $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ heated for 1 h at 700 °C (curve b), and pure phase $\text{FeVO}_4\text{-I}$ (curve c). The powder diffraction pattern of $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ sample after heat treatment at 700 °C for 1 h (Fig. 2, curve b) contains only the set of diffraction lines characteristic for $\text{FeVO}_4\text{-I}$. It means that at 670 °C $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ phase undergoes a transition to phase of $\text{FeVO}_4\text{-I}$ structure. The distinctly pronounced endothermic effect registered on DTA curve of $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ with beginning temperature at 850 °C now can be undoubtedly attributed to the melting of phase of $\text{FeVO}_4\text{-I}$ structure. Small mass gain accompanying

Fig. 1 A SEM image of a sample of initial composition of 75 mol% of V_2O_5 and 25 mol% of Fe_2O_3 heated at 900 °C for 3 h and quenched to room temperature. Magnification $\times 50$ **Fig. 2** Fragment of DTA curve (a) and TG curve (b) of $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ phase

Fig. 3 Fragments of powder diffraction patterns of phase $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ (curve a), phase $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ heated for 1 h at 700 °C (curve b), and pure phase $\text{FeVO}_4\text{-I}$ (curve c)

transition from $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ to the phase of $\text{FeVO}_4\text{-I}$ structure is probably connected with oxidation of V^{4+} , V^{3+} , or Fe^{2+} ions occupying some of the positions in the crystal lattice of $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$. On this stage of investigations, one cannot exclude, however, the presence of oxygen vacancies in the crystal lattice of the phase under study. This problem will be the subject of further investigations.

The samples representing selected stages of $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ phase synthesis were subjected to an investigation with the use of infra-red spectroscopy (IR). Figure 4 shows IR spectra of initial mixture containing 75 mol% of V_2O_5 and 25 mol% of Fe_2O_3 (curve a), sample after heating at 900 °C for 3 h and quenched to room temperature (curve b), pure $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ phase (curve c), sample obtained after heating of $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ at 700 °C for 1 h (curve d), and pure $\text{FeVO}_4\text{-I}$ (curve e). IR spectrum of initial mixture containing 75 mol% of V_2O_5 and 25 mol% of Fe_2O_3 (curve a) reflects mainly IR absorption bands characteristic for V_2O_5 . On the other hand, intensity of absorption bands characteristic for V_2O_5 in sample after heating at 900 °C for 3 h and quenched to room temperature (curve b) decrease considerably, what is accompanied with appearance of some new bands characteristic for $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ phase. IR spectrum of $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ phase given for the first time in this work (curve c) reveals the presence of absorption bands with their maxima at 946, 918, 826, 710, 648, 488, and 372 cm^{-1} . Absorption bands covering wave number region of 1,000–550 cm^{-1} can be attributed to stretching vibrations of V–O bonds in VO_4 tetrahedra [2, 21, 22]. Next absorption

Fig. 4 IR spectra of initial mixture containing 75 mol% of V_2O_5 and 25 mol% of Fe_2O_3 (curve a), sample after heating at 900 °C for 3 h and quenched to room temperature (curve b), pure $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ phase (curve c), sample obtained after heating of $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ at 700 °C for 1 h (curve d), and pure $\text{FeVO}_4\text{-I}$ (curve e)

bands recorded in the range of 550–300 cm^{-1} can be the most likely ascribed to stretching vibrations of Fe–O bonds in MO_6 octahedra and bending vibrations of M–O–M bridges [2, 21, 22]. Despite some similarities this spectrum differs from IR spectrum of $\text{FeVO}_4\text{-I}$ (curve e) what reflects difference in the crystal structure of both compounds. On the other hand, IR spectra of $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ phase heated at 700 °C for 1 h (curve d) and $\text{FeVO}_4\text{-I}$ (curve e) are very similar. As the both samples contain phases of $\text{FeVO}_4\text{-I}$ structure, it corroborates the results of X-ray qualitative phase analysis indicating that $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ phase transforms at 670 °C to phase of $\text{FeVO}_4\text{-I}$ structure.

Conclusions

In this work, the results concerning the preparation and properties of a new $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ phase were presented for the first time. The XRD, DTA/TG, SEM/EDX, and IR measuring techniques were used to study properties of this phase. $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ is a dark brown solid which density equals to 3.94 g cm^{-3} . It crystallizes in a monoclinic system and is isostructural with $\alpha\text{-MnMoO}_4$. At 670 °C $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ undergoes an irreversible transition to a phase of $\text{FeVO}_4\text{-I}$ structure. Despite some similarities, IR spectrum of $\text{Fe}_{0.83}\text{V}_{1.17}\text{O}_4$ differs from IR spectrum of $\text{FeVO}_4\text{-I}$ what reflects differences in the crystal structure of both compounds.

Open Access This article is distributed under the terms of the Creative Commons Attribution License which permits any use, distribution, and reproduction in any medium, provided the original author(s) and the source are credited.

References

- Cheng F, Chen J. Transition metal vanadium oxides and vanadate materials for lithium batteries. *J Mater Chem*. 2011;21:9841–8.
- Surca-Vuk A, Orel B, Drazic G, Colomban P. Vibrational spectroscopy and analytical electron microscopy studies of Fe–V–O and In–V–O thin films. *Monatsh Chem*. 2001;133:889–908.
- Denis S, Baudrin E, Touboul M, Tarascon J-M. Synthesis and electrochemical properties of amorphous vanadates of general formula RVO_4 ($R = \text{In, Cr, Fe, Al, Y}$) vs. Li. *J Electrochem Soc*. 1997;144(12):4099–109.
- Ali EB, Bernede JC, Ray PL. Vanadium transition metal oxide films obtained by annealing under room atmosphere of metal layers sequentially deposited. *J Cryst Growth*. 2000;208:471–81.
- Permer D, Lalignat Y. Crystal structure of the tetrapolyvanadate $Fe_2V_4O_{13}$. *Eur J Solid State Inorg Chem*. 1997;34:41–52.
- Walczak J, Ziolkowski J, Kurzawa M, Osten-Sacken J, Lysio M. Studies on Fe_2O_3 – V_2O_5 system. *Pol J Chem*. 1985;59:255–61.
- Levinson LM, Wanklyn BM. Crystal growth and magnetic behaviour of $FeVO_4$. *J Solid State Chem*. 1971;3:131–3.
- Robertson B, Kostiner E. Crystal structure and Mössbauer effect investigation of $FeVO_4$. *J Solid State Chem*. 1972;4:29–37.
- Oka Y, Yao T, Yamamoto N, Ueda Y, Kawasaki S, Azuma M, Takano M. Hydrothermal synthesis, crystal structure, and magnetic properties of $FeVO_4$ -II. *J Solid State Chem*. 1996;123:54–9.
- Laves F. On the high-pressure form of $FeVO_4$. *Acta Cryst*. 1964;17:14761477.
- Muller J, Joubert JC. Synthèse sous haute pression d'oxygène d'une forme dense ordonnée de $FeVO_4$ et mise en évidence d'une variété allotropique de structure $CrVO_4$. *J Solid State Chem*. 1975;14:8–13.
- Young AP, Schwartz CM. High pressure forms of $CrVO_4$ and $FeVO_4$. *Acta Cryst*. 1962;15:1305.
- Atfield JP. The structure of the solid solutions formed in the system $(Cr_xFe_{1-x})VO_4$. *J Solid State Chem*. 1987;67:58–63.
- Atfield JP, Cheetham AK, Johnson DC, Novet T. Magnetic frustration spirals and short-range order in $Cr_xFe_{1-x}VO_4$ -I solid solutions. *J Mater Chem*. 1991;1(5):867–73.
- Touboul M, Denis S, Segui L. Crystal structure of a new form of chromium(III) vanadate(V), $CrVO_4$ -I. *Eur J Solid State Inorg Chem*. 1995;32:577–88.
- Touboul M, Melghit K, Bernard P, Louer D. Crystal structure of a metastable form of indium orthovanadate, $InVO_4$ -I. *J Solid State Chem*. 1995;118:93–8.
- Blonska-Tabero A. New phase in the system $FeVO_4$ – $Cd_4V_2O_9$. *J Therm Anal Calorim*. 2008;93:707–10.
- Blonska-Tabero A. $Pb_2Fe_2V_4O_{15}$ —a new phase forming in the system $FeVO_4$ – $Pb_2V_2O_7$. *J Alloys Compd*. 2010;508:42–6.
- Bosacka M, Filipek E, Šulcova P, Dohnalová Ž, Paczesna A. Phase equilibria in the solid state and colour properties of the CuO – In_2O_3 system. *J Therm Anal Calorim*. 2012;109:605–10.
- Blonska-Tabero A, Bosacka M, Dabrowska G, Filipek E, Piz M, Rychłowska-Himmel I, Tabero P, Tomaszewicz E. The synthesis and properties of the phases obtained by solid–solid reactions. *J Min Metall*. 2008;44B:19–26.
- Dabrowska G, Tabero P, Kurzawa MJ. Phase relations in the Al_2O_3 – V_2O_5 – MoO_3 system in the solid state, the crystal structure of $AlVO_4$. *J Phase Equilib Diffusion*. 2009;30(3):220–9.
- Tabero P. Formation and properties of the new $Al_8V_{10}W_{16}O_{85}$ and $Fe_{8+x}Al_4V_{10}W_{16}O_{85}$ phases with M – Nb_2O_5 structure. *J Therm Anal Calorim*. 2010;101:561–6.