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$CrVMoO_7$: MICROWAVE SYNTHESIS AND STRUCTURAL CHARACTERIZATION

Gülşah Çelik Gül [1]
[1]University of Balıkesir, 10145, Balıkesir, Turkey

Abstract: In this study, microwave synthesis method was operated to obtain $CrVMoO_7$. Structural characterization of the compound was realized by powder X-ray diffraction (XRD) and Fourier transform infrared spectrometry (FTIR). Morphological property and elemental composition were determined via scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDS). Thermal nature of the sample was identified by thermogravimetric analyzer (TGA).

Keywords: $CrVMoO_7$; X-ray diffraction; Rietveld refinement method; Microwave synthesis.

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***Corresponding author**. E-mail: gulsahcelik9@gmail.com, Tel.: +90 266 612 1067/2012.

INTRODUCTION

Catalysis for most industrial processes has significance due to saving time, energy, and money. The catalysts used in these processes are generally transition metal oxides which are applied either alone or mixtures. The basic mixtures contain V_2O_5 and $MoO_3/WO_3/Fe_2O_3/Cr_2O_3$. Among them, Cr_2O_3–V_2O_5–MoO_3 three-component system has been studied in early times by many researchers (1-4). The formula $CrVMoO_7$ as unknown phase has occurred by the reaction of Cr_2O_3–V_2O_5–MoO_3 system in the solid-state form (5-7). The properties of this phase have been limitedly known , except melting point at 820 °C to form solid Cr_2O_3 (2). According to the other investigations, the related phase has been formed via incorporation of MoO_3 into the $Cr_2V_4O_{13}$ lattice (6, 7). It has not been examined in previous reports what the nature of $CrVMoO_7$ is and how the structure occurs (3, 8, 9). There are only a few documents about crystallographic morphology and thermal properties of $CrVMoO_7$. The prominent one is about indexing of powder X-ray diffraction pattern and calculation of unit cell parameters of $CrVMoO_7$ resulting a=5.53346 Å, b=6.58901 Å and c=7.86551 Å in triclinic system (8). The infrared spectrum of the compound point out that VO_4, MoO_4 and CrO_6 subgroups exist in the structure (10, 11). As a result of these, to the best of our knowledge, microwave synthesis, crystallographic, morphologic and thermal properties of $CrVMoO_7$ have been studied for the first time with this paper which has not been reported previously.

MATERIALS AND METHODS

Cr_2O_3, V_2O_5 and MoO_3 compounds have been used as analytical grade and supplied by Merck. Oxide types starting materials have been weighed in 0.5:0.5:1 molar ratio and ground in an agate mortar followed by microwave treatment in a domestic microwave oven (2.45 GHz, 850 W power) for 20 min. The final sample has been washed three times with hot pure water and ethanol. The washed material has been treated at 400 °C for 2 hours to get the best crystals.

The powder X-ray diffraction (XRD) measurement has been completed by Panalytical X'Pert Pro Diffractometer and CuK_α radiation (λ=1.54056 Å, 40 mA, 50 kV) with a scan rate of 1°/min with a step size 0.02°. The Rietveld analysis of the sample has been calculated by using powder

diffraction data via High Score Plus (HS+) Program (License number: 92000029). Fourier transform infrared spectrum (FTIR) has been formed on a Perkin Elmer Spectrum 100 FTIR Spectrometer from 4000 to 650 cm^{-1}. Scanning electron microscopy/energy dispersive X-ray analysis has been achieved in SEM JEOL 6390-LV/EDX. Thermal property of the sample has been checked by Perkin Elmer thermogravimetric analyzer TGA. A Siemens V12 domestic microwave oven has been used as the microwave source.

RESULTS AND DISCUSSION

Figure 1 displays the XRD pattern of the synthesized material. The XRD pattern of the sample corresponds to $CrVMoO_7$ with the ICSD card number 008-5712 (Fig. 2). The unit cell parameters of the observed diffraction data has been calculated by Rietveld Refinement Program. The findings are completely in accordance with database given in Table 1. There is no other phase as an impurity or starting material.

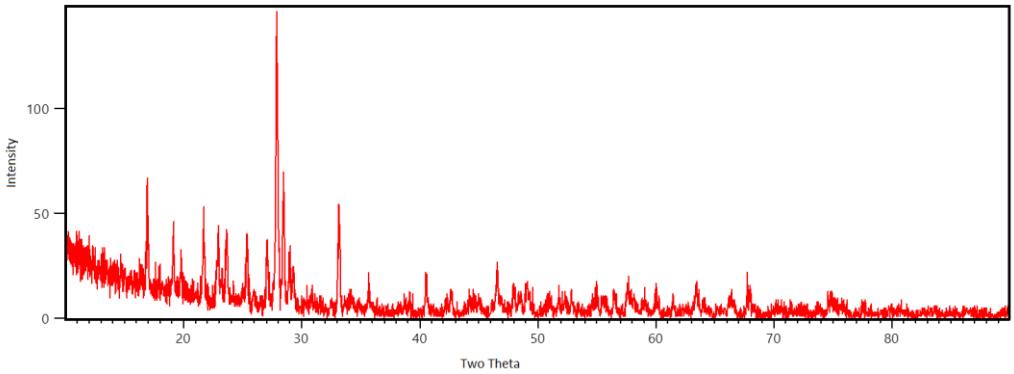

Figure 1. The XRD pattern of $CrVMoO_7$.

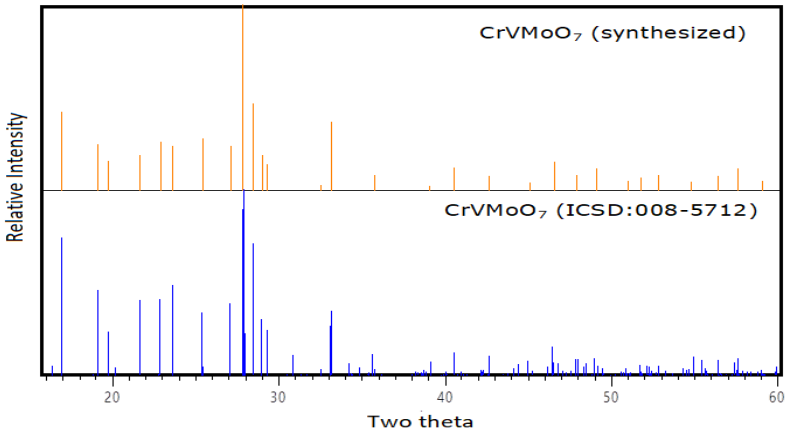

Figure 2. The comparison of powder XRD pattern between $CrVMoO_7$ (ICSD:008-5712) and $CrVMoO_7$ (synthesized).

Table 1. The comparison of unit cell parameters calculated and database values.

Compound	**Unit cell parameters**		
	a (Å)	b (Å)	c (Å)
$CrVMoO_7$ (ICSD:008-5712)	5.5310	6.5850	7.8640
$CrVMoO_7$ (synthesized)	5.5253	6.5792	7.8532

Figure 3 shows FTIR spectrum of $CrVMoO_7$. The four wavenumbers in the range of 600-1000 cm^{-1} correspond to vibrations of M–M and Mo–O bonds (12-14).

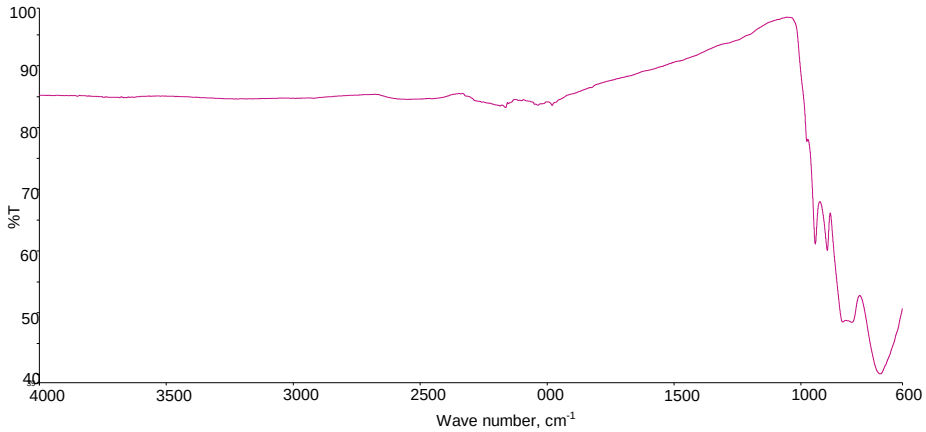

Figure 3. The FTIR spectrum of $CrVMoO_7$.

Figure 4 exhibits the SEM micrograph of $CrVMoO_7$. The homogeneous view of the sample confirm the formation of chromium vanadium molybdate. The particle size distribution of $CrVMoO_7$ is in 2-5 μm.

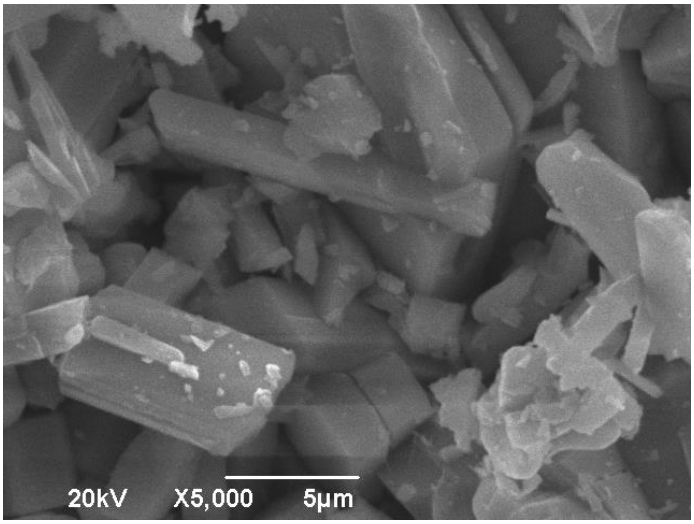

Figure 4. The SEM image of $CrVMoO_7$.

The EDS graph of $CrVMoO_7$ is given in Figure 5. The elemental composition of the compounds has been calculated as 3:3:3.2:6.8 by EDS results which are in accordance with the molecular formula.

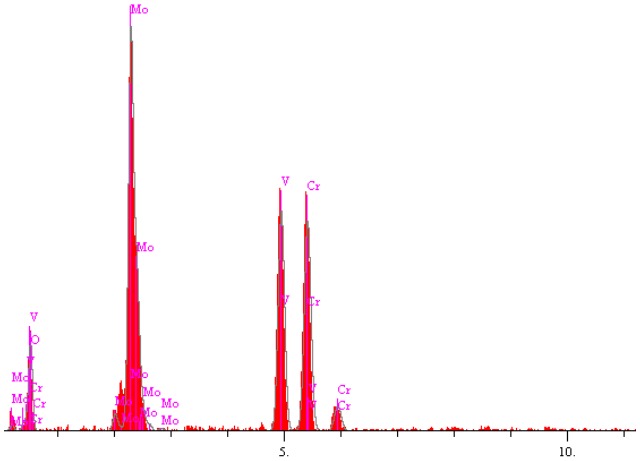

Figure 5. The EDS graph of $CrVMoO_7$.

The graph of thermogravimetric analysis is represented in Figure 6. The first smaller thermal loss starts nearly at 700 °C, and the other is in the range of 1050–1200 °C. The material lost totally 40% of its mass.

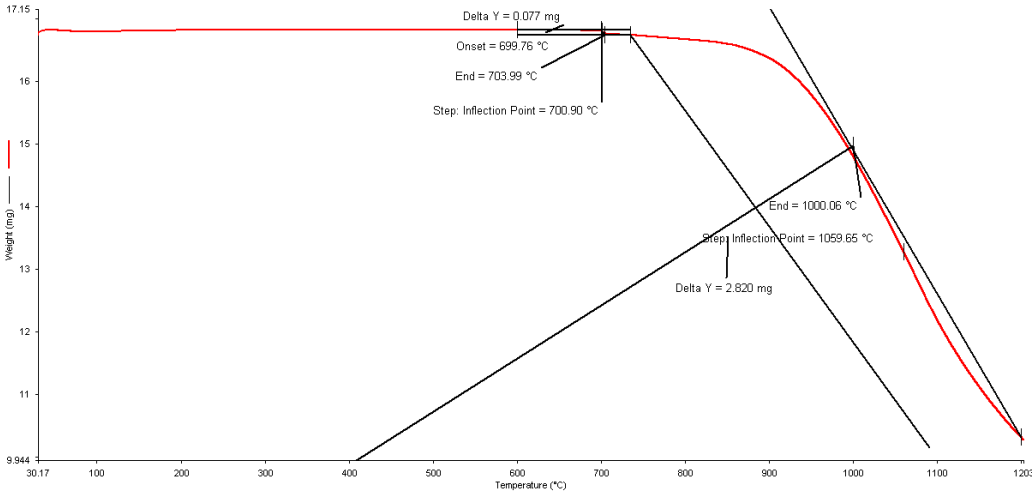

Figure 6. TGA thermograph of $CrVMoO_7$.

As a conclusion, $CrVMoO_7$ has been synthesized for the first time with microwave method at 850 W power for 20 minutes. The unit cell parameters of the compound have been calculated by Rietveld Refinement Program benefiting from XRD data. The homogeneous morphology and similar elemental composition have been confirmed via SEM and EDS results. High thermal stability of the compound has been determined from TGA.

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