

# Inventive Synthesis and Characterization of a Cesium Molybdate Compound: CsBi(MoO<sub>4</sub>)<sub>2</sub>

F. Kurtuluş, G. Çelik Gül

**Abstract**—Cesium molybdates with general formula CsM<sup>III</sup>(MoO<sub>4</sub>)<sub>2</sub>, where M<sup>III</sup> = Bi, Dy, Pr, Er, exhibit rich polymorphism, and crystallize in a layered structure. These properties cause intensive studies on cesium molybdates.

CsBi(MoO<sub>4</sub>)<sub>2</sub> was synthesized by microwave method by using cerium sulphate, bismuth oxide and molybdenum (VI) oxide in an appropriate molar ratio. Characterizations were done by x-ray diffraction (XRD), fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy/energy dispersive analyze (SEM/EDS), thermo gravimetric/differential thermal analysis (TG/DTA).

**Keywords**—Cesium bismuth dimolybdate, microwave synthesis, powder x-ray diffraction, rare earth dimolybdates.

## I. INTRODUCTION

THE bismuth-containing oxides are promising candidates for developed applications in catalysis, optics, nano-electronics and nano-photonics via their rich structural diversity and promising physical properties [1]–[7]. The bismuth molybdates, such as Bi<sub>2</sub>Mo<sub>3</sub>O<sub>12</sub>, Bi<sub>2</sub>Mo<sub>2</sub>O<sub>9</sub>, and Bi<sub>2</sub>MoO<sub>6</sub>, are known as their high electric and photoconductive property, as well their high photocatalytic and catalytic activity [8]–[16]. In addition, bismuth molybdates have been evaluated as potential gas sensors [17]–[19] and optical materials [20], [21].

Molybdates are existing source for laser applications, ferroelectrics, scintillators, nonlinear optical, and other functional materials [22]–[28]. The number of complex compounds containing Mo(VI) and oxygen ions has been expanding over the past two decades, essentially, on account of triple molybdates containing MoO<sub>4</sub> tetrahedra and two kinds of cations [29]–[35]. Complex molybdates containing alkaline elements are characterized by structural disorder of cations representing the possibility of the ionic conductivity in these compounds [36], [37]. The significant ionic conductivity of some double molybdates containing of alkaline metals (Na, Li, K) and two or three valent metals (Cd, Zn, Cu, Co, Ni, Mg, Fe, Ga, Sc, In, Bi) caused by alkaline cations was observed earlier [38]–[40]. The polymorphic transitions of molybdates resulting a crystalline state with a high ionic conductivity were defined in these previous studies. These types of molybdates are usually crystallized in three-dimensional mixed crystal structures [41]–[44]. Complex molybdates due to these

structural features are believed to have a high electronic–ionic conductivity [45].

Rare earth dimolybdates represented with general formula M<sup>I</sup>M<sup>II</sup>(MoO<sub>4</sub>)<sub>2</sub> (M<sup>I</sup> = K, Cs; M<sup>II</sup> = Bi, Dy, Pr, Er), have been created a common subject of depth studies due to their crystal diversity [46]–[50]. The layered crystal structure of these molybdates, lattice instability resulting from the electron-phonon coupling and orbital degeneracy has been identified to generate phase transition variety [51].

Double molybdates of alkali and trivalent metals are well known in literature and belong to the most numerous and studied family of molybdates having important properties and applications [52].

Some double molybdates of bismuth and monovalent metals, such as MBi(MoO<sub>4</sub>)<sub>2</sub> (M=Li–Cs, Ag) and M<sub>5</sub>Bi(MoO<sub>4</sub>)<sub>4</sub> (M=K, Cs, Tl), show interesting physical properties and may be considered as valuable ferroelastics and ferroelectrics [53], solid-state electrolytes, [54]–[56] optical materials [57], [58], phosphors [59] and laser-host materials [60]–[65].

One of the common featured compounds is CsBi(MoO<sub>4</sub>)<sub>2</sub> crystallized in the  $D_{2h}^3$  crystal structure with unit cell parameters  $a = 9.45$ ,  $b = 8.21$ ,  $c = 5.14$  Å in orthorhombic system and show the phase transition at 125, and 325 K [66], [67]. The transition at 325 K is of second-order and the crystal shows antiferroelectric properties below the related temperature. The lower transition is of the first-order and not cleared up yet [47]. Until now solid-phase synthesis and flux crystallization techniques were developed for the most of the double molybdates of alkali and trivalent metals; their crystal structures, polymorphism features, and some physical properties [68]–[70] were investigated.

The crystalline form of the CsBi(MoO<sub>4</sub>)<sub>2</sub> was synthesized by conventional high temperature method many times in the past [71]–[74]. The related process is expensive, harmful for the nature, time and energy consuming. In terms of an alternative to conventional route, microwave synthesis method is much more environmentally, cheap, practical and less energy consumptive. In the present paper, we are going to explain the microwave synthesis, crystal structure, morphology and thermal behavior of CsBi(MoO<sub>4</sub>)<sub>2</sub>.

## II. EXPERIMENTAL PROCEDURE

### A. Synthesis of CsBi(MoO<sub>4</sub>)<sub>2</sub>

The reactants were supplied by Merck Company as analytically pure. Cerium sulphate, bismuth oxide, and molybdenum (VI) oxide were measured in an appropriate molar ratio (1:0.5:2). The starting materials were grounded in

F. Kurtuluş and G. Çelik Gül are with the Chemistry Department, University of Balıkesir, Balıkesir, 10145 Turkey (phone: +90-266-6121067; fax: +90-266-6121097; e-mail: fdemiral@balikesir.edu.tr, gulsahcelik@balikesir.edu.tr).

an agate mortar and transferred in a porcelain crucible. The mixture was treated to microwave irradiation at 850 W for 20 minutes, then cooled to room temperature. The reground mixture was heated at 400°C for 2 h to obtain best crystallization. At the end of the treatments, the sample was allowed to cool down to room temperature in the furnace. In order to get clear of unreacted residues, the product was washed with hot water three times and dried at room temperature.

### B. Characterization Studies

The sample was characterized by X-ray powder diffraction (XRD) pattern using Panalytical X'Pert Pro Diffractometer and CuK $\alpha$  radiation ( $\lambda=1.54056\text{\AA}$ , 40 mA, 50kV). Infrared spectra were recorded between 4000 and 600  $\text{cm}^{-1}$  using

Perkin Elmer Spectrum 100 FTIR Spectrometer. Morphological property of the sample was realized by ZEISS Supra 40 VP. Thermo gravimetric-differential thermal analysis (TG/DTA) was carried out in a Perkin Elmer Diamond TG/DTA. Siemens V12 domestic microwave oven and Protherm furnace were used.

### III. RESULTS AND DISCUSSION

The XRD pattern of the product was given in Fig. 1. When we compare the diffraction pattern to card database, we observed the compound was formed. The compound is crystallized in orthorhombic system with unit cell parameters  $a = 9.45$ ,  $b = 8.21$  and  $c = 5.14 \text{\AA}$ .

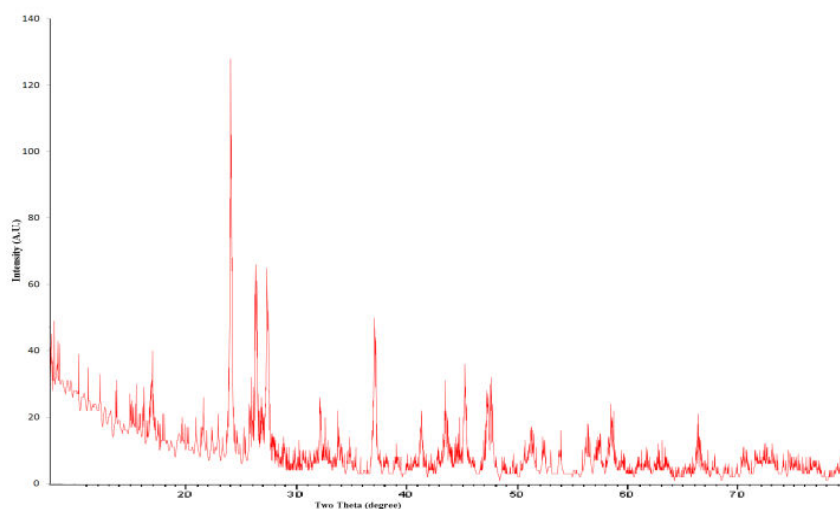


Fig. 1 The XRD pattern of CsBi(MoO<sub>4</sub>)<sub>2</sub>

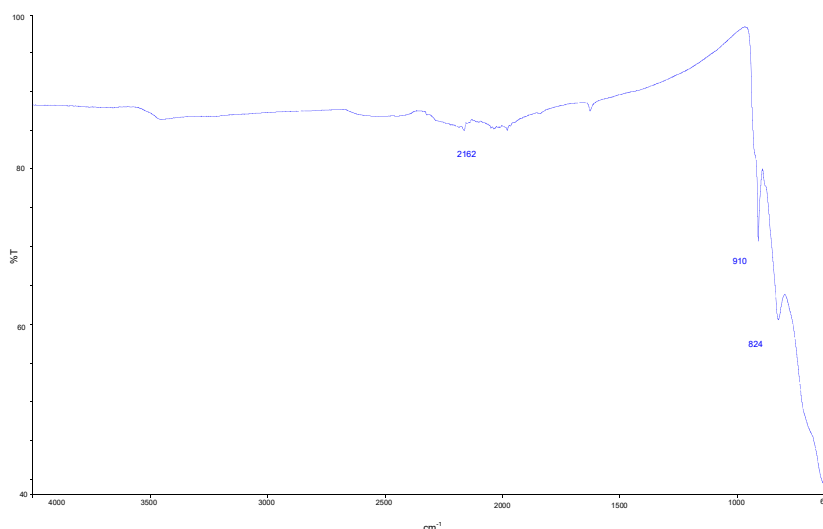


Fig. 2 The FTIR spectrum of CsBi(MoO<sub>4</sub>)<sub>2</sub>

The FTIR spectrum of the sample was shown in Fig. 2. The wave numbers at 910, 824 and 635  $\text{cm}^{-1}$  correspond to the vibrations of Mo–O, M–O, and O–Mo–O bond vibrations [75]–[79].

The morphology obtained from Scanning electron microscopy analysis was given in Fig. 3.  $\text{CsBi}(\text{MoO}_4)_2$  display plate-like structure as seen in the micrograph.

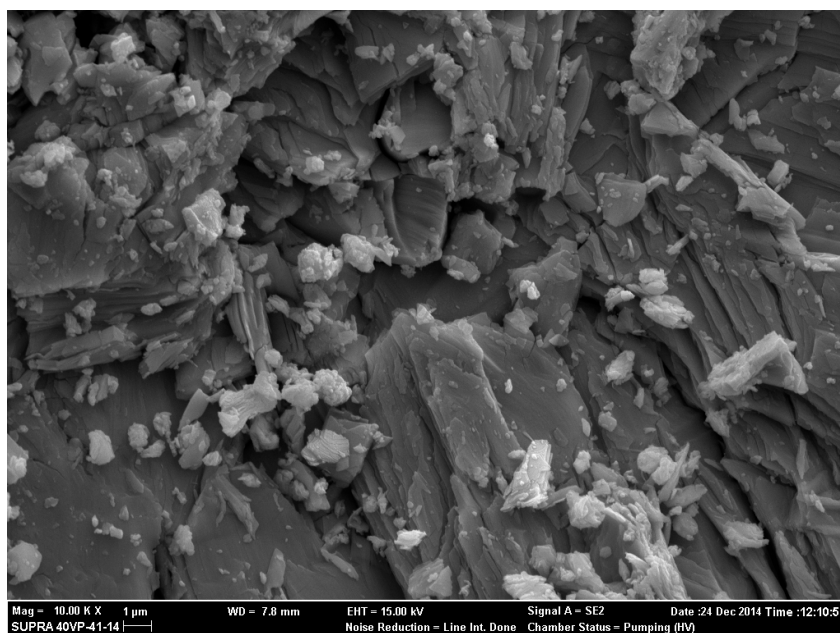


Fig. 3 The morphology of  $\text{CsBi}(\text{MoO}_4)_2$

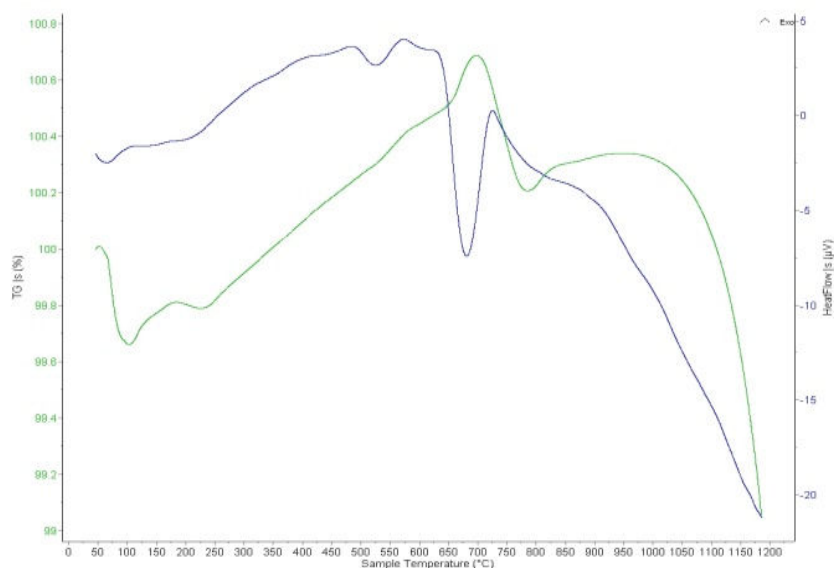


Fig. 4 The result of TG/DTA of  $\text{CsBi}(\text{MoO}_4)_2$

Thermal decomposition process was exhibited in Fig. 4. The sharp peak at around 700  $^{\circ}\text{C}$  can be attributed to melting of the sample.

#### IV. CONCLUSION

In conclusion,  $\text{CsBi}(\text{MoO}_4)_2$  is one of the dimolybdates applying to nearly all industry because of their huge variety of

crystallinity.  $\text{CsBi}(\text{MoO}_4)_2$  was synthesized by the newest synthesis process: microwave method which is low-cost, practical and respectful for the nature.  $\text{CsBi}(\text{MoO}_4)_2$  crystallize in orthorhombic system with unit cell parameters  $a = 9.45$ ,  $b = 8.21$ ,  $c = 5.14$   $\text{\AA}$  and space group  $D_{2h}^3$ . The characterization studies were achieved by XRD, FTIR, SEM and TG/DTA techniques.

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graduated from University of Uludağ, Department of Chemistry in 2008. She has a master degree of science in chemistry from Balıkesir University. She has been PhD student at Balıkesir University since 2010. Her major field is inorganic chemistry.

She works as an Project Expert at Balıkesir University since 2013. She was published eleven articles in SCI-expanded journals related with inorganic chemistry and bioinorganic chemistry. Her current and previous research interests are to material science, bioinorganic chemistry, solid state chemistry, crystallography and powder X-ray diffraction.

MSc. Çelik Gül is a member of Turkish Chemistry Society and Turkish Biochemical Society.

**Figen Kurtuluş** was born in Balıkesir, Turkey in 1972. She graduated from University of Uludağ Department of Chemistry Education in 1993. She has a master degree of science and PhD in chemistry from Balıkesir University. Her major field is inorganic chemistry.

She has been Assoc. Prof. Dr at Balıkesir University Chemistry Department since 2011. She was published twenty articles in SCI-expanded journals related with inorganic chemistry and solid state chemistry. Her current and previous research interests are to inorganic chemistry, material science, solid state chemistry, crystallography and powder X-ray diffraction.

Assoc. Prof. Dr. Kurtuluş is a member of Turkish Chemistry Society.

**Gülşah Çelik Gül** was born in Balıkesir, Turkey on 9<sup>th</sup> of July in 1986. She