

Characterization of Dehydrated Magnesium Borate Minerals Synthesized by Solid-State Method at 800°C from $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, MgO and B_2O_3

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Abstract

Magnesium borates are one of the subgroups of boron minerals, where boron minerals are that one of the important underground resources in Turkey. Minerals of boron application areas are expanding day by day because the very advantageous features. In this study, both magnesium borate and boracite syntheses are aimed using the solid-state method at 800 °C in 4 hours. $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, MgO and B_2O_3 are used as raw materials. Different mole fractions of $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}:\text{MgO}$ to B_2O_3 is investigated and the effect of concentration on the synthesis is studied. The characterizations of synthesized products are analyzed by X-Ray Diffraction (XRD) and Fourier Transform Infrared Spectrometer (FT-IR). From the results of the XRD analysis, it is seen that two types of dehydrated magnesium borate (MgB_4O_7 and $\text{Mg}_2\text{B}_2\text{O}_5$) and boracite ($\text{Mg}_6\text{B}_{14}\text{O}_{26}\text{Cl}_2$) formations are occurred.

Key words: Dehydrated magnesium borates, boracite, solid-state method, XRD, FT-IR

1. Introduction

Boron, which occurs with combination of different elements, has a strategic importance in World due to having high technology features. Turkey is known that approximately 72% of the world's boron reserves. Balıkesir/Bigadic, Kütahya/Sultancayırı and Eskişehir/Kırka are important boron deposits in Turkey [1,2].

Magnesium borates, which are sub-group of boron minerals, have a very wide field of industry because of the properties such as high corrosion resistance, high heat resistance. Magnesium borates can be obtained by synthetic production or natural sources. Magnesium borates natural existence is generally seen in salt lakes. There aren't a lot of magnesium borates in boron deposits in Turkey. For this reason, the synthesis of magnesium borate is an issue to be studied [2-4].

In the literature, several studies have been conducted about synthesis of magnesium borates on thermal method.

Obut A. (2007) had been studied the synthesis of different magnesium borate components from mixture of $\text{MgO}-\text{B}_2\text{O}_3$ and $\text{MgO}-\text{B}(\text{OH})_3$. This study was made in different mole ratios and the

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reactions took place between 500 °C and 900 °C [5]

Ay E. (2006), different mole ratios of $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}:\text{B}_2\text{O}_3$ were reacted with thermal method. Results of the study, 500 °C and 600 °C has been inadequate and 900 °C had given the better results [1].

In this study, magnesium borates are synthesized by thermal (solid-state) method. Magnesium oxide (MgO), magnesium chloride hexahydrate ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) and boron oxide (B_2O_3) are used as raw materials. Raw materials are mixed with different ratios and the reactions took place in 800 °C with air atmosphere.

2. Materials and Methods

2.1. Preparation of the Raw Materials

Magnesium oxide and magnesium chloride hexahydrate was provided from Merck Chemicals, boron oxide was retrieved from Boron Management Plant in Bandirma. Before the experimental studies, boron oxide were crushed, milled and sieved to -75 μm . $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ and MgO was used as supplied.

2.2. Thermal Synthesis

Raw materials reacted with thermal (solid-state) method. With this method, raw materials mixed different mole rates and pressed with Manfredi OL57 at 100 bar and 1 minute. Then, pellets are reacted with a heating rate of 10 °C/min to a sintering temperature of 800 °C and remained constant at this temperature for 240 minutes.

2.2. Characterization Analysis

Characterization analysis of synthesized products and raw materials are determined using the techniques of XRD (X-Ray Diffractometer) and FT-IR (Fourier Transform Infrared Spectroscopy).

3. Results

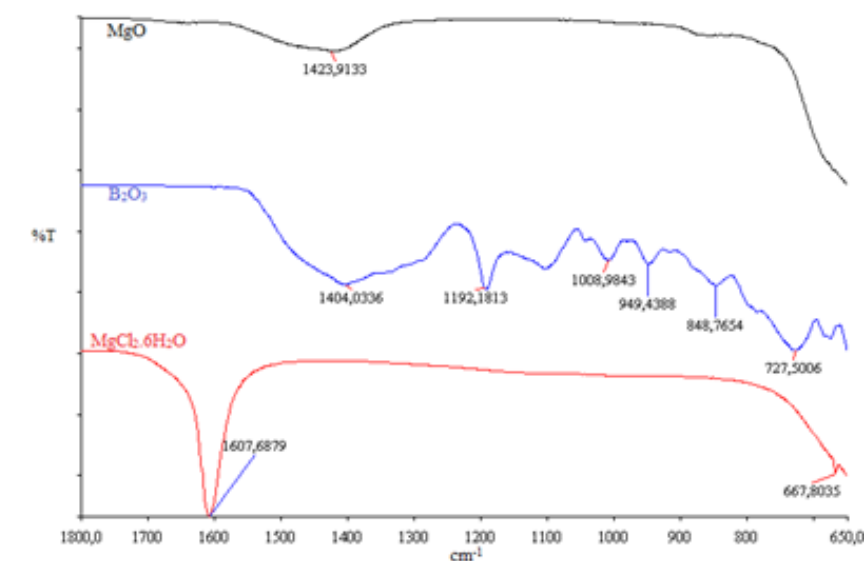
3.1. Raw Materials Characterization Results

XRD results of raw materials are given Table 1. According to results, score of boron oxide, magnesium oxide and magnesium hexahydrate are determined 42, 78 and 16 respectively.

Table 1. XRD results of raw materials

Pdf #	Mineral name	Mineral formula	Score
00-006-0297	Boron oxide	B ₂ O ₃	42
01-087-0651	Periclase	MgO	78
01-077-1268	Bischofite	MgCl ₂ .6H ₂ O	16

FT-IR results of raw materials are given Figure 1. According to the figure, characteristic peaks of the raw materials are found.

**Figure 1.** FT-IR results of raw materials

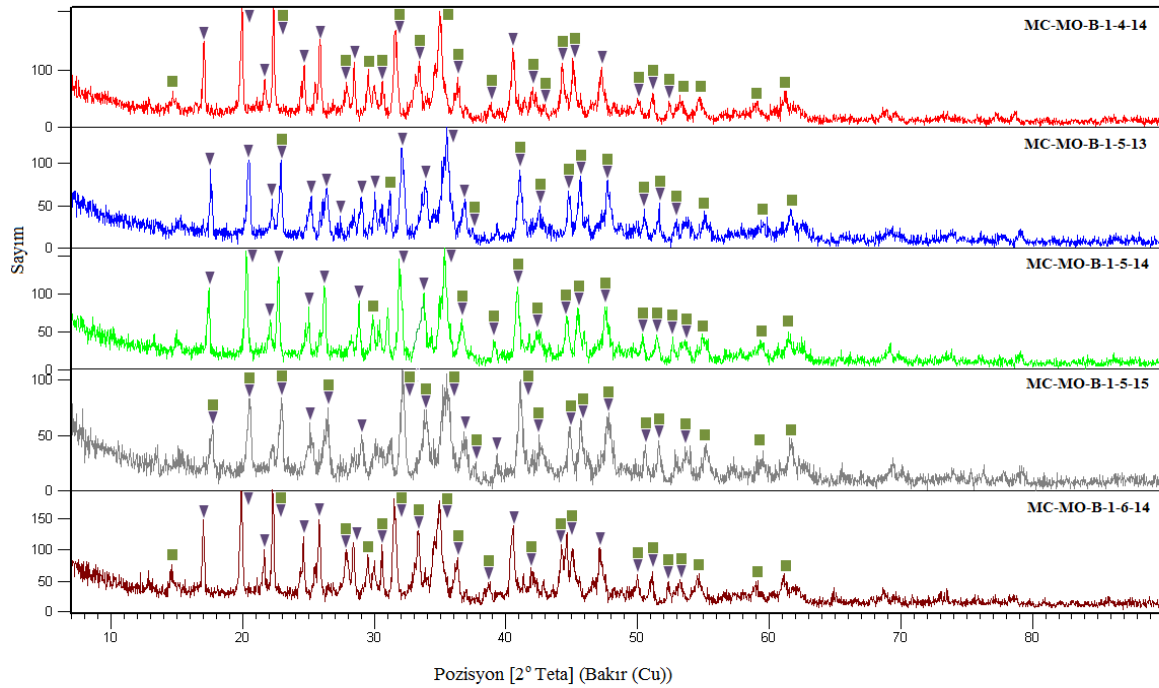
3.2. Product Characterization Results

Characterization of synthesized products are made by XRD and FT-IR. According to XRD results, boracite, magnesium borate are acquired by products. Magnesium borate, which is a major component, is reached the highest score (66) at 1:4:14 and 1:6:14. The lowest score is obtained (22) at 1:5:15. Boracite is obtained as the minor component. XRD results and patterns of synthesized products are given Table 2 and Figure 2 respectively.

Table 2. XRD results of synthesized products

MR*	Products			
	pdf no	Name	Formula	Score
1:4:14	01-071-750	Boracite	$\text{Mg}_3\text{B}_7\text{O}_{13}\text{Cl}$	31
	00-031-0787	Magnesium Borate	MgB_4O_7	66
1:5:13	01-071-750	Boracite	$\text{Mg}_3\text{B}_7\text{O}_{13}\text{Cl}$	6
	00-031-0787	Magnesium Borate	MgB_4O_7	25
1:5:14	01-071-750	Boracite	$\text{Mg}_3\text{B}_7\text{O}_{13}\text{Cl}$	17
	00-031-0787	Magnesium Borate	MgB_4O_7	65
1:5:15	01-071-750	Boracite	$\text{Mg}_3\text{B}_7\text{O}_{13}\text{Cl}$	3
	00-031-0787	Magnesium Borate	MgB_4O_7	22
1:6:14	01-071-750	Boracite	$\text{Mg}_3\text{B}_7\text{O}_{13}\text{Cl}$	31
	00-031-0787	Magnesium Borate	MgB_4O_7	66

* MR: mole ratios (Magnesium chloride hexahydrate – magnesium oxide – boron oxide)

**Figure 2.** XRD results of synthesized products

Boracite: ■ Magnesium Borate: ▼

FT-IR results of synthesized products is given in Figure 2. According to results, FT-IR peaks of all products are similar. Therefore, first peaks, which are between $1370,22 - 1397,08 \text{ cm}^{-1}$, is the asymmetric contraction of tri-coordinate boron ($\text{B}_{(3)}\text{-O}$). Second and third peaks are stretching of (B-O-H). Fourth peaks are symmetric contraction of the three coordinate. Fifth and sixth peaks are symmetric contraction of the four coordinate. Bending of $\text{B}_{(3)}\text{-O}$ is seventh and eighth peaks.

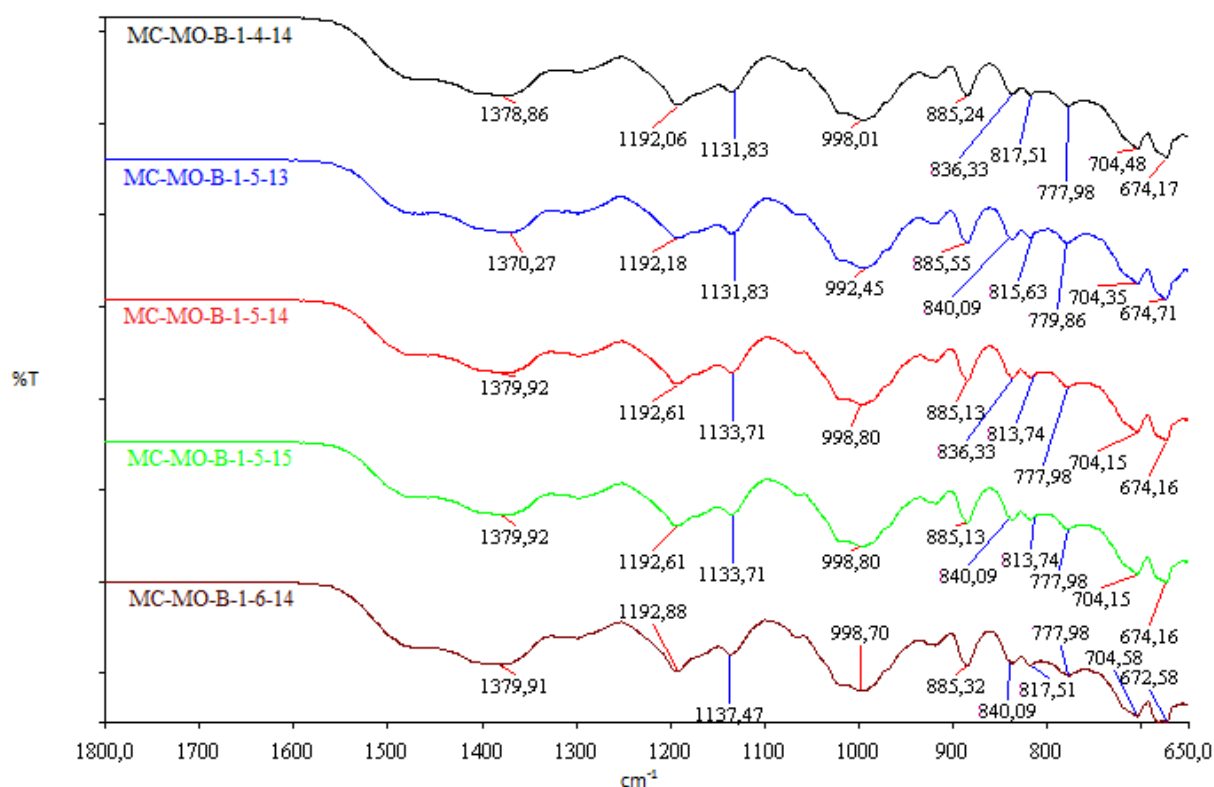


Figure 3.2 FT-IR results of synthesized products

4. Discussion

Magnesium borates is often used instead of other refined borates and metal borates, as a source of magnesium and boron. Production of superconducting magnesium diboride, in contact lens washing solutions, separation pesticides from soil are area of use of magnesium borates. A large number of different composition of magnesium borates are might derived naturally or synthesized by various methods [6-7].

In this study, magnesium borates are obtained by thermal method at $800 \text{ }^{\circ}\text{C}$. Magnesium oxide (MgO), magnesium chloride hexahydrate ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) and boron oxide (B_2O_3) are used as raw materials. According to results of XRD and FT-IR, it can be seen that different score of magnesium borate and boracite are obtained from different mole ratios.

Conclusions

Synthesis of magnesium borate, which is not found in large amounts in our country, are studied by thermal method. Effect of concentration are examined at magnesium borate production. Magnesium borate and boracite, that is minerals of magnesium borate, are obtained by thermal reactions. In addition, different studies are carried out for synthesis of magnesium borates.

Different temperatures and different times will study for the best results. This study will be a model for the future.

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