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Magnesium Borate Synthesis by Microwave Method Using $MgCl_2.6H_2O$ and H_3BO_3

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Abstract**—**There are many kinds of metal borates found not only in nature but also synthesized in the laboratory such as magnesium borates. Due to its excellent properties, as remarkable ceramic materials, they have also application areas in anti-wear and friction reducing additives as well as electro-conductive treating agents. The synthesis of magnesium borate powders can be fulfilled simply with two different methods, hydrothermal and thermal synthesis. Microwave assisted method, also another way of producing magnesium borate, can be classified into thermal synthesis because of using the principles of solid state synthesis. It also contributes producing particles with small size and high purity in nano-size material synthesize. In this study the production of magnesium borates, are aimed using $MgCl_2.6H_2O$ and H_3BO_3. The identification of both starting materials and products were made by the equipments of, X-Ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FT-IR). After several synthesis steps magnesium borates were synthesized and characterized by XRD and FT-IR, as well.

Keywords**—**FT-IR, magnesium borates, microwave method, XRD.

I. INTRODUCTION

BORON minerals are used in glass, cement and ceramic industry, irrigation, boron fibers, metallurgical industry, nuclear applications, aerospace and aviation due to boron's different characteristics [1]. On the other hand among the borates, magnesium borate is used in very wide range such as anti-wear additive [2], catalyst for the conversion of the hydrocarbons [3], production of superconducting materials, composition of detergents [4], preservative, and antiseptics [5], because it has the properties of corrosion resistance, high heat resistance, light weight [6]. Because magnesium borate has such different usage areas, in the recent years it became attractive for the researchers to synthesize it at different conditions with different raw materials [7].

The synthesis of magnesium borate powders can take place in two different methods, hydrothermal and thermal synthesis [8]. Hydrothermal method includes the way of heating reactants in aqueous solutions at significant temperatures. The aqueous solution, commonly water, enables to function both as pressure transmitting medium and as a solvent [9]. On the other hand, thermal synthesis, also called solid state synthesis, is a method in which reaction occurs in high temperature furnace. Due to drawing correspondence with solid state synthesis, another way of producing magnesium borate, microwave assisted method, can take place in thermal synthesis.

In recent years, microwave processing has been studied deeply for synthesizing magnesium borate powders, because of the disadvantages of conventional methods, such as solid-state reactions at high temperatures [10], [11]. Apart from the possibility of decomposing of the product over time in the reaction vessel, the solid state method is quiet slow. Microwave assisted synthesis; in contrast, achieves the rapid rise in temperature and uniform heating. This occur thorough microwaves which interact with materials causing the energy transfer to the reactants [12], [13]. As mentioned, the microwave synthesis method offers several advantages as useful energy sources such as short period, faster reaction times, higher reaction yield and less energy consumption [14], [15].

In literature, [16] and [17] used magnesium and boron sources with different mole ratios in various times to produce magnesium borate powders through microwave assisted method. In both studies with different reaction parameters, samples were obtained in amorphous phase. In [18], magnesium borates were obtained by microwave synthesis method using the raw materials, magnesium oxide (MgO) and boric acid (H_3BO_3). The reaction parameters were selected as 270W microwave power, 1:4 ($MgO:H_3BO_3$) molar ratio and 6, 8, 10, 12 minutes reaction times. From all characterization results, the synthesized magnesium borate at 8 minutes had the best results.

In this study; microwave assisted magnesium borate synthesis was achieved in both media with and without water. To be able to make this synthesis without water, calcination was used. In the literature, [19] calcined $MgCl_2.6H_2O$ in HCl atmosphere until 203°C and $MgCl_2.H_2O$ was obtained. Reference [20] used magnesium chloride hexahydrate and

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ammonium chloride to be able to obtain $MgCl_2.H_2O$ and calcined at 700°C (973K) for 1 hour.

Taking all these literature studies into consideration, it was aimed to produce magnesium borate with microwave assisted synthesis. For this reason, magnesium chloride $MgCl_2.6(H_2O)$ and boric acid (H_3BO_3) were used as raw materials with appropriate microwave power and various reaction times. After synthesis, X-ray diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FT-IR) were applied to identify the obtained products with selected mole ratio.

II. MATERIALS AND METHODS

A. Raw Material Preparation and Characterization

Boric acid (H_3BO_3) was supplied from the Eti Bank Bandirma Boron and Acid Plant in Balikesir, Turkey. Then it was crushed, grinded with agate mortar (Fig. 1 (a)) and sieved (Fig. 1 (b)) to a particle size below 75 microns.

The other raw material, magnesium chloride hexahydrate ($MgCl_2.6H_2O$), was provided from Sigma-Aldrich with 99.9% purity.

Fig. 1 (a) Grinding process, (b) Sieving process

Boric acid powders were subjected to X-Ray Diffraction (XRD) analysis; Philips PANalytical brand (Fig. 2) with Cu-Kα radiation at the parameters of 45kV and 40mA was used.

Fig. 2 Philips PANalytical XRD

Starting materials were also subjected to “Vibration Spectroscopy” in order to characterize the raw materials. Infrared spectra were recorded in the range 650-1800cm^{-1}, with Perkin Elmer Spectrum One (Fig. 3) Fourier Transform Infrared Spectroscopy (FT-IR). During the analysis, universal ATR sampling accessory – Diamond/Zn was applied and scan number was determined as 4 and resolution set as 4cm^{-1}.

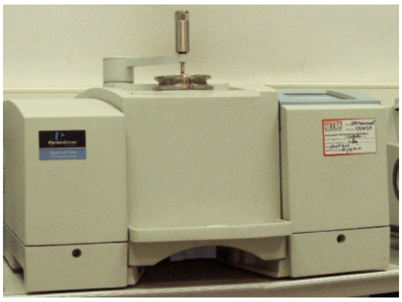

Fig. 3 Perkin Elmer Spectrum One FT-IR

B. Microwave Synthesis Using $MgCl_2.6H_2O$ and H_3BO_3

So as to increase the reaction efficiency, the powder was pelletized with Manfredi OL 57 Pelletizing Machine showed in Fig. 4. After mixing the compounds homogenously in the ratio of 1:2 ($MgCl_2.6H_2O$:H_3BO_3), the samples are pressed at a pressure of 100bar in a couple of minutes.

Subsequently, the pellets were put into the microwave oven with the selected 270W microwave power for 2, 4, and 6 minutes, respectively.

Fig. 4 Manfredi OL 57 pelletizer

C. Calcination of $MgCl_2.6H_2O$

$MgCl_2.6H_2O$ was calcined at 250°C for 4 hours with Protherm PLF 120/7 furnace which is shown in Fig. 5.

Fig. 5 Protherm furnace

D. Microwave Synthesis Using $MgCl_2$ and H_3BO_3

In order to see the effect of the calcined magnesium chloride on the reaction yield, microwave assisted method was applied using $MgCl_2$ and H_3BO_3 in the ratio of 1:2 ($MgCl_2$:H_3BO_3). The steps for the pelletization were repeated and reaction parameters were set as 270W microwave power and 2, 4, 6 minutes, as well.

E. Microwave Synthesis Using $MgCl_2$, H_3BO_3 and H_2O

H_3BO_3 and $MgCl_2$ were taken with the Mg:B mole ratio of 1:4. 1mL of deionized water was added to H_3BO_3 and then $MgCl_2$ was added. This sample was put inside the microwave oven that's power was adjusted to 270Watt for 2, 4, 6, 8, 10 and 12 minutes, respectively.

After that, samples were taken from the microwave oven; these were washed with ethanol and then dried in the Ecocell oven at 40°C.

III. RESULTS AND DISCUSSION

A. Raw Material Characterization Results

XRD results and pattern of magnesium chloride hexahydrate and boric acid were shown in Fig. 6 and Table I, respectively.

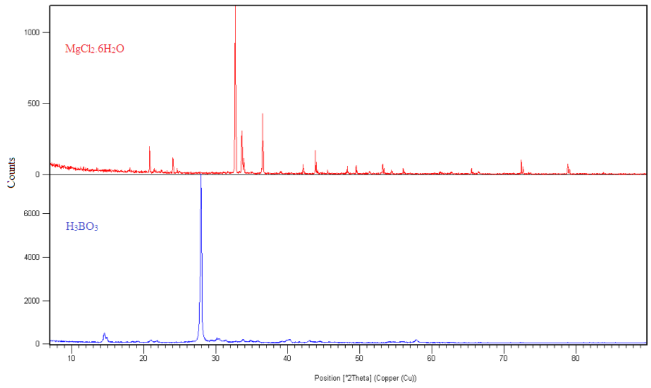

Fig. 6 XRD pattern of raw materials

TABLE I
XRD RESULTS OF RAW MATERIALS

Reference Code	Raw Material	Mineral Name	Mineral Formula
01-077-1268	Magnesium chloride hexahydrate	Bischofite, syn	$MgCl_2.6H_2O$
01-073-2158	Boric acid	Sassolite	H_3BO_3

According to the XRD results of the raw materials magnesium chloride hexahydrate ($MgCl_2.6H_2O$) was found with powder diffraction file (pdf) number of "01-077-1268". The "01-073-2158" pdf numbered boric acid represented the standard diffraction pattern of "Sassolite", as well.

FT-IR spectrums of the boric acid and magnesium chloride hexahydrate were shown in Fig. 7. From the FT-IR analysis, the spectrums were in good agreement with the scanned FT-IR ATR inorganic library and the results were given in Table II.

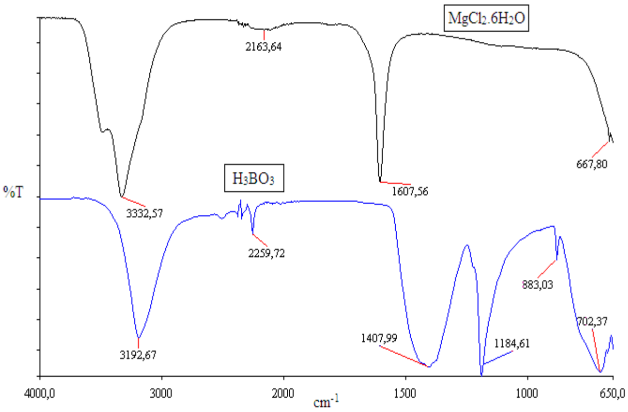

Fig. 7 FT-IR spectrums of the raw materials

TABLE II
FT-IT ATR INORGANIC LIBRARY RESULTS

Score	Library Code	Mineral Name	Mineral Formula
0.704	AI0031	Boric acid	H_3BO_3
0.864	AI0084	Magnesium chloride hexahydrate	$MgCl_2.6H_2O$

B. $MgCl_2.6H_2O$ and H_3BO_3 Microwave Synthesis Results

XRD patterns and results of the first synthesis were shown in Fig. 8.

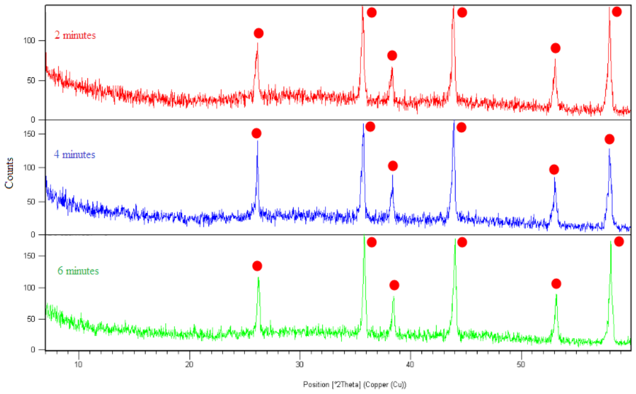

Fig. 8 XRD patterns of the first synthesized magnesium borates; ● Corundum, syn

According to the XRD patterns, no magnesium borate powders were detected. The peaks represented contaminated Corundum powders (Al_2O_3), which came from the alumina coated ceramic crucible, were used during the synthesis. Due to the adverse effect of coated ceramic crucible on the product, the next experiments were conducted with the watch glasses.

C. $MgCl_2.6H_2O$ Calcination Results

Four different forms can be obviously seen by examining the XRD results. As expected after the calcination process, magnesium chloride was appeared in two varied form with the powder diffraction file number of "01-074-0521" and "01-072-1517". Apart from dehydrated magnesium chloride, cyrstal structured hydrated forms were observed, as well. Yet, correspondingly to the literature, it can be also seen that the total conversion of $MgCl_2.6H_2O$ to $MgCl_2$ was not occured

[21], [22]. Detailed identification results are shown in Fig. 9 and Table III, respectively.

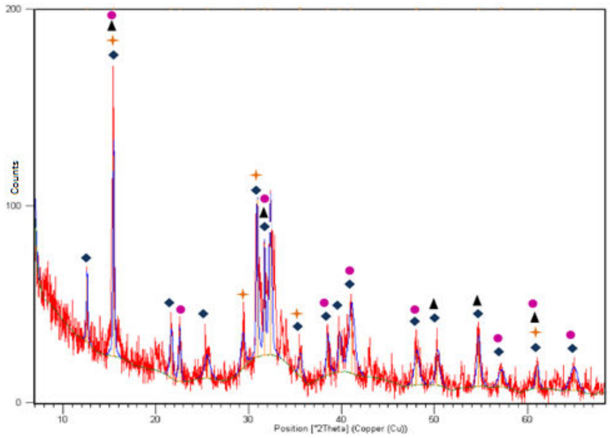

Fig. 9 XRD patterns of the calcined magnesium chloride

TABLE III
XRD RESULTS OF CALCINED MAGNESIUM CHLORIDE

Reference Code	Mineral Name	Formula	Symbol
01-074-0521	Magnesium Chloride	$MgCl_2$	✦
01-072-1517	Magnesium Chloride	$MgCl_2$	▲
00-053-0260	Magnesium Chloride Hydrate	$MgCl_2.H_2O$	◆
00-011-0328	Magnesium Chloride Hydroxide	$Mg(OH)Cl$	●

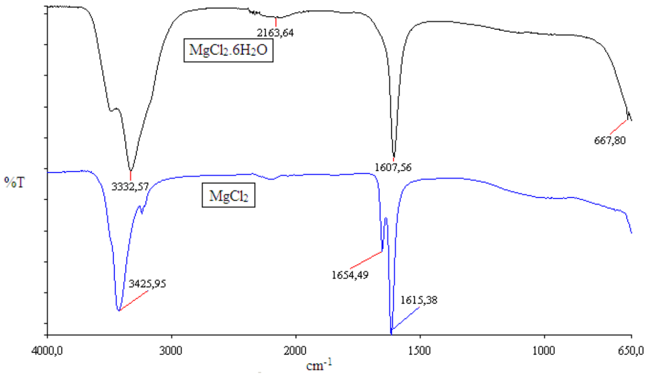

Fig. 10 FT-IR spectrums of magnesium chloride hexahydrate and calcined magnesium chloride

According to the FT-IR spectrum results in Fig. 10, it can be obviously seen that the peaks of $[OH]^-$ were formed and were not completely disappeared. With this analysis, FT-IR spectrums corroborate the results of XRD patterns.

D. $MgCl_2$ and H_3BO_3 Microwave Synthesis Results

According to the XRD results shown in Fig. 11 and Table IV, respectively, it was observed that the synthesized products were formed in amorphous-based. Although the pattern consisted of a number of peaks which belong to Mcallisterite minerals with pdf number of “01-070-1902”, exact structure of magnesium borate powders were not formed.

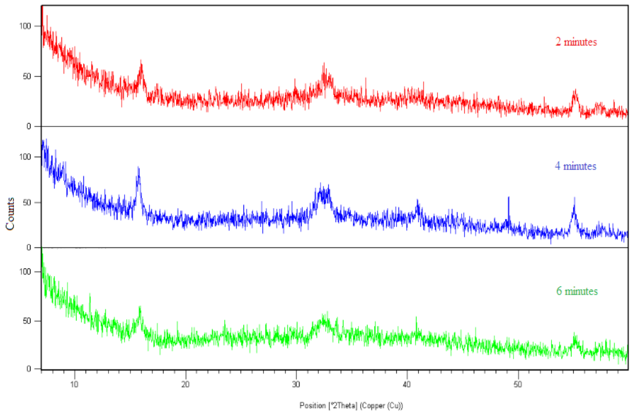

Fig. 11 XRD patterns of the second synthesized magnesium borates

TABLE IV
XRD RESULTS OF THE SECOND SYNTHESIZED PRODUCTS

Time (min)	Reference Code	Mineral Name	Mineral Formula
2	01-070-1902	Mcallisterite	$Mg_2(B_6O_7(OH_6)_2.9H_2O$
4	01-070-1902	Mcallisterite	$Mg_2(B_6O_7(OH_6)_2.9H_2O$
6	01-070-1902	Mcallisterite	$Mg_2(B_6O_7(OH_6)_2.9H_2O$

FT-IR spectrums and peak list of the vibration spectroscopy results are shown in Fig. 12 and Table V, respectively.

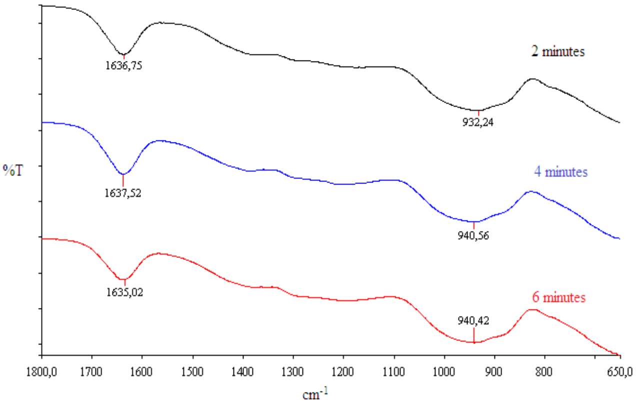

Fig. 12 FT-IR spectrums of the second synthesized magnesium borates

TABLE V
PEAK LIST OF FT-IR RESULTS

Peak	2 min	4 min	6 min	Peak Interpretation
Peak 1	1636.75	1637.52	1635.02	δ (H-O-H)
Peak 2	932.24	940.56	940.42	$\upsilon_s(B_{(3)}-O)$

FT-IR spectrums of the synthesized samples were assigned referring to the literature [23], [24]. Peak 1, between band values 1637.52 and 1635.02cm^{-1} can be assumed as H-O-H bending mode. The wide band range between 940.56 and 932.24cm^{-1} is assigned as symmetric stretching of $B_{(3)}$-O.

E. $MgCl_2$, H_3BO_3 and H_2O Microwave Synthesis Results

XRD patterns of the third synthesized products and accurate results are given in Fig. 13 and Table VI, respectively.

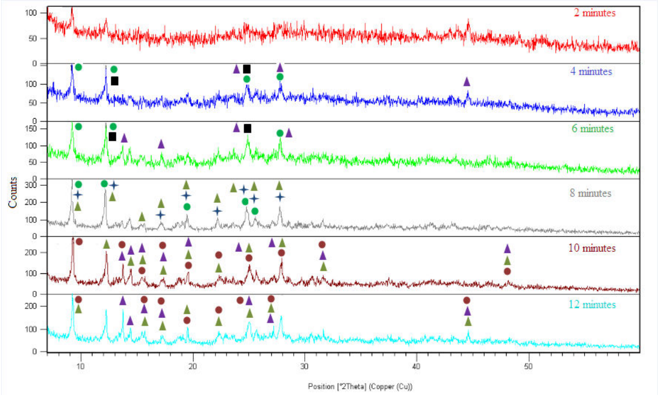

Fig. 13 XRD patterns of the third synthesized magnesium borates

TABLE VI
XRD RESULTS OF THE THIRD SYNTHESIZED MAGNESIUM BORATES

Time (min)	Reference Code	Mineral Name	Mineral Formula	Symbol
2	-	-	-	-
4	00-019-0754	MgBH	$Mg_7B_4O_{13}.7H2O$	■
	01-071-2409	Aksaite	$MgB_6O_7(OH)_6).2H_2O$	▲
	00-050-1637	Nepskoeite	$Mg_4Cl(OH)_7.6H_2O$	●
6	00-019-0754	MgBH	$Mg_7B_4O_{13}.7H2O$	■
	01-071-2409	Aksaite	$MgB_6O_7(OH)_6).2H_2O$	▲
	00-050-1637	Nepskoeite	$Mg_4Cl(OH)_7.6H_2O$	●
8	01-076-0539	MgBH	$MgO(B_2O_3)_3.6H_2O$	✦
	01-076-0540	Admontite	$MgO(B_2O_3)_3.7H_2O$	▲
	00-050-1637	Nepskoeite	$Mg_4Cl(OH)_7.6H_2O$	●
10	01-071-2409	Aksaite	$MgB_6O_7(OH)_6).2H_2O$	▲
	01-076-0540	Admontite	$MgO(B_2O_3)_3.7H_2O$	▲
	01-071-1681	Hungchaoite	$MgB_4O_5(OH)_4.7H_2O$	●
12	01-071-2409	Aksaite	$MgB_6O_7(OH)_6).2H_2O$	▲
	01-076-0540	Admontite	$MgO(B_2O_3)_3.7H_2O$	▲
	01-071-1681	Hungchaoite	$MgB_4O_5(OH)_4.7H_2O$	●

Magnesium borate hydrate: MgBH

As it can be seen from the patterns and results, no crystal structure has been formed at 2 minutes. Up to the reaction time of 8 minutes, Nepskoite mineral was monitored due to the existence of H_2O, which was added before the reaction. Formation of the minerals began at 8 minutes and continued significantly up to 12 minutes of reaction time. Comparing the intension of the observed peaks, it can be monitored that, the best formation occurred at 10 minutes.

FT-IR spectrums and peak list of the third synthesized magnesium borates are given in Table VII and Fig. 14, respectively.

TABLE VII
PEAK LIST OF FT-IR RESULTS

No	2 min	4 min	6 min	8 min	10 min	12 min	Peak Interpretation
1	809	808	807	808	806	804	$\upsilon_s(B_{(4)}–O)$
2	868	867	858	868	848	844	$\upsilon_s(B_{(3)}–O)$
3	-	-	-	-	913	913	$\upsilon_s(B_{(3)}–O)$
4	-	956	960	961	961	959	$\upsilon_s(B_{(3)}–O)$
5	995	1006	1033	1033	1084	1080	$\upsilon_{as}(B_{(4)}–O)$
6	-	-	-	-	1234	1233	δ (B-O-H)
7	1338	1334	1340	1340	1354	1353	$\upsilon_{as}(B_{(3)}–O)$
8	1651	1654	1651	1651	1650	1648	δ (H-O-H)

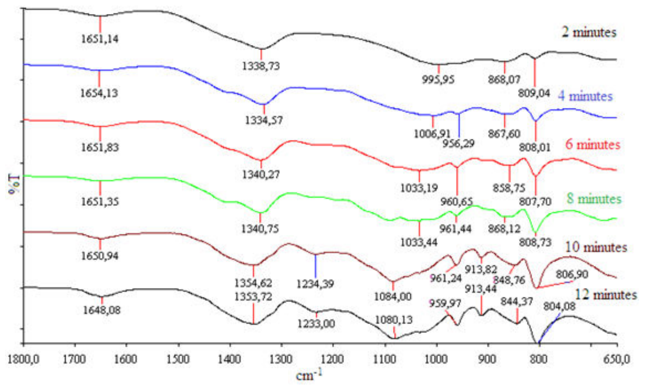

Fig. 14 FT-IR spectrums of the third synthesized magnesium borates

According to the Fig. 14, the band between 809 and 804 cm^{-1} is the symmetric streching of $B_{(4)}$-O. Peak 2, 3, and 4 are assigned as the symmetric streching of $B_{(3)}$-O. The wide band range between 1084 and 995cm^{-1} might be the asymmetric streching of $B_{(4)}$-O. The band between 1234 and 1233cm^{-1}, corresponding to peak 6, is the in-plane bending of B-O-H. Peak 7 band value varies betwenn 1353 and 1334$cm^{-1}$and this band range indicates the asymmetric streching of $B_{(3)}$-O. Last but not least, peak 8 is assigned to thw H-O-H bending mode, which is a indicative that the compound consists of crystal water.

From the FT-IR analysis results of the synthesized magnesium borates in this experiment compromise with the ones in literature and shows the characteristic magnesium borate hydrate peaks [24], [25], [26].

IV. CONCLUSIONS

With the aim of synthesizing magnesium borate powders, microwave assisted method was applied in this study. For this purpose, the experiments were conducted step by step, basically changing the raw material, mole ratio of the starting materials and reaction time. Microwave power was always set to 270W, though.

The results of the experiments were analyzed and found that no formation occurred with the raw materials of $MgCl_2.6H_2O$ and H_3BO_3. After that, calcination process was carried out in order to analyze the effect of itself on the yield of magnesium borate production. Nevertheless, accurate results were also not monitored by using the calcined magnesium borate. In the third step, H_2O was added to the calcined magnesium borate and H_3BO_3 mixture. The given results of this experiment showed that the crystal structure was formed after 4 minutes of reaction time. The optimum experimental result was monitored at 10 minutes, though.

It could be seen that, microwave method is an effective and optional way to obtain magnesium borate powders. Needles to say, it will be enhanced with further experiments.

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