

Microwave Dehydration Behavior of Admontite Mineral at 360W

E. Moroydor Derun, F. T. Senberber, A. S. Kipcak, N. Tugrul, and S. Piskin

Abstract—Dehydration behavior gives a hint about thermal properties of materials. It is important for the usage areas and transportation of minerals. Magnesium borates can be used as additive materials in areas such as in the production of superconducting materials, in the composition of detergents, due to the content of boron in the friction-reducing additives in oils and insulating coating compositions due to their good mechanic and thermal properties.

In this study, thermal dehydration behavior of admontite ($\text{MgO}(\text{B}_2\text{O}_3)_3 \cdot 7(\text{H}_2\text{O})$), which is a kind of magnesium borate mineral, is experimented by microwave energy at 360W. Structure of admontite is suitable for the investigation of dehydration behavior by microwave because of its seven moles of crystal water. It is seen that admontite lost its 28.7% of weight at the end of the 120 minutes heating in microwave furnace.

Keywords—Admontite, dehydration, magnesium borate, microwave.

I. INTRODUCTION

BORON minerals are the common additives in industry because of their characteristic properties. According to statistics, Turkey has the largest portion with the 72% of boron reserves in the world [1]. Magnesium borate minerals, which are a sub-group of boron minerals, have great importance because of their thermal and mechanical features such as high coefficient of elasticity, high heat resistance and corrosion resistance. These types of borates, which can be used as magnesium or boron sources, have the using areas in ceramic, detergent industry and the potential applications such as anti-wear, anti-corrosion materials, thermo-luminescent phosphors and as catalysts useful for the conversion of hydrocarbons [1]–[4].

Admontite is a kind of magnesium hydrate borate mineral which has a white color, a monoclinic system and molecule formula of $\text{MgO}(\text{B}_2\text{O}_3)_3 \cdot 7(\text{H}_2\text{O})$. The boron oxide (B_2O_3) content of admontite mineral is 55.6% [5]–[8].

Dehydration process can be described with the loss of crystal water from the hydrated mineral. Thermal behavior features of materials should be known for the design parameters of equipment and for the economic factor decreasing the mass of a material in order to reduce

transportation costs [9], [10].

Removal of the crystal water of hydrated mineral is easy whereas the removal of water molecules in ionic structure is difficult. Direct heating experiments in thermogravimetric analysis (TGA) instruments have shown that the removal of crystal water is a fast operation, but the removal of the structurally bonded water due to hydroxyl ions $[\text{OH}^-]$ is slower and more difficult [9]–[12].

Microwave energy, which is a nonionizing electromagnetic radiation with frequencies in the range of 300 MHz to 300 GHz, have very large applications in industry such as communication, cooking food, tempering and thawing, and curing of wood and rubber products. The use of microwave energy advantages can be listed as; reduced processing costs, better production quality, new materials and products, improved human health, reduced hazards to humans and the environment and enhanced quality of life [12]–[15].

In previous studies, thermal dehydration behaviors of boron minerals were investigated by thermal analysis methods such as Thermal Gravimetric Analysis (TGA), Differential Thermal Gravimetric Analysis (DTG) and Differential Thermal Analysis (DTA) [9]–[11]. These studies showed that boron mineral lost their crystal water mainly in two steps. First step is dehydration, which is the removal of crystal water in structure. Dehydroxylation, which is the second step, is removal hydroxyl groups on a compound as water molecules.

The examples of microwave dehydration can be seen in literature. Ores from different regions show that minerals are responsive to microwave radiation, but optimal microwave exposures haven't any adverse effects on minerals [15], [16]. In the experiments of Saito et al., dehydration behavior of goethite mineral was studied by microwave treatment [17]. The effects of microwave power levels and sample mass on drying characteristics of moisture content, drying rate, moisture ratio were studied for the ilmenite mineral. Experiment results showed that the moisture content and drying rates were found to be dramatically affected by microwave power density [18].

In this study, it is aimed to investigate the thermal dehydration behaviors of hydrated magnesium borate minerals by the use of microwave energy.

II. MATERIAL AND METHODS

A. Materials

The admontite mineral was synthesized in our previous work with the reaction of magnesium oxide (MgO) and boric acid (H_3BO_3) at a reaction temperature of 100°C and reaction

E. Moroydor Derun, F. T. Senberber, N. Tugrul, and S. Piskin are with the Yildiz Technical University, Department of Chemical Engineering, Davutpasa Campus, 34210 Esenler, Istanbul, Turkey (e-mail: moroydor@yildiz.edu.tr, tsenberber@gmail.com, ntugrul@hotmail.com, piskin@yildiz.edu.tr).

A. S. Kipcak is with the Yildiz Technical University, Department of Chemical Engineering, Davutpasa Campus, 34210 Esenler, Istanbul, Turkey (phone: 0090-212-3834751; fax: 0090-212-3834725; e-mail: skipcak@yildiz.edu.tr / seyhunkipcak@gmail.com).

time of 240 minutes. After the reaction, slurry was dried 40°C [8].

To characterized the synthesized mineral was subjected to X-ray Diffraction (XRD) analysis with PANalytical X-ray Diffraction Instrument (Fig. 1) was used.



Fig. 1 PANalytical X-ray Diffractometer

B. Microwave Dehydration Method

A programmable domestic microwave oven (BOSCH-HMT 72G 420, Turkey) with maximum output of 800 W and 2450 MHz was used for the drying experiments. The dimensions (HxWxD) of the microwave oven were 194×290×300mm.

In experiments, power levels were determined as 270 and 360W for the dehydration. Dehydration times were determined between 2 and 120 minutes. For each experiment, three parallels were done.

In compare with the microwave dehydration process conducted, admontite mineral was studied with the TG/DTG analysis. Heating rate was 10°C and analyses were done at Perkin Elmer Diamond DTA-TG Instrument (Fig. 2) [8].

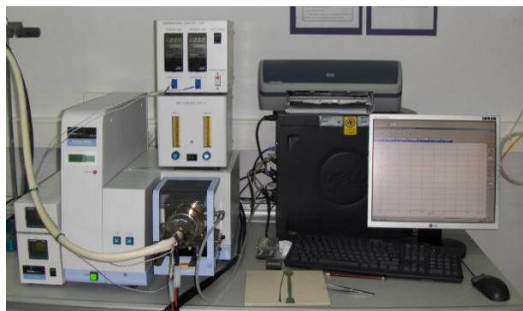


Fig. 2 Perkin Elmer Diamond DTA-TG

C. Data Analysis

After samples were dried at 40°C for 3 days, crystal water content and dehydration rates are experimented for the investigation of thermal behaviors.

The crystal water and drying rate of synthesized admontite mineral were calculated with using following equations:

$$m_c = \frac{m_t}{m} \times 100 \quad (1)$$

where m_c is the crystal water content of dried basis, %; m is the absolute amount of dried basis, g; m_t is the moisture amount at the time of t , g;

$$D = \frac{w}{(At)} \quad (2)$$

where D is the drying rate, g/s.cm²; W is the amount of releasing water from structure, g; A is the area of drying surface of admontite, cm²; t is the drying time, min.

III. RESULT AND DISCUSSION

A. Identification of the Synthesized Admontite

XRD pattern of synthesized mineral was given in Fig. 3. XRD results showed that synthesized mineral was admontite mineral with the chemical formula of “ $MgB_6O_{10} \cdot 7(H_2O)$ ” and powder diffraction file of “01-076-0540” [8].

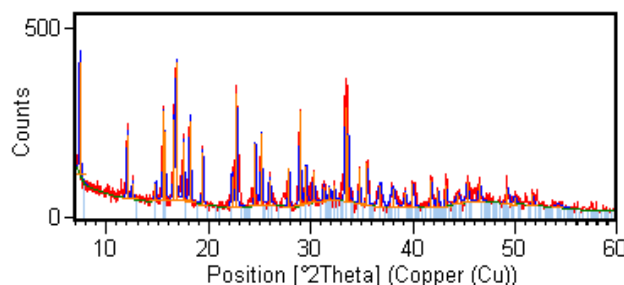


Fig. 3 XRD pattern of synthesized admontite mineral [8]

B. Results of Microwave Dehydration

The relationship between content of residual H₂O in hydrated structure and moles of releasing water from structure by changing dehydration times at 270 and 360W can be seen in Tables I and II, respectively.

In the 270W experiments (in Table I), calculations showed that approximately 2.81 moles of crystal water were released from structure of admontite and crystal water content decreased to 20.1 % at the end of dehydration time of 120 minutes.

TABLE I
RELEASING MOLES OF WATER FROM THE STRUCTURE OF ADMONTITE AT 270w

Dehydration Time (min)	Releasing Water (mole)	Content of Residual H ₂ O in Structure (%)
0	-	33.58
2	0.05	33.33
4	0.24	32.43
6	0.64	30.53
8	1.03	28.63
10	1.14	28.11
12	1.68	25.50
20	2.18	23.10
40	2.38	22.14
60	2.76	20.35
120	2.81	20.10

The dehydration process at 360W in Table II, with the increasing of power level, the residual water content of hydrate structure decreased to 4.89% and moles of dehydrated water were 5.98.

TABLE II
RELEASING MOLES OF WATER FROM THE STRUCTURE OF ADMONTITE AT 360W

Dehydration Time (min)	Releasing Water (mole)	Content of Residual H ₂ O in Structure (%)
0	-	33.58
2	0.11	33.04
4	1.01	28.73
6	1.93	24.31
8	2.30	22.56
10	2.38	22.17
12	2.76	20.33
20	3.33	17.62
40	4.36	12.69
60	4.77	10.71
120	5.98	4.89

Relationship between moisture content and drying time was given in Fig. 4. For the comparison, it could be said that power level of 360W was more suitable than 270W for the thermal behavior investigation of admontite minerals.

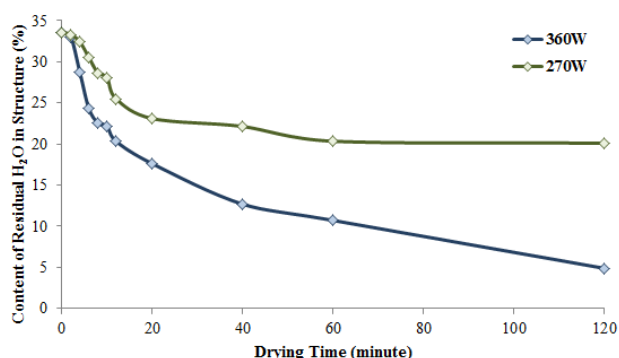


Fig. 4 Moisture contents of admontite by changing dehydration time

Rate of removal water from the crystal structure, which could be described as drying rate, changed with crystal water content in structure. Drying rates were calculated for the power level of 360W, because efficient decrease in moisture content wasn't obtained at 270W. In Fig. 5, x-axis and y-axis represent the residual water content of hydrate structure (%) and drying rate at 360W, respectively.

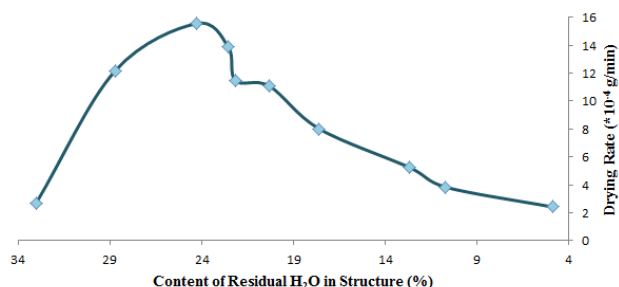


Fig. 5 Relationship between drying rate and moisture content of dried admontite at 360W

According to results of microwave dehydration process, moisture content decreased with increasing of drying time. With the releasing of capillary and mineral bond water in admontite, drying rate is higher in first minutes of dehydration. By the decreases of residual water content below 24.31%, drying rate approximately decreases in a linear way. The highest drying rate is seen in the residual water content of hydrate structure of 20.33%, and dehydration time of 12 minutes.

C. Comparison of the Results of Microwave Dehydration Process and Thermal Analysis

TG analysis result of admontite mineral was given in Fig. 6. According to thermogram, admontite mineral lost its hydrate at 2 steps, which were comprehensive with literature. First removal of water from hydrate structure occurred between the temperatures of 32.3 and 138.2°C. Second part of dehydration process was seen between the temperatures of 138.3 and 556.8°C. In thermal analysis, weight losses of admontite mineral were 11.7% and 24.6%, respectively.

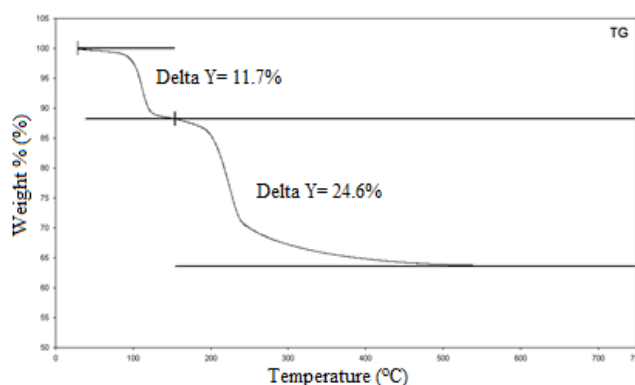


Fig. 6 Thermal dehydration of admontite by TG analysis

Percentages of the total mass loss were calculated as 28,69 % for microwave dehydration and 36,3% for thermal analysis. It can be concluded that there were some crystal water content in structure of admontite at the end of the microwave experiments. Higher power levels and different dehydration times could be experimented.

IV. CONCLUSIONS

Admontite mineral, which can be synthesized in pure form, could be a useful additive in industry due to being a type of boron mineral. Thus, its thermal behavior should be known for design parameters of using equipment in plants. In this study, thermal dehydration behavior of admontite mineral is experimented by microwave energy. Results show that moisture content of admontite mineral can be controlled by microwave energy.

Moisture content and drying rate values are determined with changing of dehydration time. In the comparison of power levels, 360W is more suitable for the dehydration process. The drying rate is at highest point, in moisture content of 20.33% and dehydration time of 12 minutes and admontite

lost 5.98 moles of water at 360W. However, determined operation conditions are inefficient for the loss of total moisture content.

In conclusion, boron minerals are suitable materials for the investigation of thermal dehydration kinetics by microwave energy. After the optimization of operation parameters, calculation of kinetic parameters can be experimented in future studies.

REFERENCES

- [1] T.R. Prime Ministry SPO, Commission. Boron Operations Group Report: Ninth development plans (2007-2013) *Chemical Industry Private Expertise*, 2006.
- [2] U. Dosler, M. M. Krzmann, D. J. Suvorov, "The synthesis and microwave dielectric properties of $Mg_3B_2O_6$ and $Mg_2B_2O_5$ ceramics", *Eur Ceram Soc.*, vol. 30, pp. 413–418, 2010.
- [3] E. M. Elssfah, A. Elsanousi, J. Zhang, H. S. Song, C. Tang, "Synthesis of magnesium borate nanorods", *Materials Letters*, vol. 61, pp. 4358 – 4361, 2007.
- [4] L. Zhihong, H. Mancheng, "Synthesis and thermochimistry of $MgO \cdot 3B_2O_3 \cdot 3.5H_2O$ ", *Thermochim Acta.*, vol. 403, pp. 181 – 184, 2003.
- [5] A.S. Kipcak, F.T. Senberber, E. Moroydor Derun, S. Piskin, "Hydrothermal Synthesis of Magnesium Borate Hydrates from MgO and H_3BO_3 at 80°C", *2011 Research Bulletin of the Australian Institute of High Energetic Materials*, vol. 1, pp. 47-55, 2011.
- [6] A.S. Kipcak, F.T. Senberber, E. Moroydor Derun, S. Piskin, "Characterization of Magnesium Borate Hydrates Produced from MgO and H_3BO_3 at 80°C, for two Different Drying Temperatures", *12th Mediterranean Congress of Chemical Engineering*, November, 2011.
- [7] A.S. Kipcak, F.T. Senberber, E. Moroydor Derun, S. Piskin, "Evaluation of the Magnesium Wastes with Boron Oxide in Magnesium Borate Synthesis", *International Conference on Environmental, Biological, and Ecological Sciences, and Engineering*, July, 2012.
- [8] A.S. Kipcak, F.T. Senberber, E.M. Derun, S. Piskin, "Characterization and thermal dehydration kinetics of admontite mineral hydrothermally synthesized from magnesium oxide and boric acid precursor" Research on Chemical Intermediates, 2013 (to be published).
- [9] Y. Saito, K. Kawahira, N. Yoshikawa, H. Todoroki, S. Taniguchi, "Dehydration behavior of goethite blended with graphite by microwave heating", *Journal of the Iron and Steel Institute of Japan*, vol. 51, pp. 878–883, 2011.
- [10] S. Sener, G. Ozbayoglu, S. Demirci, "Changes in the structure of ulexite on heating", *Thermochimica Acta*, vol. 362, pp. 107-112, 2000.
- [11] M. Tunc, H. Ersahan, S. Yapici, S. Colak, "Dehydration kinetics of ulexite from thermogravimetric data", *Journal of Thermal Analysis*, vol. 48, pp. 403-411, 1997.
- [12] S. Kocakusak, J.H. Koroglu, E. ekinci, R. Tolun, "Production of anhydrous borax using microwave heating", *Industrial & Engineering Chemistry Research*, vol. 34, pp. 881-885, 1995.
- [13] D.E. Clark, D.C. Folz, J.K. West, "Processing materials with microwave energy", *Materials Science and Engineering*, vol. A287, pp. 153–158, 2000.
- [14] K.E. Haque, "Microwave energy for mineral treatment processes—a brief review", *International Journal of Mineral Processing*, vol. 57, pp. 1-24, 1999.
- [15] W. Verstor, The effect of microwave radiation on mineral processing. University of Birmingham, PhD Thesis, 2011.
- [16] F.C. Iglesias, C.E.L. Hunt, R.M. Hutcheon, G.W. Wood, R.D. Barrand, Tests of Ontario's industrial minerals", Ontario Geological Survey, pp. 41-77.
- [17] Y. Saito, K. Kawahira, N. Yoshikawa, H. Todoroki, S. Taniguchi, "Dehydration behavior of goethite blended with graphite by microwave heating", *The Iron and Steel Institute of Japan International*, vol. 51, pp. 878–883, 2011.
- [18] Y. Li, Y. Lei, L. Zhang, J. Peng, C. Li, "Microwave drying characteristics and kinetics of ilmenite", *Transactions of Nonferrous Metals Society of China*, vol. 21, pp. 202–207, 2011.



Emek Moroydor Derun was born in Istanbul in 1976. Moroydor Derun graduated from B.Sc. in 1998, M.Sc. in 2000 and Ph. D. in 2005 from Chemical Engineering Department at Yildiz Technical University, Istanbul. Her research interest is in the area of waste management, lightweight concrete, semi conductive materials and boron technology. She has many articles and studies in international and national conference proceedings and articles.



Fatma Tugce Senberber was graduated from B.Sc. at Yildiz Technical University in 2010. After she completed her M.Sc. studies at Yildiz Technical University in 2012, she started to Ph.D. studies at the same year and same department of university. She is interested in boron technologies such as alternative synthesis methods of boron minerals and evaluation of industrial wastes in synthesis process. She also studied the characterization methods by instrumental analysis and kinetic studies of minerals.



Azmi Seyhun Kipcak was graduated from Department of Chemical Engineering in Ege University in 2002. After completing the university studies he graduated from Bilgi University from the department of Master of Business Administration in 2004. He worked in Kultur University from 2003 to 2007 as a research assistant then he transferred to Yildiz Technical University at 2008, where he started his M.Sc. studies about Chemical Engineering in 2006. He completed his M.Sc. studies at Yildiz Technical University in 2009 and started to Ph.D. studies at the same year. He studied on neutron shielding with boron minerals and the characterization of boron minerals by using XRD, XRF, FT-IR, Raman, DTA/TG, DSC and ICP-OES at the graduate studies now he is studying on the synthesis of magnesium borates from different raw materials and wastes. Also he is improving the neutron shielding studies with the synthesized materials and working on the element analysis of Turkish Teas and Coffees. Another research field about the studies is the fly ash characterization.



Nurcan Tugrul was born in Gaziantep in 1973. Tugrul was graduated from B.Sc., M.Sc. and Ph.D. in Chemical Eng. Department at Yildiz Technical University, Istanbul. Her research interest is in the area of chemical technologies, evaluation of industrial wastes, food drying. She has many articles and studies in international and national conference proceedings and articles.



Sabriye Piskin graduated from Istanbul Technical University on Chemical Engineering with M.Sc. degree in 1974. She completed a Ph.D. degree at the same department in 1983. Her research interests include boron minerals and compounds, hydrogen storage technologies, fuel cell applications, materials characterization, coal, waste management, corrosion, implants and synthetic materials production. She has more than fifty articles and eighty conference manuscripts pressed at the international area.