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Enrichment of Low Grade Eti Mine-Espey Colemanite Stocks Using Microwave Methods

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Abstract: In this study, the benefication of low-grade Espey -10 mm. colemanite stocks of more than 30,000 tons, which have been waiting for longer years, can not be evaluated in any way in Emet Boron Works in order to get salable product. Chemical, physical, mineralogical and thermal characterization of low-grade colemanite stock, in the sizes of 10 mm, are located in Espey region primarily conducted. Grades of 10 mm size of Espey used in this study is 28.87% B_2O_3. In the microwave experiments, a concentrate of 50.82% B_2O_3 was obtained with the efficiency of 88.13%. Evaluating colemanite concentrate by microwave technology may have great importance in solving environmental problem.

Key words: Colemanite, microwave, environment.

1. Introduction

Turkey has the largest boron reserves in the world. Boron deposits are located in four regions, namely, Eskisehir-Kirka, Kutahya-Emet, Balıkesir-Bigadic and Bursa-Kestelek. Boron ores are generally upgraded by concentration techniques including physical, chemical and physicochemical processes [1].

As a new method, microwave heating has been found wider and wider applications in process of synthesis and materials because the heating is based on the interaction between microwaves and materials [2-6]. During microwave heating, materials having small and polar molecules absorb more microwave energy than others. Microwave heating provides advantages as a volumetric heating rather than a surface heating or conventional heating by thermal diffusion. Very fast heating rates (several degrees per second) can be obtained through the microwave heating in relation to volume [7].

Microwave equipment can be easily adapted to automated systems, and can be quickly started and stopped, with its power level adjusted electronically [8]. Microwaves can offer considerable savings of energy and processing times. In addition, in microwave heating it is possible to control the spatial distribution of the energy transferred to the material. That is, it is possible to heat the materials either in selected and localised regions, or in uniform regions, depending on the specific application [9].

With microwaves, the energy transfer is not primarily based on conduction or convection as in conventional heating, but through dielectric loss. Thus, propensity of a sample to undergo microwave heating is highly dependent on its dielectric properties. The dielectric loss factor (loss factor; *ε* __) and the dielectric constant (*ε* _) of a material are two determinants of the efficiency of heat transfer to the sample (for the ulexite sample used in this study, the dielectric loss factor *ε* __ and the dielectric constant *ε* _ were measured as respectively 0.2294 and 8.7314). Their quotient (*ε* __/*ε*_) is the dissipation factor (tan δ), high values of which indicate ready susceptibility to microwave energy. Materials dissipate microwave energy by two main mechanisms: dipole rotation and ionic conduction.

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At 2,450 MHz, the field oscillates 4.9×10^9 times per second, and sympathetic agitation of molecules generates heat. The efficacy of heat production through dipole rotation depends upon the characteristic dielectric relaxation time of the sample, which is affected by temperature and viscosity. The second dissipation mechanism, ionic conduction, is the migration of dissolved ions with the oscillating electric field. Heat generation is due to frictional losses which depend on the size, charge and conductivity of the ions, and their interactions with the solvent [2].

Microwave heating of solid state reaction mixtures is the most established application in inorganic chemistry. Reactions can be considerably accelerated by microwave irradiation. Rate enhancement factors up to over one thousand, in comparison with classical methods of heating, have been recorded. This remarkable rate increase was attributed to microwave heating having “specific effects” or “non-thermal effects” [5, 10, 11]. Thermal analysis using microwave radiation was investigated by Karmazsin [12, 13] with differential thermal analysis of calcium hydrogen phosphate dihydrate ($CaHPO_4 \cdot 2H_2O$).

Microwave energy is a nonionizing electromagnetic radiation with frequencies in the range of 300 MHz to 300 GHz. Microwave frequencies include three bands: the ultra high frequency (UHF: 300 MHz to 3 GHz), the superhigh frequency (SHF: 3 GHz to 30 GHz) and the extremely high frequency (EHF: 30 GHz to 300 Gfu). Microwaves have extensive application in the field of communication; however, the federal Communication Commission (FCC) has allocated certain frequencies for Industrial, Scientific, Medical and Instrumentations (ISMI) applications (14, 15, 16). Currently, 2,450 MHz is the most commonly utilized frequency for the home microwave oven which was invented by Percy L. Spencer almost 50 years ago (14, 17). It is reported that annual sale of home microwave ovens in the USA is $1.5 to $2.0 billion (14, 17).

Microwaves can be reflected from a simple mirror such as a metallic sheet, be refracted at a dielectric interface, and be focused by parabolic reflectors or horn antennas (14). Microwave energy is derived from electrical energy with a conversion efficiency of approximately 50% for 2,450 MHz and 85% for 915 MHz. Microwaves have longer wave lengths and lower available energy quanta than other forms of electromagnetic energy such as visible, ultraviolet or infrared light (14).

Microwave heating is unique and offers a number of advantages over conventional heating such as:

- non-contact heating;
- energy transfer, not heat transfer;
- rapid heating;
- material selective heating;
- volumetric heating;
- quick start-up and stopping;
- heating starts from interior of the material body;
- higher level of safety and automation.

It can be transported from the source through a hollow nonmagnetic metal tube (i.e., waveguide).

A number of advantages at microwave technology, in this study, has been tried at boron minerals as a calcination.

2. Material and Methods

2.1 Material

The sample is taken from Espey -10 mm. Low grade colemanite with 28.87% B_2O_3 have been waiting for approximately long years in the stocks of Emet Boron Works and cannot be utilized in any way.

Approximately 500 kg of sampleis stocked for sieve analysis, chemical analysis (XRF), mineralogical analysis (XRD). TG-DTA analysis is done for microwave experiments. The chemical analyses of the sample is given in Table 1.

According to the XRD analysis, the sample contains colemanite, quartz, calcite and clay minerals. The XRD analysis of the sample is given in Fig. 1.

Table 1 The chemical analyses of the sample.

Content	B_2O_3	As (ppm)	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	SrO	SO_4	L.I
Quantity (%)	28.87	869	13.48	2.43	20.07	0.88	9.12	3.15	0.13	12.45

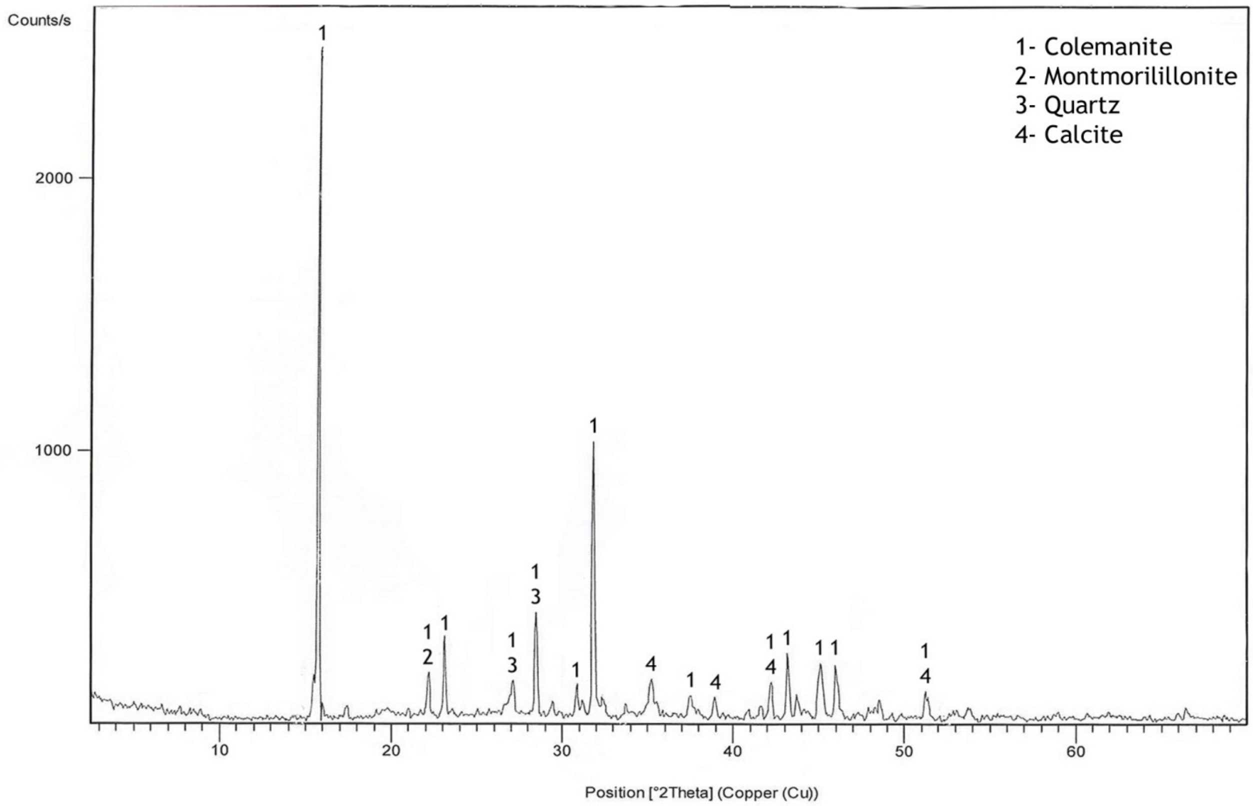

Fig. 1 The XRD analysis of the sample.

In DTA-TG analysis, the sample begins to lose water and 2.39% in total of the loss takes place at 100 ^oC level. The sample begins to lose the crystal water between 387 ^oC and 550 ^oC in a large portion. At 740 ^oC, there is a result of the crystallization exothermic process. The DTA-TG analysis of the sample is given in Fig. 2.

2.2 Method

In the studies, Arcelik MD554 (intellowave) microwave oven is used. Samples were prepared for studies as 100 grams. After setting the sample at certain periods, different Powers (watt) were subjected to experiments.

In this study, 360 watt, 600 watt, 900 watt and 1,200 watt of power have been used in 10, 20, 30, 40, 50, 60 and 70 minute of periods. First, about 360 watts for one hour of soak, but no enrichment could be detected.

Then it has started to explode with 600 watt capacity of colemanite increases and time intervals were identified. After a certain period, sintering has begun and it has stirred at regular intervals. Clay and colemanite converge in the form of agglomerates. 900 and 1,200 watts has been suited on the same studies, and the results of the experiments are given in Table 2, Fig. 3 and Fig. 4. Espey -10 mm low-grade colemanite stocks, depending on the duration and yield strength.

In Fig. 3, it can be seen that, in conditions of 1,200 watt and 30 min. it reaches maximum yield of 88.13%.

In Fig. 4, in conditions of 30 min and 1,200 watt, it has reached maximum grade of 50.82% B_2O_3, indicating optimum result in these conditions. In Table 3, it has given chemical analysis of optimum results.

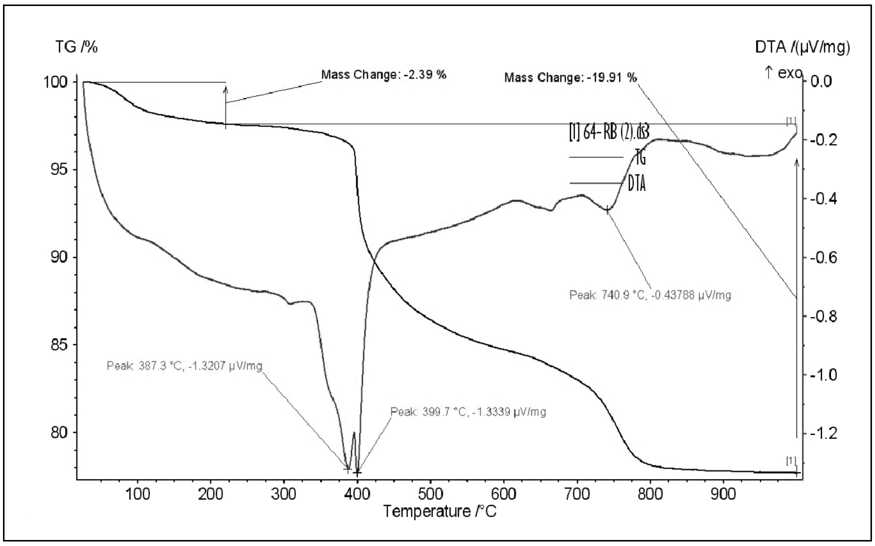

Fig. 2 DTA-TG analysis of the sample.

Table 2 The effect of the time and power in microwave studies.

Time (min)	Products	600 watt			900 watt			1,200 watt		
		Weight (%)	B_2O_3 (%)	Yield (%)	Weight (%)	B_2O_3 (%)	Yield (%)	Weight (%)	B_2O_3 (%)	Yield (%)
	Concentrate	33.53	44.63	46.71	48.81	49.5	68.64	54.33	50.43	77.02
20	Tailing	66,47	25.69	53.29	51.19	21.6	31.36	45.67	17.9	22.98
	Total	100	32.04	100	100	35.2	100	100	35.57	100
	Concentrate	45.08	48.38	66.8	55.84	49.8	80.52	*59.49*	*50.82*	*88.13*
30	Tailing	54.92	19.73	33.2	44.16	15.2	19.48	*40.51*	*10.05*	*11.87*
	Total	100	32.64	100	100	34.6	100	*100*	*34.3*	*100*
	Concentrate	48.75	49.01	71.68	59.77	50.1	86.55	56.73	49.35	84.09
40	Tailing	51.25	18.42	28.32	40.23	11.6	13.45	43.27	12.24	15.91
	Total	100	33.33	100	100	34.6	100	100	33.29	100
	Concentrate	58.5	48.9	78.68	56.75	49	82.28	55.14	48.25	80.33
50	Tailing	41.5	18.68	21.32	43.25	13.8	17.72	44.86	14.52	19.67
	Total	100	36.36	100	100	33.8	100	100	33.12	100
	Concentrate	56.4	48.54	79.13	54.18	48.3	76.14			
60	Tailing	43.6	16.56	20.87	45.82	17.9	23.86	-		
	Total	100	34.6	100	100	34.3	100			
	Concentrate	55.06	47.16	78.27						
70	Tailing	44.94	16.04	21.73	-			-		
	Total	100	33.17	100						
Grade of the sample		32.62								

As a result of the studies, on low power long time or high power short time, optimum results can be achieved. As it can be seen from the figures and tables in the studies, 600 watt, 900 watt and 1,200 watt have been used. The optimum results obtained at 1,200 watt and 30 min. As optimum results the chemical analysis is given at Table 3.

In Table 1, from the analysis, the grade of sample is found to be 28.87% of B_2O_3. Preliminary studies showed that 0.5 mm is optimum for sieving. The grade of the sample sieved from 0.5 mm, increased to 32.62% B_2O_3. In conditions of 1,200 watt and 30 min, 50.82% B_2O_3 is obtained with the yield of 88.13%. The B_2O_3 grade of the tailing is reduced to 10.05%.

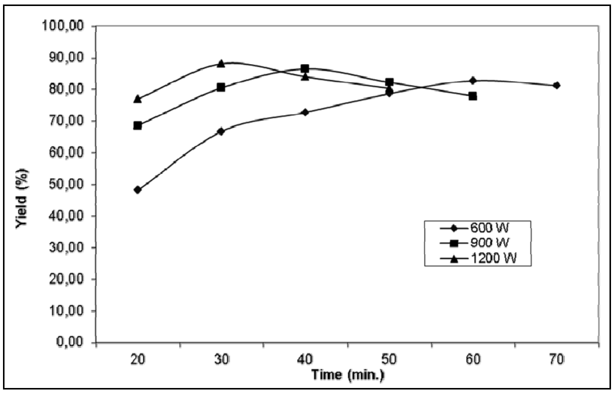

Fig. 3 The changes of the yield according to the power and time.

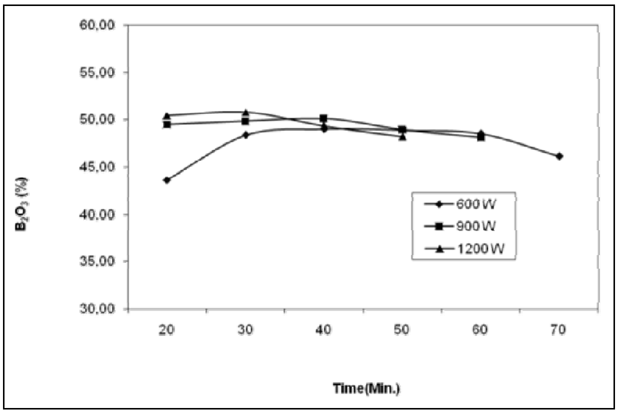

Fig. 4 The changes of the grade according to the power and time.

Table 3 The optimum results of the sample.

Content	Sample	1,200 Watt- 30 min.	
(%)	(%)	Concentrate (%)	Tailing (%)
B_2O_3	32.62	50.82	10.05
SiO_2	9.49	3.98	20.9
Al_2O_3	1.72	1.11	4.21
CaO	22.04	32.57	25.6
Fe_2O_3	0.64	0.55	2.46
MgO	5.1	1.63	6.36
K_2O	1	0.47	1.99
Na_2O	0.08	0.02	0.11
SrO	2.31	2.63	1.17
SO_4	0.08	0.08	0.58
As (ppm)	719	348	1,159.00
TiO_2	0.38	0.05	0.4

3. Results

At the end of the studies; the benefication of low-grade Espey -10 mm. colemanite stocks of more than 30,000 tons which have been waiting for longer years, is evaluated by microwave technology. This is a new technology for boron minerals and there is much need to do more studies about microwave technology. According to the results, Espey -10 mm sample's grade is found to be 28.87% B_2O_3. Preliminary studies showed that 0.5 mm is optimum for sieving. After sieved from 0.5 mm, the sample's grade is rosed to 32.62% B_2O_3.

During the microwave studies 600 watt, 900 watt and 1,200 watt power are seperately used at 20, 30, 40, 50, 60 and 70 min, for 10 min, is not suitable for utilization because colemanite has not started to explode in 10 minutes. Optimum result is obtained at 1,200 watt power and 30 min. The concentrate is producted in 50.82% B_2O_3 grade with the 88.13% yield. It is understood that colemanite can be enriched with microwave method easily.

- In microwave studies low-grade sample is enriched and salable product is achieved. Microwave method should be put into application and methods of implementation should benefit from the advantages of an industrial area.
- It can be concluded that large stocks would indulge environmental problems.
- Using microwave method, adversities of the wet method will be eliminated. The need for a new tailings dam will disappear.
- The tailings that are produced during decrepitation can be evaluated in various fields like ceramic, cement and brick industry.
- The colemanite concentrate will be sold as a microwave product. Thus adversities caused by the wet method will be eliminated.

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