

Dehydration of ulexite by microwave heating

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Abstract

Microwave heating of a solid state reaction is an established application in inorganic chemistry. Dehydration of ulexite samples having different particle sizes were investigated using 150, 300, 450 and 600 W, 2450 MHz microwave radiation energy. The rate and temperature increase with decreasing particle size and increasing microwave power.

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1. Introduction

About 62% of the world's known boron reserves are in Turkey [1,2]. Boron minerals are hydrated borates combined with alkaline-earth and alkaline-boron metals such as Na, Ca, and Mg. The boron reserves commercially recoverable are mostly in the form of hydrated boron minerals such as pandermite $\text{Ca}_4\text{B}_{10}\text{O}_{19} \cdot 7\text{H}_2\text{O}$, ulexite $\text{NaCaB}_5\text{O}_9 \cdot 8\text{H}_2\text{O}$, tincal (borax) $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ and colemanite $\text{Ca}_2\text{B}_6\text{O}_{11} \cdot 5\text{H}_2\text{O}$ [3].

Dehydration of hydrated boron minerals is important in the production of boron compounds [4]. The dehydration has been investigated generally with thermogravimetric methods. Şener et al. determined the thermal reactions of ulexite by TG, DTG and DTA. Decomposition occurred from 60 to 500 °C with two steps followed by two dehydroxylation stages [4]. Erdoğan et al. analysed the dehydration kinetics of howlite, ulexite, tunellite by DTA and TG methods. Activation energies, rate constants and pre-exponential factors were calculated. The dehydration reactions were first order [2]. Tunç et al. investigated the kinetic parameters of thermal decomposition of ulexite by TGA. The Suzuki and Coats–Redfern methods were applied. The process was first-order and the

activation energy and frequency factor decreased with decreasing particle size [1]. Okur and Eymir studied calcination kinetics of ulexite by thermogravimetry (TG) with Coats–Redfern and genetic algorithm methods. They concluded that the Coats–Redfern method would not be used for reactions having low activation energies [5]. Davies et al. investigated thermal decomposition of powdered colemanite by flash calcination. The study demonstrated an optimum calcination temperature around 600 °C. Calcines produced at this temperature have a very porous structure, probably caused by explosive dehydroxylation [3]. Çelik and Suner studied the decrepitation properties of colemanite and ulexite. A thermodynamic evaluation of fourteen boron minerals has shown that only colemanite exhibits decrepitation [6].

When boron minerals are heated, the mineral first loses water of crystallization, followed by production of amorphous material or recrystallization into new phases. Colemanite decrepitates as a result of the sudden release of confined water vapour within micropores during thermal treatment, while ulexite does not decrepitate. Instead, it exfoliates as a result of gradual water vapour removal, and the structure becomes amorphous with numerous microcracks and interstices [4].

Recently, the method of microwave heating has been finding wider and wider applications in synthesis and processing of materials because the heating is due to the interaction

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between microwaves and materials [7–11]. During microwave heating, materials having small, polar molecules absorb microwave energy more than others. Microwave heating provides advantages as a volumetric heating rather than the surface heating and thermal diffusion afforded by conventional heating. Very fast heating rates (several degrees per second) can be obtained, depending on the microwave power used in relation to volume [12]. Microwave equipment can be adapted easily to automated systems, can be quickly started and stopped, and its power level can be adjusted electronically [13]. Microwaves can offer considerable energy savings and a shortening of 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, localised regions or more uniformly, depending on the specific application [14].

With microwaves, the energy transfer is not primarily by conduction or convection as in conventional heating, but by 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 ($\tan \delta$), high values of which indicate ready susceptibility to microwave energy. Materials dissipate microwave energy by two main mechanisms: dipole rotation and ionic conduction. At 2450 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 in turn, is dependent on 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 [7].

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 microwave heating having “specific effects” or “non-thermal effects” [10,15,16]. Thermal analysis using microwave radiation was investigated by Karmazsin et al. [17,18] with differential thermal analysis of calcium hydrogen phosphate dihydrate ($\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$). The experiments were performed in a single-mode microwave cavity. Bond et al. [19] also used microwave radiation to linearly increase the temperature of a sample within the cavity.

Harrison and Rowson [20] investigated the heating rates of pure minerals and concluded that most valuable minerals are good absorbers of microwave energy, but gangue miner-

als are not. As most mineral ores do not occur in pure form in nature, but as mixtures of valuable minerals and gangue, the different response of the ore components to dielectric heating causes differential heating. Because minerals expand with an increase in temperature, differential stresses occur at the grain boundaries, thereby weakening the material [21]. Spherical composites of Fe_2O_3 and carbon were heated with microwave power (2.45 GHz) and their heating rate was studied by Standish et al. [22]. They improved a mathematical model that described the principal features of heating these solids by microwave energy.

2. Experimental

Ulexite samples used throughout the study were supplied from the deposits around Eskisehir-Kirka in Turkey. Hand picked coarse crystals were cleaned by a brush to remove impurities such as dispersed clay minerals. Cleaned samples were crushed and ground by a jaw-crusher and a laboratory type ball mill. Then samples were screened through 1180 and 212 μm ASTM standard screens. Four different particle sizes ($-1180 + 850$, $-850 + 600$, $-600 + 425$, $-300 + 212 \mu\text{m}$) of the mineral were used in the experiments. The composition of the sample determined by chemical analysis was 7.14% Na_2O , 13.56% CaO , 42.33% B_2O_3 , 35.53% H_2O and 1.44% other.

A laboratory type microwave reactor (Fig. 1) with a frequency of 2450 MHz and a maximum output power of maximum 1 kW was used. The apparatus occurs from five sections. The first section of the apparatus includes the generator that can be adjusted with manual and automatic control. The second section consists of a PHILIPS microwave circulator with a frequency of 2.475 GHz. The third section is an R26 standard rectangular waveguide. Three manually adjustable stub tuners inserted in the waveguide section and the tuning plunger of the applicator are used to maximize microwave absorption by minimizing the reflected power. The fourth section consists of an MUFGGE DIR COUPLER MM 1002C model coupler that measures reflected power. The fifth part is the cavity section with two 15 mm holes. The bottom one of these holes is open and the other one closed. Mass was measured by an MW II-300 model scale. For temperature measurements, an optical fiber connected to a transducer (PT-100) and Ni–Cr thermocouples were used. The experimental design is given in Fig. 1.

In the study, two pyrex tubes with diameter of 11 mm were used. Microwave irradiated parts of tubes were filled with 2–2.5 g ulexite and they were installed in the applicator at the position of the highest electric field. To measure loss of weight the bottom of first tube was placed on the scale by a Teflon support. The second tube was installed through the other hole in the cavity. Changes in the temperature were determined by means of a PT-100 thermocouple inserted into the second tube. Weight loss and temperature were recorded as a function of time under constant power.

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