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Influence of milling parameters on particle size of ulexite material

Sezai Kutuk *

Department of Physics, Faculty of Science and Letters, Artvin Coruh University, 08100 Artvin, Turkey
Department of Electronics and Automation, Vocational School of Technical Sciences, Recep Tayyip Erdogan University, 53100 Rize, Turkey

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ABSTRACT

Commercial raw ulexite (U) materials were milled from 0 h to 8 h by using mechanical milling technique for various initial powder size (−3 mm; −75 μm), ball to powder ratio (5:1; 10:1), and ball size (10 mm; 5 mm). Particle size, morphology, elemental, and crystal structure measurements of the milled powders were performed. In the particle size distribution analysis, the smallest d_{50} and d_{10} values were respectively found as 8.846 μm and 790 nm for U_5m powder (obtained from that initial powder with −3 mm in size was milled by balls with 5 mm in size) at 0.5 h. In the morphology analysis, the microstructure of the U_5m powder was observed to be more homogeneous by means of milling process. In the elemental analysis, it was deduced that the U_5m powder is not a pure compound. In the crystal structure analysis, it was determined that the crystal structure of the U_5m powder is exponentially deteriorated with increasing the milling time, and then it has become an amorphous structure at the end of 8 h. The crystalline size of the U_5m powder is reduced to 15.5 nm after 4 h of milling. The results of this study are considered to be useful for future nano studies and industrial applications of ceramic ulexite compound, which is a boron mineral.

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1. Introduction

Approximately 73% of boron reserves, containing 955 million tons of boron oxide (B_2O_3) known on the worldwide, exist in Turkey [1]. A large number of commercially recovered boron reserves consist of colemanite, ulexite, tincal and pandermite minerals (borates, which are an extraordinarily large group of minerals). Among these minerals, the ceramic ulexite compound has a chemical formula of $Na_2O{\cdot}2CaO{\cdot}5B_2O_3{\cdot}16H_2O$ (sodium-calcium-borate hydrate) and has found in large amounts in Turkey. The ulexite mineral has been usually obtained together with other borates. Demand for the ulexite mineral has rapidly increased up to now because of the fact that it is utilized as a raw material in the production of boron compounds. The ulexite mineral has extensive industrial applications such as fabrication of glass, porcelain, leather, cosmetics and photographic chemicals, detergent materials, polymer, catalysts, steel, refractory materials, fertilizers, disinfectant, food preservative, textiles, nuclear fiberglass, insulators, etc. [2,3]. Besides, it has potential technological applications such as hydrogen energy ($NaBH_4$) [4], superconducting material (MgB_2) [5], shielding material [6], ultra-high temperature ceramic material (ZrB_2) [7], cement concrete [8], asphalt concrete [9], brick [10]. Therefore, the ulexite mineral is an important mineral at both scientific and technological community owing to its attractive physical and mechanical properties.

Nano-sized materials have been intensely studied due to their better physical, mechanical and magnetic properties compared to micro-sized materials. This reason is in reduction of crystalline/particle size of the material to nano-scale level. In order to accomplish this, there are many preparation techniques. One of the beneficial techniques is mechanical milling (MM). The MM technique can easily provide the nano-sized powders with cheaper equipment, and thus it can be a great potential to be used for mass production [11]. This technique is a solid-state reaction process including repeated deformation, welding and fracturing of the powder particle. A lot of materials such as amorphous alloys, inter-metallic compounds, composites and ceramics have been prepared by using the MM technique. As a result of the MM technique, the final product powder has generally a nano-sized structure indicating better properties in comparison to micro-sized structure [12]. For example, nano-sized ceramic materials show better sinterability, strength, fracture toughness, resistance to oxidation and creep resistance [7].

The MM technique is performed by the high-energy ball milling. There are various types of ball milling techniques in relation to the motion of balls and vial(s) such as planetary, vibration and attritor. Among them, the planetary ball milling depends on different parameters affecting the particle size refinement. These parameters are milling time, ball to powder (weight) ratio (BPR), process control agent (PCA), size of the ball, size of the vial, rotation speed, atmosphere of milling, etc.

* Department of Physics, Faculty of Science and Letters, Artvin Coruh University, 08100 Artvin, Turkey.
E-mail addresses: sezai_kutuk@hotmail.com, sezai.kutuk@erdogan.edu.tr.

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Therefore, selection of the parameters is significant since it changes considerably with type of the material used [13,14].

Li et al. revealed that nano-particles of boron nitride (BN) were synthesized from B_2O_3 by a ball milling technique and annealing process. Firstly, average particle size of the B_2O_3 powder is reduced from 200 μm to 20 μm after 60 h of milling time. Eventually, the nano-particles generally disperses uniform and their diameters are in the range of 20–100 nm. In addition, most of them are close to 70 nm in diameter (average particle size) [15]. Jung et al. published that nano-sized BN powder was produced by a ball milling technique and its average grain/particle size decreases from 10 μm (initial size) to 100 nm (final size) [16].

Alizadeh et al. declared that nano-structured B_2O_3 powder was prepared by a ball milling technique and the crystalline size of the B_2O_3 powder is reduced from >100 nm to 19 nm after 5 h of milling time [11].

Xu et al. investigated that boron (B) powder was ground by using a ball milling technique and it was employed to fabricate a superconducting material (MgB_2). They determined to improve the critical current density (J_c) of the material with the crystalline size refinement of 100 nm [17]. Jung et al. reported that B powders with average initial particle size of 800 nm were ground by a ball milling technique under dry milling and wet milling processes. The smallest particle sizes are 58 nm and 220 nm under the dry and wet milling processes, respectively [18].

Zhang et al. examined that SiBCN powder largely comprises of near-spherical agglomerates 6.6 ± 5.3 μm in average particle size resulting from the hard agglomeration of nano-primary particles, which were fabricated by using a ball milling technique. However, they observed that in fact these nano-primary particles are 190.8 ± 33.6 nm in average particle size [19]. In another study, they found that SiBCN powder has an average particle size ranging from 4.6 μm to 5.3 μm due to agglomerate, but average particle size of nano-primary particles is 190.8 ± 33.6 nm. Furthermore, optimization parameter values of milling process were determined as 600 rpm in milling speed, 20:1 in BPR and 30–40 h in milling time [20].

Vignola et al. investigated that nano-structured B powder was achieved with average grain size of 80 nm. The B powder was used to synthesize a MgB_2 superconducting material and consequently increases its superconducting properties such as J_c and the critical temperature. Thus, it is indicated that the B powder works very well which makes it possible for a development on large scale fabrication [5].

No studies have been conducted yet considering optimum condition of nano-sized ulexite material by milling in terms of both science and applied engineering in the literature. Therefore, the main aim of this study is to examine the effect of milling parameters on the particle size of the ulexite material. Afterward, in another study, the best results obtained from here will be employed in both cement concrete and asphalt concrete at civil engineering for the purpose of investigating influence of the nano-sized ulexite material.

2. Experimental details

2.1. Materials

Commercial raw ulexite (U) materials in this study were supplied from Eti Mine Works General Management's Bigadic Boron Mine (Eti Maden) in Turkey. These materials which were eliminated −3 mm (coarse powder, U-3m) and −75 μm (fine powder, U-75μ) scaled sieves with the ASTM (the American Society for Testing and Materials) standard were used as initial materials. Chemical composition analyses of the initial materials were carried out by the Eti Maden, and their results are presented in Table 1 [21,22]. Composition difference between the U-3 m and the U-75 μ materials is attributed that the raw ulexite material mined passes through several physical and chemical processes (washing, crushing, sieving, decomposition, dissolution, milling and so on).

Table 1
Chemical composition analyses of ulexite initial materials.

Compound	Ulexite (wt.%)	
	−3 mm	−75 μm
B_2O_3	25.50 ± 1.50	37.00 ± 1.00
CaO	21.00 ± 3.00	19.00 max
SiO_2	13.00 max	4.00 max
Na_2O	2.00 min	3.50 max
SO_4	0.60 max	0.25 max
As	40 ppm max	40 ppm max
MgO		2.50 max
SrO		1.00 max
Al_2O_3		0.25 max
Fe_2O_3		0.04 max
Humidity		1.00 max

2.2. Milling process

The initial materials were ground by a planetary high-energy ball mill for 0.5 h, 1 h, 2 h, 4 h and 8 h, respectively. Mechanical milling process was performed by using this mill (Retsch, model 'PM 100') and using a 250 ml zirconium oxide vial and both 5 mm and 10 mm in diameter zirconium oxide balls at room temperature [23]. BPR and rotational speed was 10:1; 5:1 and 500 rpm, respectively. In order to prevent overheating during milling, the mill was stopped every 15 min and then resumed after 5 min in the opposite direction. Table 2 shows details of the milling experiments.

Milling names were henceforth respectively termed as U; U_75μ; U_10:1; U_5m for simplicity instead of that U_3m_5:1_10m; U_75μ_5:1_10m; U_3m_10:1_10m; U_3m_5:1_5m were written in following format: Powder name_Initial powder size_BPR_Ball size (see Table 3).

2.3. Measurements

The particle size distributions of the initial materials and milled powders were measured by a laser size analyzer (Malvern, model 'Mastersizer Hydro 2000 MU'). This analyzer scanned four times and then averaged for each powder. d_{50}, d_{10} and d_{min} values corresponding to respectively 50%, 10% and minimum percent passing volumes were determined from the particle size distribution.

Table 2
Milling parameters and their average particle sizes.

Experimental no.	Initial powder size (m)	BPR	Ball size (mm)	Milling time (h)	d_{50} (μm)
1	−3m	5:1	10	0	444.220
2				0.5	14.331
3				1	63.836
4				2	67.631
5				4	22.528
6				8	19.034
7	−75μ	5:1	10	0	11.524
8				0.5	21.453
9				1	18.585
10				2	15.679
11				4	13.964
12				8	18.526
13	−3m	10:1	10	0.5	22.282
14				1	25.757
15				2	18.732
16				4	12.087
17				8	10.582
18	−3m	5:1	5	0.5	8.846
19				1	13.786
20				2	14.979
21				4	17.994
22				8	14.242

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