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The Effects of Ball-Mill Grinding Parameters on Lignite Morphology

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Abstract: In recent years, because of the decreasing liberation sizes of the minerals, processes such as grinding need to be evaluated in more detail. As is well known, size reduction processes are very important both in mineral processing and in many industrial applications. However, to increase the efficiency of the processes after size reduction, variations in particle morphology should also be evaluated, along with particle size. Although the effectiveness of grinding media (ball, rod, autogenous) has been shown for different materials, there are very few studies on the effect of the powder/grinding media ratio and grinding time on particle morphology in terms of shape factor and roughness values. This study aims to investigate the variations in the morphology of lignite samples under different grinding conditions such as grinding time and powder/grinding media ratio (U). The results of these analyses showed that while the d_{80} size of the ground lignite was 1.1 mm after 2 min grinding time, it decreased to 0.5 mm following 15 min grinding time. The roundness values of particles vary in the range of 0.746–0.790 with increasing grinding time. In addition to the grinding time, while the roundness of particles was found to be 0.739 for 0.34 U values (powder/grinding medium rate), it increased to 0.788 when the U value was adjusted to 0.67. The average roughness (Ra) values of particles increased from 60.9 nm to 107.9 nm upon increasing the grinding time from 2 min to 16 min. Due to these findings, it can be suggested that lignite samples became rounder with increasing grinding times, and roughness analyses made in a 10 × 10 µm surface area with an Atomic Force Microscope (AFM) indicated that particle roughness increased in direct proportion to grinding time.

Keywords: morphology; roughness; grinding; lignite; AFM

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Citation: Guven, O.; Ehsani, A.; Kaymakoğlu, B. The Effects of Ball-Mill Grinding Parameters on Lignite Morphology. *Minerals* **2023**, *13*, 1185. https://doi.org/10.3390/min13091185

Academic Editor: William Skinner

Received: 25 June 2023
Revised: 4 September 2023
Accepted: 7 September 2023
Published: 9 September 2023

1. Introduction

To meet the basic needs of an increasing world population, the demand for raw materials and mines is increasing [1]. This situation obligates the mineral processing of ores, which has been problematic in previous years due to both economic and technological reasons. For this reason, much research is being conducted on how to bring low-grade, difficult-to-grind, and finer-sized ores to the economy [2].

This situation makes it necessary to examine parameters such as the type of grinding material (coal/ore), grinding type/medium, grinding technique, particle size, shape, size distribution, morphology, liberation size, etc., in detail for the application of more efficient enrichment techniques, especially for flotation, leaching, etc. [3–6]. In the literature, many studies have been carried out to show the contribution of different parameters on not only particle size but also the morphology of the particles, which in turn significantly affects the aforementioned processes. In this context, the literature survey demonstrates that, as opposed to well-studied grinding conditions for different parameters, to our knowledge, very few studies investigated the effect of grinding time and the ratio of powder/grinding media ratio "U" value [7–9] to the morphology of the ground particles. As presented in these studies, the response to grinding time will vary based on the properties of the target material and the particle size range. In addition, although the effect of grinding time was investigated partially for improving the recovery of difficult-to-float coal particles [10], the effect of grinding time on lignite particles for its effects on morphology in an isolated manner has never been studied. Furthermore, the selection of grinding conditions plays a vital role in the size and morphology of the lignite particles, which in turn determine the possible particle-particle or particle–bubble interactions that would be effective in the success of future processes for the beneficiation of fine-sized lignite.

Thus, it is a known fact that particle shape and morphology play a vital role in enrichment processes where physicochemical surface properties are important. In recent years, there have been some studies in the literature on the optimization of grinding conditions to achieve finer particle liberation sizes [11]. However, in these studies, the relation between the grinding parameters and particle size was partially examined, and detailed studies should be conducted. It has also been emphasized in some studies that the morphological characteristics of the particles are very important, in addition to the particle size in the enrichment processes to be carried out after the grinding processes [12,13]. Due to variations in particle–bubble and particle–particle interactions in different processes, the determination of morphology becomes a factor for the decision of grinding conditions along with their role in particle size [14,15]. In this context, the particle morphology is explained under two main headings: "Shape Factor" and "Roughness" [16]. In the literature, many studies examine the effects of different methods, such as treatment with acids [15], sandblasting [17], and the effects of grinding media [18–20] on particle morphology. However, to our knowledge, only in very few studies have the effects of grinding time and U factor (powder/grinding media) values on these parameters been investigated [14,21].

Considering the lack of knowledge in the literature, in this study, the effects of grinding parameters such as the grinding material (i.e., ball)/grinding media ratio (U) and the grinding time on the morphology of lignite particles were investigated.

2. Materials and Methods

2.1. Preparation and Characterization of the Coal Sample

The lignite coal sample used in this study was obtained from the Adana region in Turkey. The lignite samples used in the experiments were prepared by following a similar methodology to those previously reported in the literature [22,23]. Ash analysis of the representative sample was carried out following the procedures defined in ASTM 2012, and the ash content value was found to be 20.36% on an original basis.

In the experimental studies, the particle size of the lignite coal sample was first reduced to −3.36 mm by using a laboratory-type single-toggle jaw crusher. Then, the crushed samples were sieved through a 1 mm aperture size sieve, labeled, and put in sealed sample bags separately to keep their original moisture content. After these processes, the crushed and screened samples of −3.36 + 1 mm in size were ground using a ball mill at different grinding times. A similar sample preparation procedure was reported in the literature for glass beads [18] and hard coal [14]. In these grinding series, the particle size distribution of ground material was determined with a Retsch AS200 model vibrating screen (using a series of screens with 3.36; 2; 1; 0.5; 0.212; 0.150; 0.106; and 0.075 mm aperture) for 15 min after each grinding time to determine the effect of grinding time on particle size.

2.2. Morphological Analyses

The shape factor analyses of the particles obtained at the end of each milling period were processed using the free software ImageJ® (version 1.53e), at the target fraction of −0.212 + 0.075 mm (Figure 1). During these analyses, the sample was initially separated into three size ranges of −0.212 + 0.150 mm, −0.150 + 0.106 mm, and −0.106 + 0.075 mm to prevent the possible effect of very fine-sized materials on the measurement of roundness value. After image analysis for each size range, the roundness value for −0.212 + 0.075 mm was calculated based on the weight percentages of each size. At least 300 particles were counted for each size range to obtain reliable and representative data for roundness values. The equation used to calculate the high-precision roundness value with the image analysis program is given below.

$$R = 4\pi A/P^2 \tag{1}$$

where A is the area of the particle and P is the perimeter. As roundness increases, the high-precision roundness value approaches one. The reason for using high-precision roundness values can be explained by the variation in particle shape based on very minor differences in their roundness values. In other words, as cited in [6,24], when the particle roundness values were in the range of 0.5–0.65, the particle shape was defined as "very angular", but when they reached 0.65–0.70, the shape was then defined as "angular". Accordingly, to present the morphology of the particles, high-precision measurements are essential for gathering the relationship between different production conditions. It can be clearly understood that even very small differences would result in different shapes. The accuracy of the method for making measurements of quartz was presented in our previous studies, which were used as reference point [21]. Additionally, these characteristics are very important in future processes like flotation due to the interaction degree of particles and bubbles, which in turn affects the flotation recovery values.

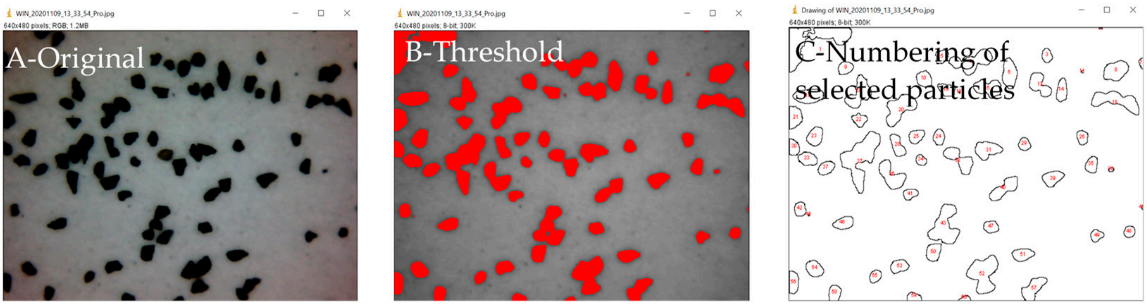

Figure 1. Representative pictures showing the steps of image analysis with ImageJ software. (**A**)—original, (**B**)—threshold analysis, and (**C**)—numbering of selected particles.

It is worth noting that although many shape parameters, such as aspect ratio, circularity, chunkiness, etc., are reported in the literature [6,25], they are also effective in interactions of particles or particle–bubble interactions during their flotation. Roundness and roughness parameters generally dominate the contribution of morphology in terms of these interactions [13–15]. Therefore, among others, based on its contribution rate, the roundness parameter was selected to determine the effect of different grinding conditions on the morphology of ground particles.

As shown in Figure 1, a similar methodology presented by Guven et al. [14] was followed for image analysis. In other words, after taking the image with a definite magnification with a microscope, the particles within the defined particle size range were labeled in red by using the threshold method, and then the shape factor of each particle was determined after these analyses. If the roundness of the particle was decreasing, the high precision roundness value approached zero.

For roughness measurement, the Atomic Force Microscope (NX10) (Park System Company, Suwon, Korea) located in Çukurova University Central Laboratory was used. A similar procedure reported by Guven et al. [14] was used for the roughness analysis. Accordingly, the roughness values of the coal particles were determined using values such as average roughness (Ra) in the frame of a 10 × 10 μm scanning area in tapping mode. In addition, the scan rate was used as 1 Hz during all measurements. In these studies, images were taken along the above-mentioned area by adjusting the cantilever to an appropriate distance from the glass surface on which the coal particles were placed. Ra values were used in the presentation of the roughness value, which was obtained from these images.

2.3. Grinding Processes

From an industrial point of view, there are many studies in the literature on the type and conditions of the grinding process that have a significant effect on particle morphology [8,9,11,14,15,19,20,26]. Parameters such as roundness, angularity, and flatness, which are classified as shape factors under the main heading of particle morphology, are evaluated as two-dimensional, while roughness should be evaluated as a three-dimensional parameter [27].

Within the scope of this study, the changes in the "U" parameter (the ratio of the amount of ground material to the grinding media) in the grinding experiments performed with a laboratory-scale ball mill were investigated for the grinding process of lignite samples. The following equations were used in the calculation of the "U" parameter, and the values of these parameters are shown in Table 1.

$$\text{J} = (\text{Average Ball Weight}/\text{Ball Density})/(\text{Mill Volume}) \times 1/0.6 \tag{2}$$

$$\text{Fc} = (\text{Material Weight}/(\text{Bulk density of the material}))/\text{Mill Volume} \tag{3}$$

$$\text{U} = \text{Fc}/(0.4 \times \text{J}) \tag{4}$$

Table 1. Grinding parameters determined for the particle size of −3.36 + 1 mm.

Parameters	Units	Values		
Mill volume	cm^3	4832	4832	4832
Ball weight	g	5227	5227	5227
Ball apparent density	g/cm^3	4.01	4.01	4.01
Ball diameter	cm	3.00	3.00	3.00
Bulk density of the material	g/cm^3	0.84	0.84	0.84
Ball charge rate (j)	-	0.45	0.45	0.45
U	-	0.34	0.50	0.67
Material charge rate (Fc)	-	0.06	0.09	0.12
Material weight	g	248	365	489

J is the ratio of ball volume to mill volume, and Fc is the ratio of material volume to mill volume. The U parameter is the filling-in-the-gaps fraction given as the ratio of the Fc and J parameters. To determine the maximum ball volume in the mill, parameter J is divided by the constant 0.6, which can be explained by the volume percentage of grinding media in the mill. Therefore, in the calculation of the U parameter, the J parameter is multiplied by the constant 0.4 to indicate the percentage of material volume in the mill (the rest of the volume in the mill besides grinding media). As mentioned in Section 2, the lignite sample with a particle size range of −3.36 + 1 mm was used for grinding tests. To prevent excessive grinding of the materials, the grinding tests were carried out at the same "U" values, and the changes in the shape factor were evaluated as a function of the different grinding times (1, 2, 4, 8, and 16 min). It is worth noting that the speed of the mill is another important parameter to be considered for the grinding processes and affects not only particle size but also the shape of the particles [28]. The diameter of the laboratory-type ball mill was 19 cm, and using that value the critical speed of the mill was calculated using Equation (5).

$$N_c = 420/\sqrt{(D)} \tag{5}$$

N_c is the critical speed of the mill (rpm), and D is the diameter of the mill (cm). All the grinding tests were performed at 75% of the critical speed of the mill, at around 72.3 rpm.

After each grinding period, the ground material was dry-sieved by using 2, 1, 0.5, 0.212, 0.150, 0.106, and 0.074 mm sieves.

3. Results and Discussion

3.1. Size Distribution Analyses

The size distribution graphs of the samples obtained as a result of grinding operations performed at different grinding times under a constant U value of 0.5 are shown in Figure 2a. The effect of "U" values at a constant grinding time of 16 min is shown in Figure 2b.

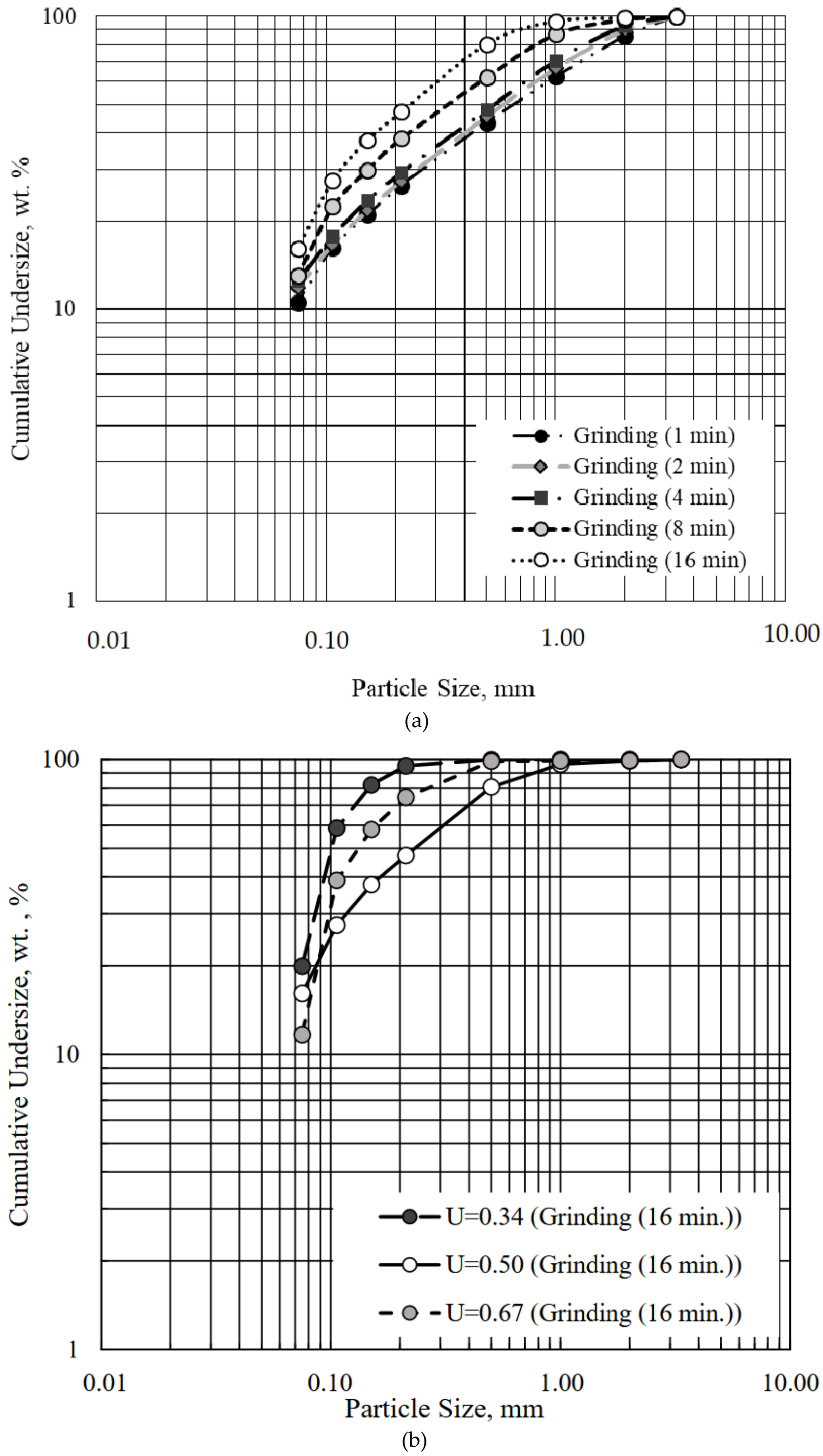

Figure 2. (**a**) Cumulative undersize values of lignite sample obtained from different grinding times (under constant U value as 0.5). (**b**) Cumulative undersize values of lignite sample obtained under different "U" values (under constant grinding time (16 min).

As can be seen in Figure 2a, the d_{80} size decreased to 0.5 mm in the lignite sample after 16 min of grinding, while it decreased to about 0.75 mm at the end of 8 min of grinding, and to about 1.1 mm at less than 8 min of grinding time. This causes an increase in the fraction of −0.212 + 0.075 mm as the target size and, at the same time, an increase of material in the fine fractions.

However, in this study, laboratory-type standard sieves were used instead of measuring with the laser diffraction and sedimentation methods mentioned above, since the target size was −0.212 + 0.075 mm when determining the particle size distribution.

3.2. Shape Factor Analyses

The shape factor analyses of the coal samples obtained with different "U" values and grinding times were performed using ImageJ® software,(version 1.53e) as stated in Section 2.

To determine the changes caused by grinding time in shape factors as well as the size distribution of the coal samples, grinding tests were carried out at the same U value of 0.5 with different grinding times (1, 2, 4, 8, and 16 min). Then, grinding tests were carried out at the same grinding time of 16 min with different U values (0.67 and 0.34). Accordingly, the effects of the conditions on the morphological features of the samples were investigated in detail. The size range and the number of measurements were previously presented in Section 2.

The roundness values obtained depending on the grinding time of the sample are shown in Figure 3, and the roundness values obtained depending on the U parameter are shown in Figure 4. As can be seen from the values shown in the figures, the relative decreases in the particle size of the material due to the increase in grinding time, and the morphological structure of the material, which was originally amorphous, led to a more rounded material. Therefore, it has been shown that the increase in fine size amount of the material affects the roundness of the material increasingly.

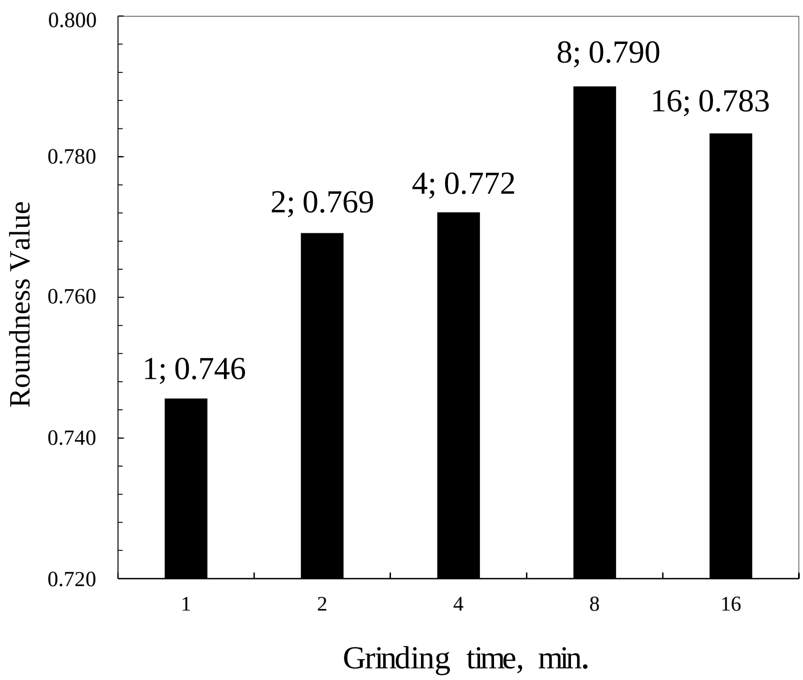

Figure 3. Variations in roundness values of particles as a function of grinding time (grinding time and roundness values are shown above each histogram).

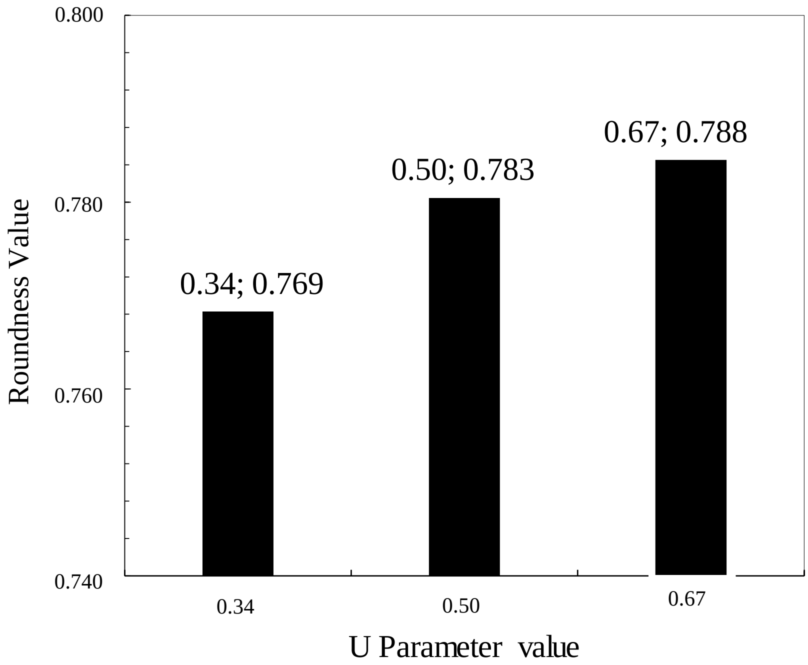

Figure 4. Variations in the roundness values of the particles as a function of the U parameter (U parameter value and roundness values are shown above each histogram).

3.3. Roughness Analysis

The roughness values of the samples are shown in Figure 5. As can be understood from the results, the roughness values of the coal samples, which were examined in terms of roundness value in the previous section, increased depending on the grinding time. As can be seen from the AFM images, the roughness value of the material was measured as 60.883 nm after 2 min of grinding, while this value increased to 90.311 nm after 4 min of grinding and to 107.9 nm after 16 min of grinding.

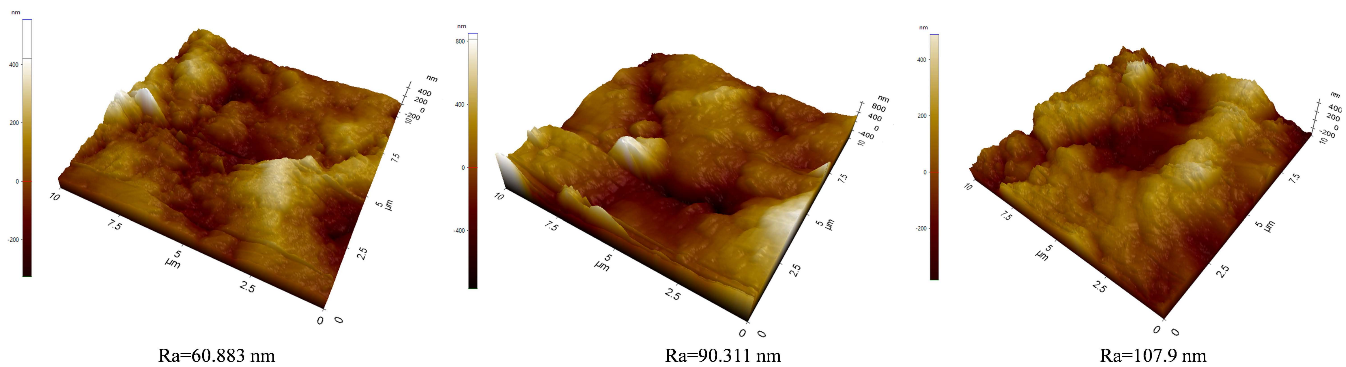

Figure 5. Effect of grinding time on the roughness of lignite coal (from left to right: 2 min, 4 min, and 16 min of grinding time).

4. Discussion

As mentioned in previous sections, the optimization of grinding conditions keeps its importance since finer-sized particles are required to provide the liberation and suitable methodology needed for their enrichment. In the literature, studies on the effects of grinding conditions on size distribution generally include evaluations according to the type of grinding media (ball, rod, or autogenous). A recent study [29] determined the effects on the size distribution of the quartz sample under different grinding conditions using two different methods, i.e., laser diffraction and sedimentation methods. In the study, while the d_{50} size of the material as a result of grinding with the rod mill was obtained as

0.036 mm in the measurements made with the laser, it was found to be 0.040 mm by using the sedimentation method. Although very high differences were not obtained between these results, this reveals the importance of the method used in determining the particle size distribution. In another recent study on the effect of grinding type [28], it was found that while the roundest barite particles were obtained by using an autogenous grinding system, more angular particles can be obtained by rod and ball milling, respectively. From that point of view, not only time but also grinding type should be considered for the design of grinding plants. In another study investigating the effect of ball and gyro milling on the shape of hard coal and lignite [28], the results indicated that the particles (regardless of whether hard coal or lignite) ground by ball milling became more elongated or needle-like while using gyro milling produced rounded or plate-shaped particles with a high circularity degree. The authors explained these findings by the active major breakage mechanisms during grinding processes: impact, chipping, and abrasion. In other words, particle size and shape change depending on the most active mechanisms during grinding. Similarly, in our study, although the same grinding type was used during the grinding tests, the grinding time dominated the possible particle size distribution and morphology of the particles. At that point, the authors suggested that while the gyro mill may produce rounder particles, the ball mill will produce angular particles.

In the experimental study carried out by [30] with three different pulverized coal samples, the size distribution curves determined with standard mesh sieves were compared with the Rosin–Rammler and Gates–Gaudin–Schuman distributions, and it was found that the values obtained were closer to the Rosin–Rammler curve. Another point emphasized by the study is that coal combustion efficiency is affected depending on the size distribution of the coals. Moreover, it is stated that it is possible to determine how much material will remain under which fraction from the distribution curves obtained by pilot-scale tests carried out before plant-scale production.

In sum, many parameters such as the amount of feed material, charge type, time, grinding unit type, etc., should be considered for making the right selection of grinding unit. Accordingly, the characteristics of the ground product, like size range, and morphology, may differ based on these selections. Thus, the results presented in this study provide a piece of information on the grinding characteristics of lignite coal and the role of grinding time on size and morphology.

5. Conclusions

In this study, changes in the morphological properties of the lignite sample ground under different conditions were determined in terms of "roundness" and "roughness" values.

- In the grinding tests performed depending on grinding time, it was determined that the roundness value of the lignite sample increased from 0.746 to 0.790 after 8 min of grinding while the d_{80} sizes of the ground products decreased from 1.6 mm to 0.85 mm. This result shows that grinding time not only plays a significant role in the size distribution of the lignite particles but also in the angular structural features that become round under the influence of the grinding media.
- In the tests performed with different U parameters in the same grinding time (16 min), it was determined that the roundness value of the lignite samples increased from 0.769 to 0.788 when the values of U parameters were 0.34 and 0.67, respectively. These results can be attributed to the increased amount of ground material.
- In the roughness values of particles determined with AFM as a function of grinding time, it was found that the material became rougher independently of the particles' roundness structure. It was found that while the average roughness value of the lignite sample was 60.883 nm for 2 min of grinding time, it increased to 107.9 nm for 16 min of grinding time.
- As a result of this experimental study, it was found that the effects of grinding conditions on the morphology of other minerals should also be evaluated for lignite, and

the morphological properties should also be considered for adjusting the grinding conditions to obtain a suitable size fraction for further processes.

Author Contributions: Conceptualization, O.G.; methodology, O.G.; software, O.G.; formal analysis, O.G., A.E. and B.K.; investigation, O.G. and A.E.; writing—original draft preparation, O.G. and A.E.; writing—review and editing, O.G. and A.E. All authors have read and agreed to the published version of the manuscript.

Funding: This research was supported by Adana Alparslan Türkeş Science and Technology University Science Research Coordination Unit (Project Number: 20103003).

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

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