Dry Grinding of Bentonite by Stirred Media Mill

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Abstract

In this study, a pin-type vertical stirred media mill was used to perform the dry grinding of bentonite. Grinding time and stirring speed were investigated for effect on the particle size distribution and energy consumption (E_c), while ball charge and bentonite charge were kept constant as 60% and 40%, respectively. It was observed that raw bentonites could be ultra-fine dry ground using vertical stirred media mills for the potential dry uses of bentonites. The experimental results revealed that a ground bentonite product having a d_{80} value of about 10 μm was obtained with 128.1 kWh/ton energy consumption. Optimum operational parameters were selected as 60% ball charge, 40% bentonite charge, 600 rpm stirring speed, and 20 min grinding time.

Keywords: Bentonite, Montmorillonite, Grinding, Stirred media mill.

Bentonitin Karıştırılmalı Ortam Değirmeni ile Kuru Öğütülmesi

Öz

Bu çalışmada bentonitin kuru öğütülmesi için pim tipi dikey karıştırma ortamı değirmeni kullanıldı. Tane boyutu dağılımına ve enerji tüketimine (E_c) etkisi için öğütme süresi ve karıştırma hızı araştırılırken bilye ve bentonit şarj oranı sırasıyla %60 ve %40 olarak sabit tutuldu. Bentonitlerin potansiyel kuru kullanımları için ham bentonitlerin dikey karıştırılmalı öğütücüler kullanılarak ultra ince kuru öğütülebildiği gözlemlendi. Deneysel sonuçlar, 128.1 kWh/ton enerji tüketimi ile yaklaşık 10 μm d_{80} değerine sahip öğütülmüş bentonit ürününün elde edildiğini ortaya koydu. Optimum çalışma parametreleri %60 bilye şarjı, %40 bentonit şarjı, 600 rpm karıştırma hızı ve 20 dk öğütme süresi olarak seçildi.

Anahtar Kelimeler: : Bentonit, Montmorillonit, Öğütme, Karıştırılmalı ortam değirmeni.

1. Introduction

Bentonite, a type of clay ore, mainly contains smectite minerals (montmorillonite, saponite, nontronite, hectorite). Smectite minerals occur by the decomposition of the volcanic tuffs, ashes, and lavas. Montmorillonite, the most abundant smectite mineral in bentonite ores, is aqueous aluminum silicate and has a layered crystalline structure with the general chemical formula of $(\text{Na}, \text{Ca})_{0.33}(\text{Al}, \text{Mg})_2(\text{Si}_4\text{O}_{10})(\text{OH})_2 \cdot n\text{H}_2\text{O}$. Bentonites have superior properties such as high cation exchange capacity, large surface area, high swelling capacity, plasticity, good mechanical properties, and chemical stability [1–5]. Therefore, bentonites are used in various applications such as adsorption, pharmaceutical and cosmetic industry, drilling mud, food, paper, paint, and ceramics [1, 6–8].

Recently, great attention has been paid to the fine grinding of clay minerals by planetary or stirred ball mills. Because the physical properties of clay minerals (especially structural and textural) are developed for new application areas by this way [9]. Colloidal dispersions for various applications, food protective materials, drug delivery systems, paint, paper, cosmetic, and dry bentonite beneficiation can be shown for the new application uses of fine ground bentonites. There are many studies performed to investigate the operating parameters that affect the grinding performance of stirred media mills [10–14]. However, to our best knowledge, there are few studies on dry grinding of the bentonite by vertical stirred media mill. Dry grinding is a necessary application in some areas, especially in dry beneficiation of clay minerals by sieving, aero cyclones, and dynamic separators.

This study focused on the dry grinding of sodium bentonite using a pin-type vertical stirred media mill. Operational parameters such as grinding time and stirring speed were investigated. Particle size distribution of the ground material and energy consumption of grinding application analyzes were also performed to determine optimum grinding conditions. A product having a d_{80} value of about 10 μm was aimed to obtain for potential uses in the cosmetic, chemistry, and pharmaceutical industries.

2. Material and Method

2.1. Materials

Bentonite sample was obtained from Karben Bentonite Cooperation located in Reşadiye Tokat, Turkey. Yttrium stabilized zirconium oxide balls (3 mm and 5 mm) were used as grinding media.

2.2. Methods

2.2.1. Characterization Studies

The bentonite sample was dried at 55°C for 24 h and then ground using a laboratory scaled ring mill before all characterization analysis. The chemical composition of the sample was analyzed by Rigaku ZSX Primus II XRF (x-ray fluorescence) device. X-ray diffraction (XRD) pattern was scanned with Cu-K α radiation (40 kV, λ : 1.54184 Å) using a Shimadzu XRD-6000 device to determine mineralogical components. The sample was scanned at the angular range of 2–40° with 0.02°/step scan speed. LEO 1430 VP scanning electron microscope (SEM) was used to examine the morphology of the bentonite sample. Particle size distributions were determined by the Malvern Master-sizer 2000 instrument using dry analysis apparatus.

2.2.2. Grinding Studies

Grinding studies were performed using a laboratory scaled vertical stirred media mill (Figure 1) with a pin-type stirrer. The effective volume of the mill chamber was 1.0 L. The grinding chamber was equipped with a water jacket for cooling. Yttrium stabilized zirconium oxide balls (3 mm: 598 g, 5 mm: 1967 g, total volume: 530 mL) were used as grinding media. The volume of the grinding media and the bentonite sample was kept constant as 530 mL (60%) and 350 mL (40%), respectively, for all grinding experiments.

Figure 1. Pin-type vertical stirred media mill used for the grinding experiments.

The grinding experiment was performed as a batch process. First, the raw bentonite sample dried at 50°C for 24 h and then crushed under 1 mm using a roll mill. 350 mL of pre-ground bentonite sample (-300 μ m) and 530 mL grinding media were placed into the mill chamber. The circulation of the cooling water and then stirring of the pin type stirrer was started. After a certain time, stirring was stopped, and the ground sample was separated from the balls using a 1 mm sieve. The energy consumption of each grinding experiment was measured using an electricity meter connected to the mill. Operational parameters used in the grinding tests are summarized in Table 1.

Table 1. Operational parameters of the grinding experiments.

Experiment No	Stirring speed (rpm)	Grinding time (min)	Ball charge (%)	Material charge (%)
1	500	5	60	40
2	500	10		
3	500	20		
4	500	30		
5	500	40		
6	600	20		
7	700	20		
8	800	20		

3. Results and Discussion

3.1. Characterization of the bentonite

The chemical analysis (XRF) results (Table 2) showed that the bentonite sample had high percentages of silicon and aluminum oxides (81.3 wt.% in total), indicating the probability of a clay mineral. $(\text{Na}_2\text{O}+\text{K}_2\text{O})/(\text{CaO}+\text{MgO})$ ratio was determined to be 0.69. A value high than 0.33 is attributed to the Na-smectites [2].

Smectite minerals (montmorillonite, etc.) usually exhibit a morphology that resembles a honeycomb, cornflake, leafy, and rosette-like appearance [2, 15]. SEM micrograph (Figure 2) revealed that the bentonite sample used in this study consisted of smooth-surfaced grain aggregates and had a leaf layer morphology with curved edges.

Table 2. Chemical composition of the bentonite sample.

Component	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	Na ₂ O	CaO	K ₂ O	LOI
Content, wt. %	62.23	19.06	3.63	2.70	2.61	2.05	0.65	6.30

LOI: loss on ignition.

Figure 2. SEM micrograph of the bentonite sample.

The XRD pattern of the bentonite sample is given in Figure 3. The sample mostly consisted of montmorillonite minerals and non-clay minerals such as feldspar, calcite, opal (C-T), quartz, mica, and clinoptilolite (zeolite). Characteristic reflection at 2θ : 7.07° with the d_{001} basal spacing of $12.4 \text{ \AA}$ confirmed that the bentonite sample contains a high amount of Na^+ -rich montmorillonite [16] which was in good agreement with XRF analysis (Table 2). Furthermore, the d_{060} value ($1.5 \text{ \AA}$) showed that the sample was composed of dioctahedral montmorillonites [17].

Figure 3. XRD pattern of the bentonite sample.

3.2. Grinding of the bentonite

3.2.1 Effect of the Grinding Time

Time-dependent grinding experiments were performed at a stirring speed of 500 rpm. The effects of grinding times of 5, 10, 20, 30, and 40 minutes on the particle size distribution and energy consumption (E_c) were investigated. The effect of the grinding time on the particle size distribution of the bentonite and E_c was given in Figure 4 and Table 2. As clearly seen in Figure 4, grinding time had a significant effect on the particle size and so energy consumption. An increase in the grinding time decreased the product size and simultaneously increased the E_c . It has been proven once again that the energy consumption (E_c) in the mill has a linear relationship with the grinding time. When the mill reaches meta-steady state dynamics, the power consumption of the mill becomes constant [18]. Similar results were also reported by other studies [14, 19-21].

Table 2. The d_{50} and d_{90} sizes of the products change depending on the grinding time.

	5 min	10 min	20 min	30 min	40 min
d_{50}	25.2	18.5	6.1	4.7	3.9
d_{80}	69.0	69.0	22.0	15.2	10.0

Figure 4. Effect of the grinding time on the particle size distribution and the energy consumption (E_c).

Energy consumption in the mill as a function of the particle size (d_{80}) of the ground bentonite products (Figure 4) revealed that E_c increased as particle size was more reduced by higher grinding times. The results displayed that increase in the grinding time from 5 min to 40 min decreased the product size (d_{80}) from 69.0 to 10.0 μm , whereas E_c was increased significantly from 33.7 to 209 kWh/t. The optimum grinding time was selected to be 20 min for the ongoing grinding experiment as a function of stirring speed.

3.2.2 Effect of the Stirring Speed

The stirring speed creates a high energy strength environment in the grinding chamber, increasing the probability of the grinding media impact and attrition of the particles. As the stirring speed increases, the stress intensity inside the mill chamber also increases, which seriously affects the interaction between the grinding media and the material to be ground [18, 22-23]. Grinding experiments were performed as a function of stirring speed (400, 500, 600, 700, 800 rpm) while grinding time was kept constant as 30 min. The effect of the stirring speed on the particle size distribution of the bentonite and E_c was given in Figure 5. The results displayed that increase in the stirring speed from 500 rpm to 800 rpm decreased the product size (d_{80}) from 22.0 to 8.0 μm , whereas E_c was increased significantly from 107.9 to 168.5 kWh/t. Increasing the mixing speed increases the stress intensities and eventually grinding speed due to the high

probability of particle collisions with the grinding media. Furthermore, as the mixing speed increases, the grinding media reaches higher kinetic energy, which is transferred to the particles, making breakage easier.

Figure 5. Effect of the grinding time on the particle size distribution and the energy consumption (E_c).

The increase in grinding performance with the increase in stirring speed is a condition that is effective up to a certain stirring speed. Even if the mixing speed increases after a certain speed, the grinding performance decreases as the grain size reduction rate will be meager. Figure 5 and Table 3 revealed that the grinding performance was decreased when the stirring speed was increased to 800 rpm. Therefore, optimum operational parameters were selected as 60% ball charge, 40% bentonite charge, 600 rpm stirring speed, and 20 min grinding time to obtain a product having a d_{80} value of about 10 μm .

Table 3. The d_{50} and d_{90} sizes of the products change depending on the stirring speed.

	500 rpm	600 rpm	700 rpm	800 rpm
d_{50}	6.1	3.6	3.5	3.3
d_{80}	22.0	11.0	7.5	8.0

4. Conclusion

Dry grinding experiments of the raw bentonite sample using a pin-type vertical stirred media mill were successfully performed. Characterization studies revealed that the bentonite sample mostly consists of Na^+ rich montmorillonite. Grinding time and stirring speed were investigated as two operational parameters while material and ball charge were kept constant. It was concluded that bentonites could be dry ground to ultrafine particle sizes using stirred media mills for the potential dry uses. A ground bentonite product having a d_{80} value of about 10 μm was obtained with 128.1 kWh/ton energy consumption using selected operational parameters, which are 60% ball charge, 40% bentonite charge, 600 rpm stirring speed, and 20 min grinding time.

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References

- [1] H. Çiftçi, "An introduction to montmorillonite purification", In: Montmorillonite clay, Eds. F. Uddin, London, IntechOpen, pp. 1-7, 2021.
- [2] H. Çiftçi, B. Ersoy, and A. Evcin, "Purification of Turkish Bentonites and Investigation of the Contact Angle, Surface Free Energy and Zeta Potential Profiles of Organo-Bentonites as a Function of CTAB Concentration," *Clays and Clay Minerals*, vol. 68 no. 3, pp. 250-261, 2020.
- [3] J. H. Yang, J. H. Lee, H. J. Ryu, A. A. Elzatahry, Z. A. Alothman, and J. H. Choy, "Drug-clay nanohybrids as sustained delivery systems," *Applied Clay Science*, vol. 130, pp. 20-32, 2016.
- [4] J. Nones, H. G. Riella, A. G. Trentin, and J. Nones, "Effects of bentonite on different cell types: A brief review," *Applied Clay Science*, vol. 105-106, pp. 225-230, 2015.
- [5] S. Jain, and M. Datta, "Oral extended release of dexamethasone: Montmorillonite-PLGA nanocomposites as a delivery vehicle," *Applied Clay Science*, vol. 104, pp. 182-188, 2015.
- [6] S. Jayrajsinh, G. Shankar, M. Pharm, Y. K. Agrawal, and L. Bakre, "Montmorillonite nanoclay as a multifaceted drug-delivery carrier: A review," *Journal of Drug Delivery Science and Technology*, vol. 39, pp. 200-209, 2017.
- [7] S. E. Elmore, N. Mitchell, T. Mays, K. Brown, A. Marroquin-Cardona, A. Romoser, and T. D. Phillips, "Common African cooking processes do not affect the aflatoxin binding efficacy of refined calcium montmorillonite clay," *Food control*, vol. 37, pp. 27-32, 2014.
- [8] V. Summa, and F. Tateto, "Clay minerals as adsorbents of aflatoxin M1 from contaminated milk and effects on milk quality," *Applied Clay Science*, vol. 88, pp. 92-99, 2014.
- [9] W.K. Mekhamer, "Stability changes of Saudi bentonite suspension due to mechanical grinding", *Journal of Saudi Chemical Society*, vol. 15, pp. 361-366, 2011.
- [10] O. A. Orumwense, and E. Forssberg, "Superfine and ultrafine grinding, A literature survey, *J. Mineral Processing and Extractive Metall Rev*, vol. 11, pp. 107-127, 1992.
- [11] J. Lichter, and G. Davey, "Selection and sizing of ultrafine and stirred grinding mills," *Advances in Comminution*. Colorado, Society for Mining, Metallurgy and Exploration, 2006.
- [12] K. Ohenoja, M. Illikainen, and J. Niinimäki, "Effect of operational parameters and stress energies on the particle size distribution of TiO₂ pigment in stirred media milling," *Powder Technology*, vol. 234, pp. 91-96, 2013.
- [13] A. Jankovic, "Variables affecting the fine grinding of minerals using stirred mills," *Minerals Engineering*, vol. 16, pp. 337-345, 2003.
- [14] O. Y. Toraman, and D. Katircioglu, "Study on the effect of process parameters in stirred ball mill," *Advanced Powder Technology*, vol. 22, pp. 26-30, 2011.
- [15] W. D. Keller, and R. C. Reynolds, A. Inoue, "Morphology of clay minerals in the smectite-to-illite conversion series by scanning electron microscopy," *Clays and Clay Minerals*, vol. 34, pp. 187-197, 1986.
- [16] G. Brown, and G. W. Brindley, "X-ray diffraction procedures for clay mineral identification," in: G. W. Brindley, G. Brown (Eds.), *Crystal Structures of Clay Minerals and their X-ray Identification*, Monograph 5 (pp. 305-360), London, Mineralogical Society, 1980.
- [17] R. E. Grim, "Clay Mineralogy. International series in the earth and planetary sciences," pp. 596, New York, Mc Graw-Hill Book Co. Inc., 1968.
- [18] T. Santosh, K. Rahul Soni, C. Eswaraiah, D. S. Rao, and R. Venugopal, "Optimization of stirred mill parameters for fine grinding of PGE bearing chromite ore," *Particulate Science and Technology*, vol. 39, no. 6, Jul., pp. 663-675, 2020. DOI: 10.1080/02726351.2020.1795016
- [19] C. Eswaraiah, N. Venkat, B. K. Mishra, and R. Holmes, "A comparative study on a vertical stirred mill agitator design for fine grinding." *Separation Science and Technology*, vol. 50, pp. 39-48, 2015. doi:10.1080/01496395.2015.1065888.

- [20] O. Y. Toraman, "Dry fine grinding of calcite powder by stirred mill," *Particulate Science and Technology*, vol. 31 no. 3, pp. 205-209, 2013. doi:10.1080/02726351.2012.694135.
- [21] H. Hacıfazlıoğlu, and A.V. Korkmaz, "Performance comparison of stirred media mill and ball (BOND) mill in bauxite grinding," *Particulate Science and Technology*, vol. 38, no. 4, pp. 404-408, 2020. DOI: 10.1080/02726351.2018.1547342
- [22] O. Altun, H. Benzer, and U. Enderle, "Effects of operating parameters on the efficiency of dry stirred milling," *Minerals Engineering*, vol. 43-44, pp. 58-66, 2013. doi:10.1016/j.mineng.2012.08.003
- [23] M. Hasan, S. Palaniandy, M. Hilden, and M. Powell, "Calculating breakage parameters of a batch vertical stirred mill," *Minerals Engineering*, vol. 111, pp. 229-237, 2017. doi:10.1016/j.mineng.2017.06.024.