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Enhancing Iron Ore Grindability through Hybrid Thermal-Mechanical Pretreatment

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Abstract: Grinding is an important process of ore beneficiation that consumes a significant amount of energy. Pretreating ore before grinding has been proposed to improve ore grindability, reduce comminution energy, and enhance downstream operations. This paper investigates hybrid thermal mechanical pretreatment to improve iron ore grinding behavior. Thermal pretreatment was performed using conventional and microwave approaches, while mechanical pretreatment was conducted with a pressure device using a piston die. Results indicate that conventional (heating rate: 10 °C; maximum temperature: 400 °C), microwave (2.45 GHz, 1.7 kW, 60 s), and mechanical (14.86 MPa, zero delay time) pretreatments improved the studied iron ore grindability by 4.6, 19.8, and 15.4%, respectively. Meanwhile, conventional-mechanical and microwave-mechanical pretreatments enhanced the studied iron ore grindability by 19.2% and 22.6%, respectively. These results suggest that stand-alone mechanical pretreatment or microwave pretreatment may be more beneficial in improving the grinding behavior of the studied fine-grain iron ore sample. The results of the mechanical pretreatment obtained in this study may be used in a simulation of the HPGR system for grinding operations of similar iron ore

Keywords: microwave heating; thermal treatment; comminution energy; ore grinding; work index

Citation: Adewuyi, S.O.; Ahmed, H.A.M.; Anani, A.; Saeed, A.; Ahmed, H.M.; Alwafi, R.; Luxbacher, K. Enhancing Iron Ore Grindability through Hybrid Thermal-Mechanical Pretreatment. *Minerals* **2024**, *14*, 1027. https://doi.org/10.3390/min14101027

Academic Editors: Murray M. Bwalya and Ngonidzashe Chimwani

Received: 9 September 2024
Revised: 8 October 2024
Accepted: 12 October 2024
Published: 14 October 2024

1. Introduction

Huge energy consumption for ore grinding [1] and tailing management [2,3] are major challenges in mineral processing. Reducing energy consumption and sustainable tailing management are among the current challenges in the mining industry. Efficient comminution operation improves mineral liberation, leading to improved mineral recovery and low tailing production, making comminution a critical operation in mineral processing [4]. Therefore, efforts to improve comminution operations are receiving significant attention. With ore grades declining globally, demand for metals and industrial minerals increasing, and the transition to industrial 4.0, the energy requirement for comminution processes is increasing, causing greater concern than ever before. Among the mining operations, grinding consumes up to 70% of the total energy consumption [5,6]. Studies have been performed to reduce grinding energy by focusing on factors to improve grinding performance. Among the factors affecting grinding performance are grinding media, machine type, operating conditions, and ore characteristics [7–9]. Attempts have been made to improve ore grinding operations by optimizing the ore-to-media ratio, mill-length-to-diameter ratio, mill speed, media size distribution, and varying grinding balls such as mild steel, stainless steel, and nano-ceramic [5]. Furthermore, efforts have been expended on ore

comminution machine design, resulting in the production of the high-pressure grinding roll (HPGR), which can minimize energy loss as noise and heat—leading to improved ore grinding and reduction in comminution energy [10]. However, HPGR produces a coarser particle size distribution due to the edge effect [11]. Since the grindability of ore is a material's characteristic, researchers proposed that ores may be pretreated to improve their physical properties, which may enhance grinding behavior and lead to significant energy savings [5]. Different ore pretreatment methods have been explicitly presented in the literature [5]. Thermal pretreatment is among the focused techniques that may be performed by conventional (via furnace) or microwave (MW). Both heating approaches have been employed for several minerals with varying degrees of heating responses and induced cracks on the tested ore/mineral samples [5]. The type of cracks developed by the material samples and the extent of cracks determine the effect of absorbed heat energy on the material's grinding behavior [5]. Ratan et al. [12] discussed that conventional heating of hematite ore to 400 °C followed by water quenching may reduce grinding energy by 45%. Omran et al. [13] investigated the effect of conventional heating on the grinding behavior of phosphorus-oolitic iron ore and results indicated that particles passing a sieve size of 0.125 mm increased from 46.6 to 50.8% after heating to 600 °C for 1 h. Based on the available literature, little is known about the grinding behavior of conventionally heated iron ore, but the approach has been widely studied to convert hematite to magnetite [14]. Most researchers have focused on the grinding behavior of conventionally heated quartzite and calcite [15].

For microwave heating, studies are increasingly focusing on reducing grinding energy requirements in the mining industry through microwave pretreatment of ores. Walkiewicz et al. [6] found that cracks developed on iron ore samples after being subjected to microwave heating, resulting in an improvement in work index up to 23.7%, for hematite obtained from Republic mine, Michigan, USA. Micro-fractures were observed on microwave-treated hematite selected from Orissa, India, that resulted in an improvement in the specific rate of breakage up to 50% [16]. Song et al. [17] reported that intergranular cracks developed on hematite ore after microwave irradiation, which improved hematite liberation from quartz and apatite by up to 30%. Singh et al. [18] pretreated iron ore (containing hematite and jasper) at 900 W for 5 min. Grindability test results indicated that microwave pretreatment decreased d_{80} by 20.83% compared to untreated ore samples [18]. Abdur Rasyid et al. [19] studied the power-saving capability of microwave pretreatment during the crushing operation of kimberlite rock using a single-roll crusher. It was found that specific crushing energy of up to 18% can be saved using microwave pretreatment. Hao et al. [20] studied the microwave-damage mechanism of magnetite ore. Their results showed that increasing microwave power led to deceased ore mechanical properties due to intergranular and intragranular cracks developed after microwave irradiation, leading to improved crushing degree. Omran et al. [13] compared the effect of conventional and microwave pretreatments and found that intergranular fractures developed between hematite and its gangue (fluoroapatite and chamosite) after microwave treatment, while a small proportion of micro-cracks were noticed on the ore's surface after conventional heating. Grindability tests showed that the weight percentage passing sieve size 0.125 mm increased by 13.16% after microwave pretreatment compared to a 4.2% increase for conventional pretreatment [13]. Recently, results of possible energy reduction due to microwave pretreatment of different ores/minerals have been presented in the literature [5]. Findings indicated that despite intensive research on microwave applications for improved ore grinding for better energy utilization in the mining industry, there is little research on iron ore (Table 1).

The piston-die test is usually employed to study particle-bed breakage for understanding interparticle breakage, estimating the energy required for comminution by compressive load, and predicting the breakage behavior of HPGR [21,22]. A comparison of the particle liberation using particle-bed breakage (by piston-die compression), hammer mill, and ball mill has been discussed in the literature [23]. Particle-bed breakage generally enhanced the particle liberation for clinker [24], calcite [25], and quartz [22]. Meanwhile, low-grade

porphyry copper ore at a size fraction below 150 µm exhibited similar particle liberation in hammer mill and piston-die compression [23]. The selected iron ore samples in this study consist of interwoven fine-grain hematite, magnetite, and diopside; these usually require fine grinding, consume a high amount of comminution energy, and cause metallurgical challenges [26]. Pretreating this type of iron ore may improve its grinding behavior. This paper investigates hybrid thermal-mechanical pretreatments to enhance the selected iron ore grinding behavior. Thermal pretreatment was studied using conventional and microwave approaches, while mechanical pretreatment was conducted using a piston-die test. The grinding behavior of the investigated pretreatment approaches was evaluated using a laboratory standard Bond ball mill.

Table 1. Microwave pretreatment of iron ore samples for improved ore grindability (MW frequency: 2450 MHz), temp. = temperature.

Sample Location	Mineral Phases	Size Fraction (mm)	Sample Mass (g)	MW Power (kW)	MW Time (min)	Average MW Temp. (°C)	Improvement in Grindability (%)	Reference
Republic mine, Michigan	Hematite, quartz	−3.35	350	3.0	3.5	840	23.7	[6]
Empire mine, Michigan	Magnetite, quartz	−3.35	350	3.0	3.5	840	21.4	[6]
Orissa, India	Hematite, alumina, silica	−19.05 + 12.7	500	0.9	2.0	148	50	[16]
Hubei province, China	Hematite, quartz, chlorite, apatite	-	50	1.2	5.0	-	-	[17]
Aswan region, Egypt	Hematite, quartz, fluoroapatite, chamosite	-	100	0.9	1.0	546	-	[13]
Joda, Odisha, India	Hematite, jasper	−3.35	50	0.9	5.0	510	20.8	[18]

2. Materials and Methods

2.1. Sample Collection and Preparation

The iron ore sample was collected from the Wadi Sawawin area, Tabuk Province, Saudi Arabia. This sample was considered in this study due to the increasing demand for iron ore around the world and the high level of energy consumption during grinding operations. Also, iron ore is known for its high dielectric and electric properties, which are crucial for investigating rock's response to thermal treatment via microwave or furnace, making it suitable for this study. The as-received samples (20–25 cm) were preliminary crushed by a hand-held hammer and further primary and secondary crushing operations were conducted using a laboratory jaw crusher (BB300 Mangan Retsch, Retsch-Allee Haan, Germany) and a laboratory roll crusher (Sew-Eurodrive GmbH & Co. KG, Bruchsal, Germany), respectively. These operations continued until a 100% passing sieve size of 3.35 mm, required for the standard Bond ball mill grindability (BBMG) test, was achieved (Figure 1). A quartzite sample collected from the Al Masane Al Kobra (AMAK, Nejran, Saudi Arabia) mining company [15] was used in this study for the optimization of mechanical pretreatment. The conning and quartering method (Figure 2) and a mechanical riffle-splitter (KHD Humboldt Wedag AG, Colonia-Allee 3, Cologne, Germany) were employed to obtain representative sub-samples. The same sampling procedure was used for the studied quartzite and iron ore samples. In this approach, the crushed sample was thoroughly mixed and shaped into a cone and then quarterly subdivided till sub-samples of approximately 5 kg each were obtained. One of the sub-samples was subdivided using a mechanical riffle-splitter until a mass equivalent to 700 cm^3 (required as the first feed for the BBMG test) was obtained. The average mass of five repeated experiments for the quartzite sample was 1282.5 g with a standard deviation of 0.2160 [15]. This indicates that the employed sampling

procedure produced representative samples. Specifically, to obtain a sample of 1282.5 g for a BBMG test, a 5 kg sub-sample was thoroughly mixed for homogeneity and divided into four—each with approximately 1250 g, using a mechanical riffle-splitter. One 1250 g sub-sample was split into four with approximately 312.5 g each. One of these sub-samples was also divided into four. One of the obtained sub-samples was then divided into two to get approximately 39 g sub-samples each. A 39 g sub-sample was thoroughly mixed with a 1250 g sub-sample in a pan and formed a cone. The pan was then placed on a digital chemical balance where a spatula was used to reduce the mass by 6.5 g from four-quarter sides of the cone. To perform the particle size analysis of the sub-samples, eight sieves with size range 3150 µm–75 µm were selected [15]. The representative sub-samples obtained using a laboratory mechanical riffle-splitter were dry-screened by a laboratory electric sieve shaker (AS 200, Retsch, Retsch-Allee Haan, Germany) by placing the sample on the top sieve (3150 µm) [15]. The cumulative percentage of weight passings was calculated and recorded based on the weight passing each sieve. Particle size was plotted against the cumulative %weight passing to determine the 80% passing size (F_{80}) required for the grindability tests [15]. The repeated BBMG tests performed by employing the above sub-sampling procedure using a quartzite sample showed variation within ±0.4% [15], which falls within the acceptable industrial standard of ±3.4% [16]. Therefore, the sub-sampling procedure outlined above was applied to the studied iron ore sample. However, the feed mass obtained for the BBMG test of the investigated iron ore was 1710.5 g—indicating a packing density of 2.4436 g/cm^3. To get this mass, a stepwise mechanical riffle-splitter operation produced 1718.5 g, which was thoroughly mixed and shaped like a cone in a sample pan. This mass was then reduced to the required mass (1710.5 g) for the BBMG test, using the same approach as quartzite.

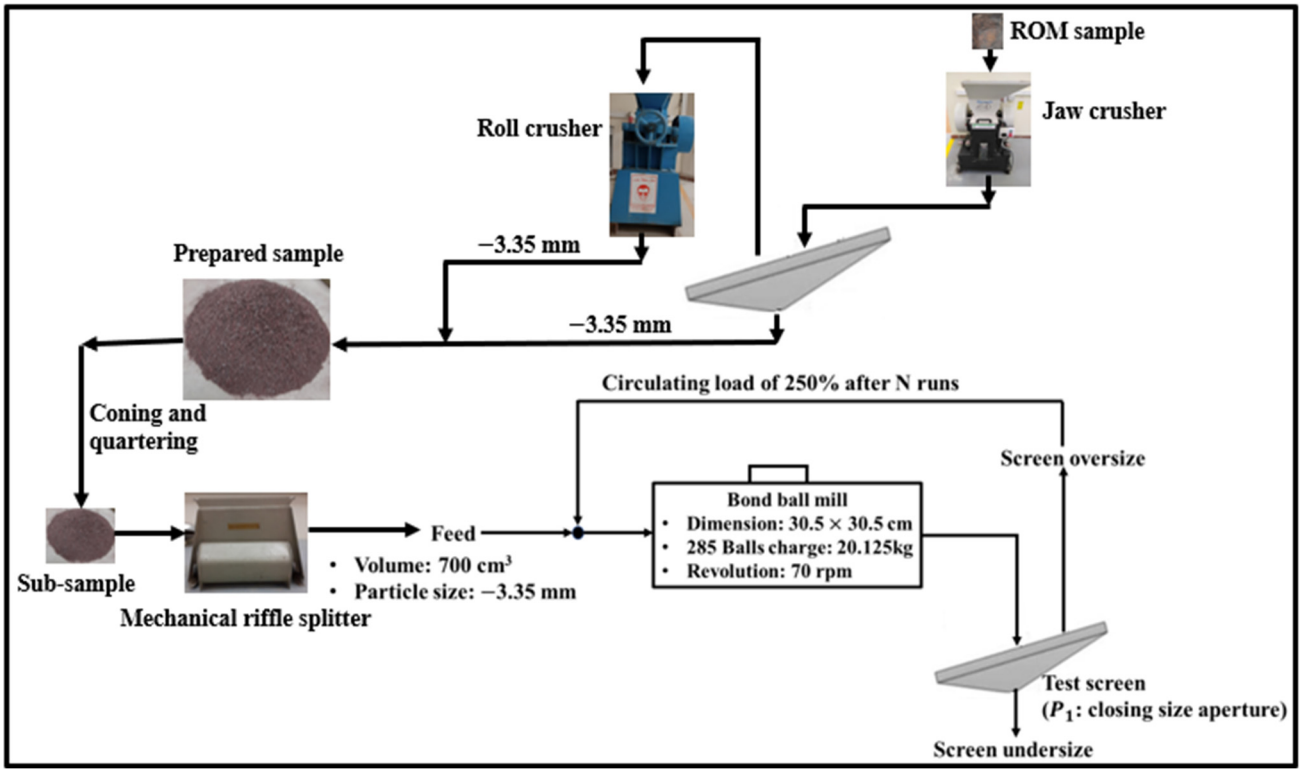


Figure 1. Sample preparation and Bond ball mill grindability (BBMG) test procedure (rpm—revolution per min, ROM—run-of-mine).

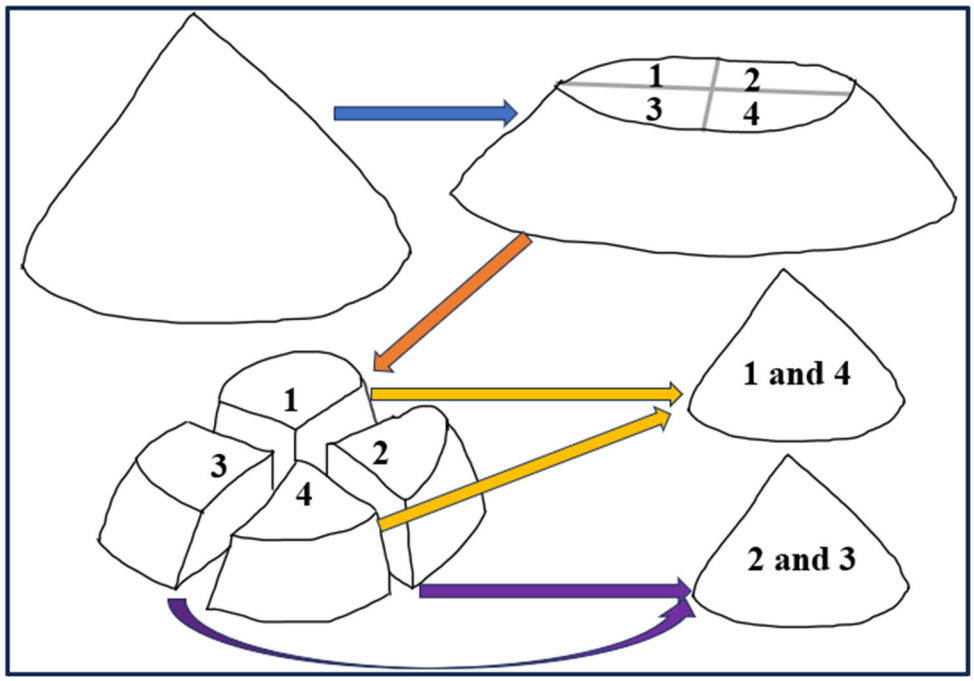


Figure 2. Coning and quartering technique (→: by relying on radial symmetry, a conical heap is divided into four, by cross; →: the sample is divided into four, along the cross; →: opposite quarters (1 and 4) are combined to form a sub-sample, →: opposite quarters (2 and 3) are combined to form a sub-sample).

2.2. Sample Characterizations

To perform chemical, mineralogy and morphology analyses of the studied samples, the laboratory mechanical riffle-splitter was used to appropriately obtain representative sub-samples from the prepared sample (Figure 1). Using a ball mill, the sample was ground to below 75 µm sieve size. The miniature sampling technique, using a spatula usually employed for powder material, was used to obtain sub-samples for chemical, mineralogy, and morphology analysis [15]. Mineralogy analysis was performed using the X-ray Diffraction method (Regaku, Ultima 1V X-ray diffractometer, Tokyo, Japan) as described in the literature [15]. Chemical analysis was also performed using a Fourier-transform infrared (FTIR) spectroscope (Nicolet iS50 FTIR, Thermo Fisher Scientific, Waltham, MA, USA) between 4000–400 cm^{-1} in transmittance mode. Morphology analysis was conducted using a Field Emission Scanning Electron Microscope (FESEM, JSM-7600F, JEOL Ltd., Musashino, Akishima, Tokyo, Japan) [15,27]. The description of FESEM for powder sample (without coating) characterization was adopted as presented in the literature [15,28,29]. The electric and dielectric properties of the studied samples were characterized to understand the selected sample's response to temperature. To do so, a cuboid-shaped iron ore sample was prepared with dimensions of 12, 10, and 2.5 mm. Sandpaper was used to smoothen the surfaces of the prepared sample, and the surfaces were then painted using a conductive platinum paste (Nanoshel LLC, Willmington, DE, USA) to form electrodes [30]. The iron ore cuboid was oven-dried at 150ºC for 2 h. The electrical resistance, capacitance, and tangent loss of the prepared iron ore cuboids were measured and recorded from 300 K to 1100 K using an impedance analyzer (LCR bridge; 0.05% accuracy, 4 Hz–8 MHz, IM3536, Hioki, Nagano, Japan) [30]. A constant voltage mode was employed during this measurement, and temperatures were recorded between 5 kHz–8 MHz. The recorded data (resistance, capacitance, and tangent loss) were transferred to a personal computer via an RS-232C serial port that was connected to the impedance analyzer [30]. The electrical

conductivity (σ, $\Omega^{-1}m^{-1}$), dielectric constant (ε'), and dielectric loss (ε'') were calculated using Equations (1), (2), and (3), respectively.

$$\sigma = \frac{t_s}{A * R} \tag{1}$$

$$\varepsilon' = \frac{Cd}{\varepsilon A} \tag{2}$$

$$\varepsilon'' = \varepsilon' \, tan\delta \tag{3}$$

where R (Ω) is the experimentally measured electrical resistance of the studied sample, A is the cross-sectional area of the sample (m^2), t_s is the thickness of the sample (m), C is the experimentally measured electrical capacitance of the sample, $tan\delta$ represents the measured tangent loss, and ε is the free space permittivity.

2.3. Bond Ball Mill Grindability Test

In the mineral industry, the BBMG test is usually employed to estimate the comminution energy, scale up the comminution operation, and compare the material response to ball milling [15]. The test was proposed by Bond in 1961 [31], and ever since, it has been regarded as the industrial standard for estimating the energy requirement for ore milling using the material's index known as the Bond work index (W_i). The grindability test of the studied sample was performed using a laboratory standard Bond ball mill (395-51, BICO Braun International, Burbank, CA, USA) [32]. The sample preparation and BBMG test procedure is presented in Figure 1. A 5 kg representative sample was obtained using a coning and quartering method, and the particle size analysis was performed, as described in Section 2.2. The interstitial volume equals 700 cm^3 within 285 steel balls of different sizes, as described by BICO Braun International, was determined to establish the required feed for the BBMG test [32]. The obtained feed mass (1710.5 g), equivalent to 700 cm^3, was used to determine the particle pack density of the investigated sample. The 285 steel balls (20,125 g) and 1710.5 g samples were fed into the Bond mill, and the cover was lined with rubber material before closing the feed opening to avoid losing particles during milling. The mill was run at 70 rpm for 100 revolutions, after which the product was discharged into a pan. The steel balls were properly cleaned using a brush and re-fed into the mill for further milling operation. The product was batch dry-screened using a laboratory electric sieve shaker (AS 200, Retsch, Retsch-Allee Haan, Germany) by putting the product on a sieve size 106 µm (test sieve size—closing sieve aperture). The undersized product was used to calculate the next number of revolutions that can produce a 250% recirculating load [15]. The representative sample, equivalent to the mass of the undersized product, was added to the recirculating load for the next milling operation. The number of grams per revolution (G_{bp}) was calculated and recorded. This procedure was repeated until G_{bp} becomes constant or changes trend direction. The Bond Equation (4) was employed to calculate the Bond work index of the studied sample.

$$W_i = \frac{44.5}{{P_1}^{0.23} G^{0.82} \left(\frac{10}{\sqrt{P_{80}}} - \frac{10}{\sqrt{F_{80}}} \right)} \tag{4}$$

where W_i represents Bond work index (kWh/t), P_1 is the closing sieve aperture (mm), G represents the ideal grindability (g/rev), P_{80} is the 80% passing size (µm) of product, and F_{80} is the 80% passing size (µm) of feed.

Comparative Method of Grindability Test

Berry and Bruce proposed a comparative method to determine the work index of test samples due to the large sample and time required for the standard Bond grindability test approach [33]. The method was based on the condition that the reference and test samples consume the same energy (E) when ground under the same grinding conditions (sample

mass, grinding time, mill, and grinding media). Based on Bond's energy Equation (5), a comparative work index Equation (6) was proposed [33]. When testing ore samples at the same operating conditions, the equation provides a ratio between reference and test ore grinding characteristics. Different researchers have used this approach to calculate the work index of an ore using another ore of known work index [34–36].

$$E = 10W_i\left(\frac{1}{\sqrt{Y_{80}}} - \frac{1}{\sqrt{X_{80}}}\right) \quad (5)$$

$$W_{it} = W_{ir}\frac{\frac{10}{\sqrt{P_r}} - \frac{10}{\sqrt{F_r}}}{\frac{10}{\sqrt{P_t}} - \frac{10}{\sqrt{F_t}}} \quad (6)$$

where W_{ir}—reference work index (obtained using the standard method), Wi_t—test work index, F_r—80% passing size (µm) of reference feed, F_t—80% passing size (µm) of test feed, P_r—80% passing size (µm) of the reference product, and P_t—80% passing size (µm) of test product.

Since the W_i of the as-received sample has been estimated, a comparative method was used to establish the Bond work index (as a measure of grindability) of the microwave-pretreated iron ore samples. To do so, a bulk-treated sample (1710.5 g) was fed into the laboratory standard Bond Ball mill using the same set of steel balls (20,125 g) as that used to perform the grindability test of as-received samples. The sample was ground for 425 runs (based on the average number of runs for the BBMG test of the as-received sample). The product particle size analysis was then performed to obtain P_t. Also, grindability tests were conducted for the separate batch-microwave-treated samples (125 g each to make a total of 1750 g, of which 1710.5 g was sampled for the grindability tests) at 30, 60, and 90 s residence times. The sub-sample of 125 g can be obtained by dividing a 500 g sample into four pieces (see Section 2.5). Since 1710.5 g is required for the BBMG test, 14 sub-samples of 125 g were merged (1750 g). The studied sample cannot be microwaved for longer than 90 s due to the observed increased work index due to fusion of iron ore particles (see Sections 2.5 and 3.4).

2.4. Thermal Pretreatment via Furnace

Thermal pretreatment of the studied samples (Figure 3a) were performed using an industrial furnace with a maximum heating temperature of 1800 °C (Nabertherm VHT 8/22-GR, Lilienthal, Germany; Figure 3b). The studied sample was batch-treated by putting 1710.5 g of the sample in a clay pot placed at the center of the furnace's heating chamber. Heating was performed from room temperature to 150 °C at a heating rate of 10 °C/min. After reaching the target temperature, the sample was heat-shocked for 1 h before cooling to room temperature within the heating chamber. This procedure was repeated at target temperatures of 200, 300, and 400 °C using separate representative samples.

2.5. Thermal Pretreatment via Microwave

Microwave pretreatment of minerals/ores is one of the innovative methods researchers have focused on to improve grinding operation and reduce comminution energy. In this work, a multimode microwave oven (Figure 3c) with cavity dimensions of 381 × 330 × 216 mm (Amana RC17S2, 2.45 GHz frequency, Benton Harbor, MI, USA) was employed for the microwave pretreatment of the prepared representative iron ore samples. The sample to be microwave-treated was prepared by step-wisely dividing a 5 kg sub-sample using a riffle-splitter, which produced approximately 507.5 g. The obtained sub-sample was then reduced to 500 g, as discussed in Section 2.1. The effect of irradiation time on the final temperature reached by the sample was studied using 500 g specimens, each from 30 s up to 180 s at 30 s intervals. At 90 s irradiation time and above, localized burning of iron ore particles was observed, leading to the fusion of particles. For each test, the final bulk temperature was immediately measured after reaching the target heating time using an infrared thermometer with a temperature range of 0–550 °C and reading accuracy of ±3 °C

(Habotest HT650B, Liheng Village, QingXi Town, Dongguan, China). The experiment was repeated three times using separate representative samples (obtained using the same approach as discussed above), and the average final bulk temperature was calculated and recorded. This procedure was repeated using 125 g samples (obtained by dividing 500 g sample into 4 sub-samples using a riffle-splitter) but with a maximum microwave residence time of 90 s, in response to having noticed particles fusing from this temperature upward. The effect of sample quantity on microwave treatment of the studied sample was studied using 125, 250, 500, 1000 (obtained by adding two 500 g sub-samples), and 1500 g (obtained by adding three 500 g sub-samples) specimens at 30 s radiation treatment time. In each case, the average final bulk temperature of three repeated tests was calculated and recorded. To study the effect of microwave pretreatment on the grindability of the selected samples, a representative sample (1710.5 g, sample required for ball milling; see Section 2.1) was bulk treated at 30 s microwave residence time. Also, separate representative samples were batch-treated at 30 s (125 g each to make a total of 1750 g, of which 1710.5 g was sampled for the grindability test). The grindability of the bulk-microwave-treated sample was compared with that of the batch-microwave-treated one. Based on the results obtained, the batch-microwave treatment approach was repeated at 60 and 90 s microwave residence time.

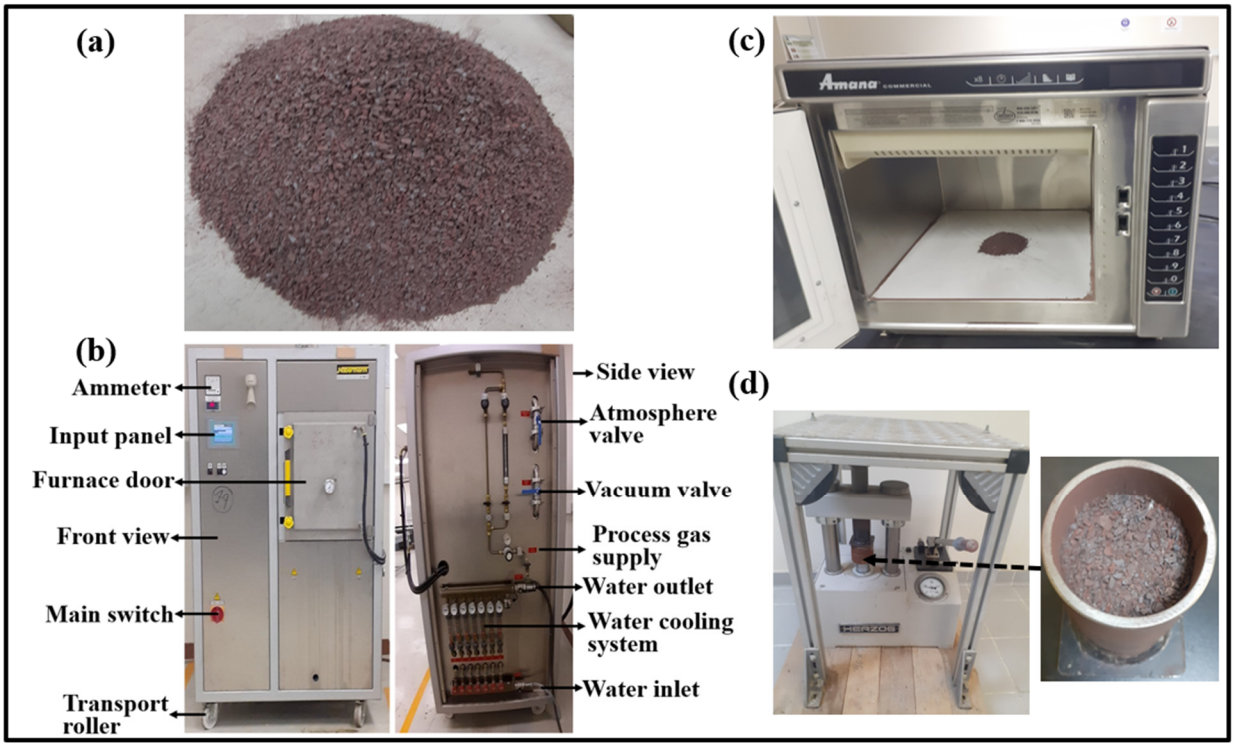


Figure 3. (**a**) Prepared iron ore sample (−3.35 mm), (**b**) industrial furnace (Nabertherm VHT 8/22-GR, Lilienthal, Germany), (**c**) microwave treatment, (**d**) mechanical pretreatment using piston die machine.

2.6. Mechanical Pretreatment

Mechanical pretreatment, as used in this study, is the application of load on crushed rock samples placed inside a pressure pot using a piston-die machine to create cracks in the samples (Figure 3d), which may improve the sample grindability. The compression device used in this study has a force capacity of up to 200 kN (Herzog, TP 20P, 16600 Sprague Road, Suite 400, Cleveland, OH 44130, USA). A pressure pot (cylindrical shape iron container: diameter—53.4 mm, height—90.0 mm) and a support die (diameter—53.3 mm, thickness—9.3 mm) were fabricated and used in this study. The sample mass (441.3 g) filled up to 80.7 mm of the pressure pot was calculated based on its packing density (2.4436 g/cm^3)

and the volume of the container (180.6 cm^3). The effect of applied pressure on the sample displacement was investigated to establish the maximum pressure at which the sample's displacement becomes constant. The support die was placed on the sample, occupying 9.3 mm of the upper space of the pressure pot. The load cell's base was lower and centered on the support die such that the applied load could be uniformly distributed on the sample by slowly jacking the hydraulic pump through its handle (at approximately 10 kN/min). Different confined bed compression tests were performed using applied forces of 10, 20, 30, and 40 kN. In each case, the sample displacement was measured and recorded. The procedure was repeated using separate prepared samples of quartzite. Also, the effect of applied force (at different delay times) on the sample's grindability was studied using separate quartzite samples (due to a shortage of iron ore samples). The packing density of the studied quartzite is 1.8321 g/cm^3 [15] and the volume of the container is 180.6 cm^3, hence, the sub-sample mass for the compaction test is 330.88 g. The stage compaction test (using 330.88 g sample each) was performed at 10 kN until approximately 5 kg of sample was reached—the sample required for the standard BBMG test. This sample was then properly mixed for homogeneity purposes. A BBMG test was performed as described earlier. The procedure was repeated for 20 and 30 kN using separately prepared sub-samples. The effect of load delay time was also investigated at 10, 20, and 30 min using various applied forces (10, 20, and 30 kN). The Bond Ball mill grindability test was performed in each case. Based on piston-die test optimization performed for the quartzite sample, 30 kN, zero-time delay condition was employed for the studied iron ore sample. The sub-sample (441.3 g) was batch-treated (four times) to obtain a total mass of 1765.2 g (of which 1710.5 g was sampled for the grindability test as discussed earlier; see Section 2.1 and Figure 4).

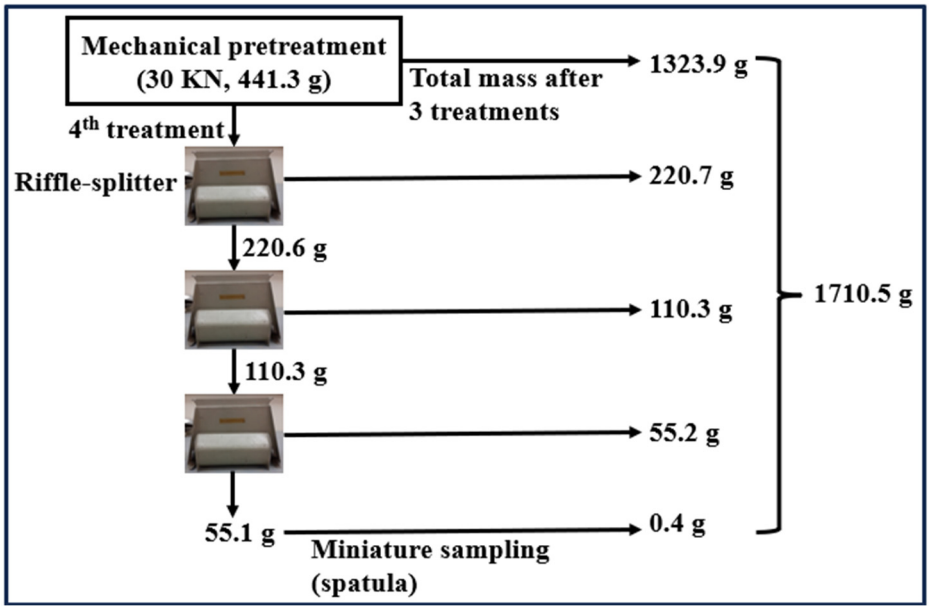


Figure 4. Sub-sampling procedure for hybrid thermal-mechanical-treated iron ore sub-samples.

2.7. Hybrid-Thermal Mechanical Pretreatment

To study the effect of hybrid thermal-mechanical pretreatment on the grindability of the studied samples, only thermal conditions (furnace or microwave) that have the most improved grindability on the studied samples were investigated. Previous studies showed that particles become smaller after a piston die test, which will reduce the impact of microwave radiation on materials' grindability since larger particles respond better to microwave treatment than smaller ones [5]. Therefore, we performed a piston die test of microwave-treated samples and investigated the effect of this process on the grindability of the studied samples. To achieve this, 125 g sub-samples were prepared (Section 2.5) and

batch microwave pretreatment was performed (f—2.45 GHz, microwave power—1.7 kW, radiation treatment time—60 s). The process was repeated 15 times to obtain the required sample (1710.5 g) for the BBMG test (Section 2.5). To obtain the sample mass for a piston die test (444.3 g, 30 kN, zero-time delay), the steps in Figure 5 were followed. A riffle-splitter was used to subdivide a 444.3 g thermal-mechanical pretreated sample into appropriate sub-samples and mixed with three times 444.3 g sub-samples. The 1710.5 g sample for the BBBMG test was obtained and the grindability test was performed (Section Comparative Method of Grindability Test). After these processes, the Bond work index of the sample was estimated using the comparative method. Also, the furnace-pretreated sample (heating rate:10 °C; maximum temperature: 400 °C) was mechanically treated (using a piston die device at 30 kN, zero-time delay), and the grindability test was performed using the same grinding condition as the microwave-treated one.

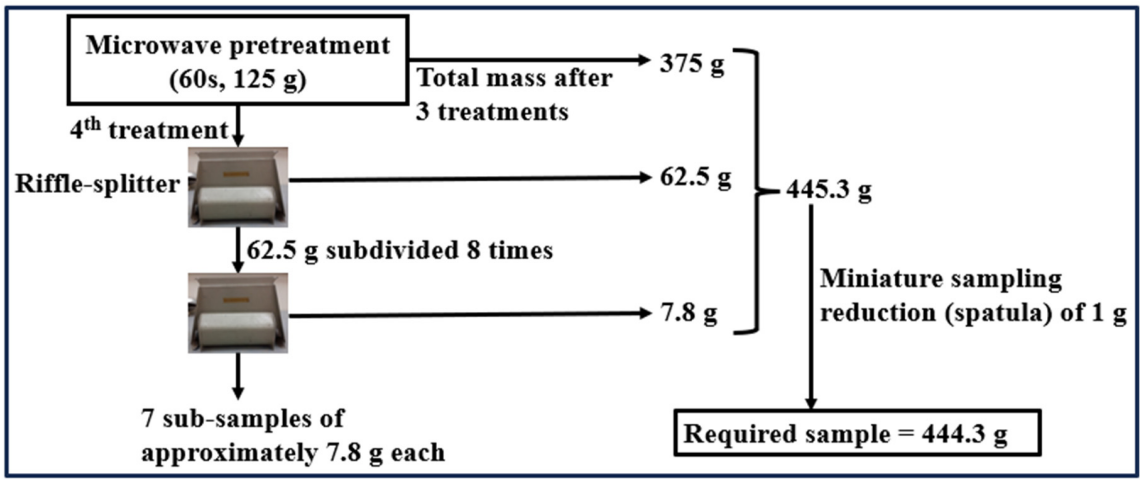


Figure 5. Sub-sampling steps for thermal-mechanical pretreatment of the studied iron ore.

3. Results and Discussions

3.1. FTIR and XRD Analyses

Figure 6 presents the FTIR spectra of the studied iron ore sample. The weak transmittance peak at 563 cm^{-1} is associated with F-O vibration related to hematite [37,38] and magnetite [38]. The FTIR transmittance peak at 785 cm^{-1} is associated with Si-O stretching of silicate minerals [37], while the peak around 873 cm^{-1} is associated with the $CrO4^{-2}$ vibrations—both peaks may be related to diopside mineral [38]. Meanwhile, the peak at around 1430 cm^{-1} is within the carbonate mineral bands, suggesting the vibration of calcium with oxygen [39], which may be linked to diopside. These results suggest that the studied sample contains hematite, magnetite, and diopside. It can be noted that the transmittance peaks of the studied samples remain nearly the same after microwave treatment (Figure 6).

The results of the XRD analysis of the studied sample are presented in Figure 7. Findings indicate that the studied sample contains three minerals: magnetite (Fe_3O_4, card number: 01-076-7161, 3.7% ± 2% by weight), hematite syn (Fe_2O_3, card number: 01-076-4579, 37.3% ± 4% by weight), and diopside ($(Mg_{0.845}\ Fe_{0.048}\ Al_{0.083}\ Ti_{0.002}\ Cr_{0.023})\ (Ca_{0.828}\ Na_{0.102}\ Mg_{0.045}\ Fe_{0.024}\ Mn_{0.001})\ ((Si_{0.981}\ Al_{0.019})_2\ O_6)$, card number: 01-075-9998, 59% ± 7% by weight). This result is in good agreement with the FTIR analysis. It can be inferred that the selected sample is an iron ore with diopside as the gangue mineral. Diopside may be found with interstitial magnetite and may have different colors, including white, grey, light blue, purple, light to dark green, and maybe multicolor [27]. Other mineral constituents may change the color of the diopside, as in the case of this studied sample (Figure 3a). The effect of microwave irradiation on this type of ore has not been presented in the literature. The crystallographic directions of the mineral phases remain the same after microwave treatment, indicating that microwave irradiation has no significant change in mineral

phases of the studied sample. This result agrees with that of the FTIR. The crystallite size and lattice strain of the mineral phases in the investigated untreated sample are presented in Figure 7. Since the crystallite sizes of hematite and diopside have close values, such minerals may be difficult to liberate, resulting in high grinding energy requirements, production of fine particles, and challenges in downstream operations. Previous studies suggested that interlocking of magnetite with diopside may cause metallurgical challenges [27]. In addition, the presence of a small amount of magnetite with fine grain disseminated in the sample's matrix may require more energy to liberate it. Nevertheless, the use of microwave pretreatment for the studied samples improved the grinding process, leading to a reduction in the work index (Section 3.4). This improvement may be attributed to changes in crystallite sizes of the mineral phases and crack propagation on the particles' matrix after microwave pretreatment (see Section 3.2). The XRD analysis showed that the crystallite size of the studied iron ore significantly changed after microwave treatment—the crystallite sizes of hematite and magnetite decreased from 430(6) Å to 26(4) Å and 265(3) Å to 33.82(6) Å, respectively. Meanwhile, that of diopside increased from 438(9) Å to 501.7(9) Å. Further studies are still necessary to determine the effect of microwave irradiation on the mineral liberation of this type of sample and its effect on downstream operations, including the effect on magnetic susceptibility and iron recovery.

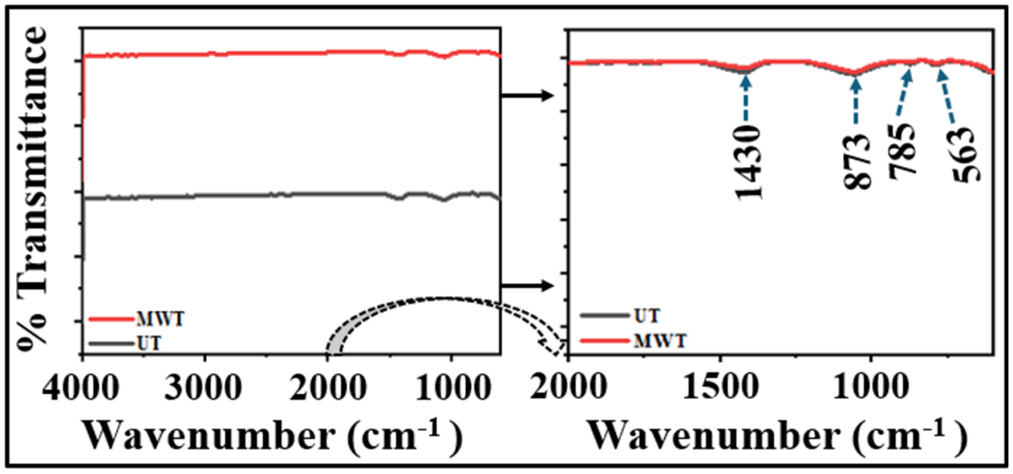
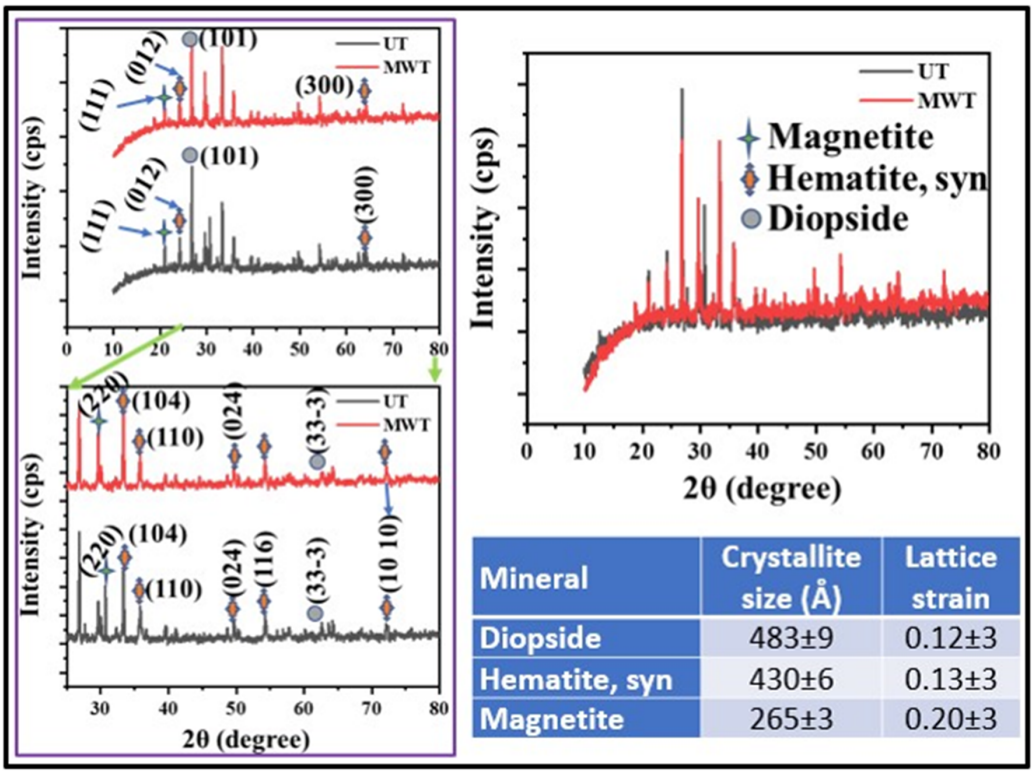


Figure 6. Results of the untreated (UT) and microwave-treated iron ore samples.

3.2. SEM Analysis

The studied sample exhibits a coarse, rough surface texture with various crystallite sizes (Figure 8a–c), which agrees with that of XRD analysis (Figure 7). Minor intergranular and intragranular cracks can be observed on the larger particles after 60 s microwave treatment residence time (Figure 8d,f). After further microwave treatment for up to 90 s, more cracks developed on the particles, as shown in the SEM micrographs (Figure 9a–c). Meanwhile, a fusion of smaller iron particles can be noticed after microwave treatment (Figure 9d–f). This fusion of smaller particles produced bigger ones that take more energy to grind because of strength reinforcement. A similar fusion of particles during microwave treatment had been reported for porphyry copper ore, which increased the Bond work index by 7% [40]. The XRD analysis results indicate changes in minerals' crystallite sizes after microwave treatment. Also, the FTIR transmittance spectra of the studied samples do not change after microwave treatment. However, it may be inferred that the observed fused particles are related to changes in the crystallite sizes of the investigated samples after microwave treatment (see Section 3.1). The results obtained in this study suggest that developed cracks on the larger particles (Figure 9a–c) led to an improved Bond work index of the microwave-treated sample by 6.9%. However, due to the presence of fused particles (Figure 9d–f), the work index at 90 s microwave treatment is higher than that at 60 s microwave treatment (Section 3.4).



Mineral	Crystallite size (Å)	Lattice strain
Diopside	483±9	0.12±3
Hematite, syn	430±6	0.13±3
Magnetite	265±3	0.20±3

Figure 7. XRD analysis before and after microwave treatment at 1 min microwave irradiation time (UT—untreated sample, MTD—microwave-treated sample for 1 min).

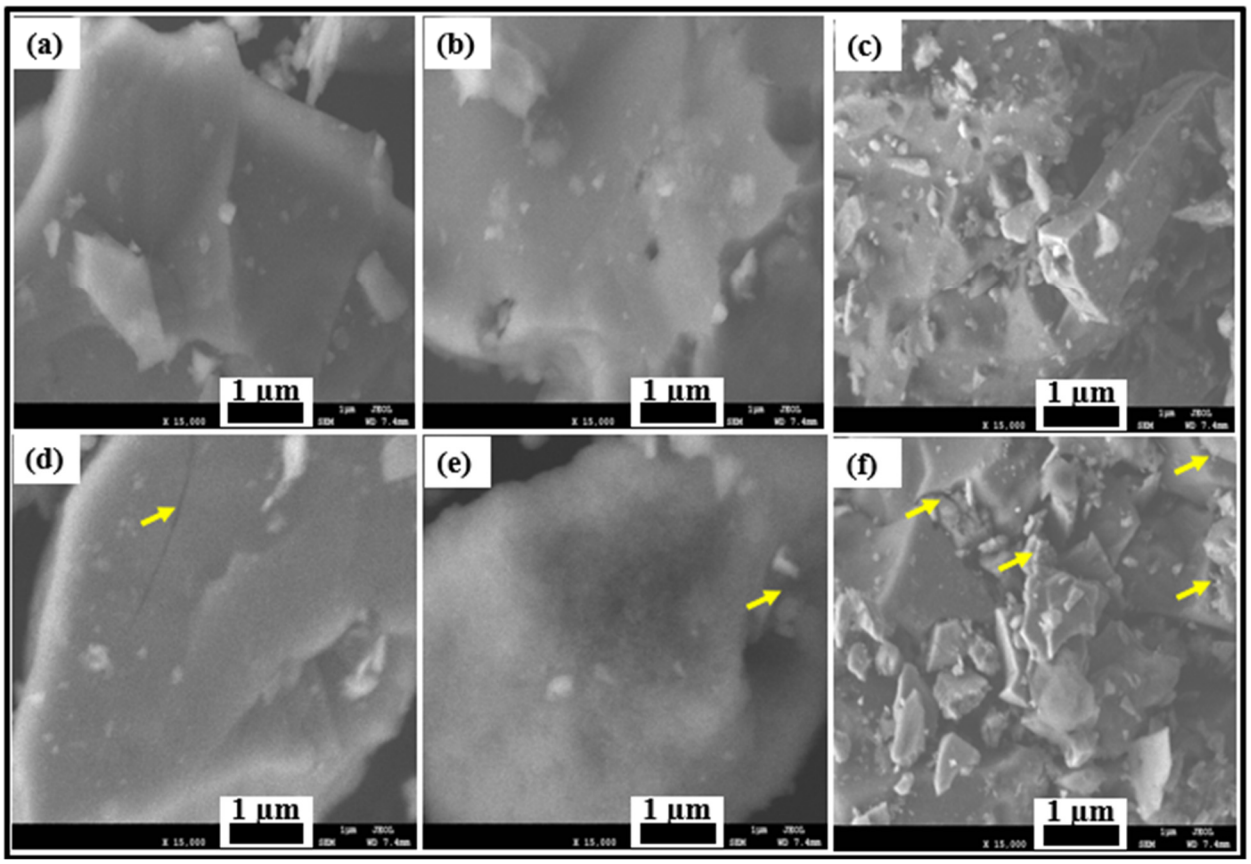


Figure 8. SEM images (15,000×, 5 kV) of the untreated and microwave-treated (2.45 GHz, 1.7 kW, 60 s) samples: (**a**–**c**): untreated, (**d**–**f**): microwave-treated.

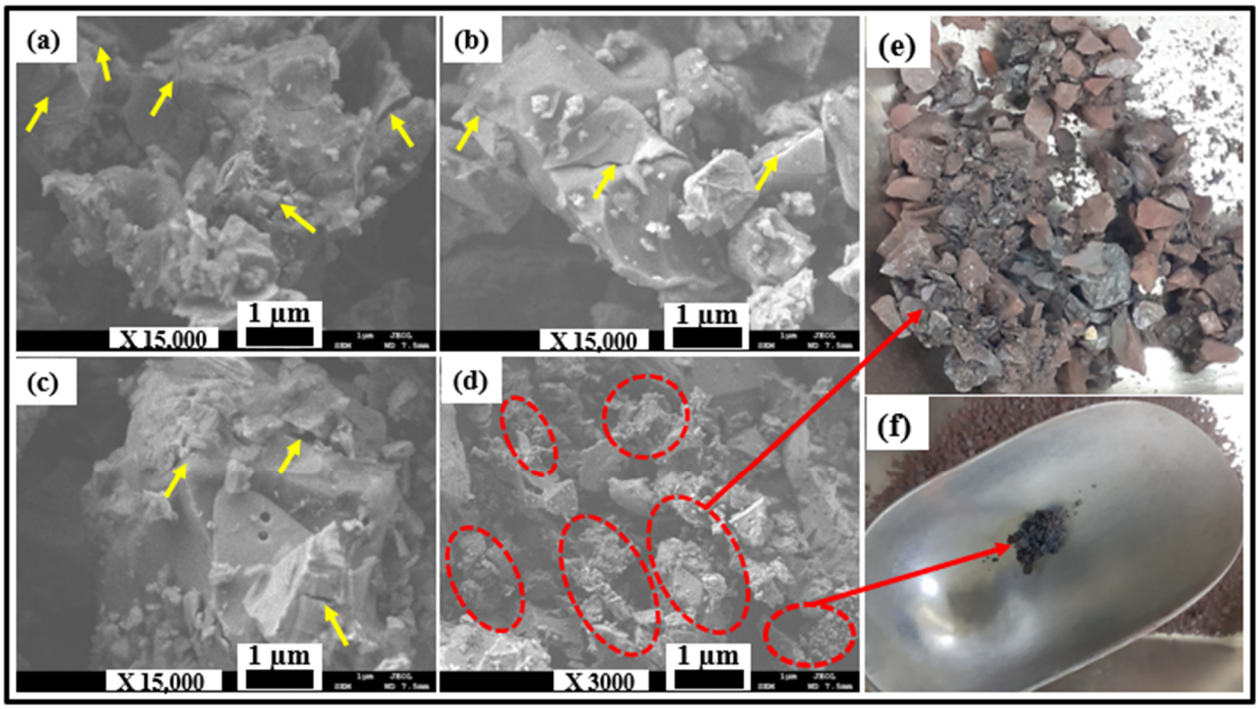


Figure 9. (**a**–**d**) SEM micrographs of the studied microwave-treated (2.45 GHz, 1.7 kW, 90 s) sample; (**e**,**f**) images of fused iron ore particles.

3.3. Electrical Conductivity and Dielectric Properties

The electrical and dielectric properties of iron ore, consisting of magnetite, hematite, and diopside, were analyzed over a temperature range from 300 K to 1100 K. This study examines how temperature affects electrical conductivity and dielectric properties. Figure 10a shows the variation of electrical conductivity (σ) with inverse temperature (1000/T) for different frequencies. The σ increases with temperature for all frequencies, indicating typical semiconducting behavior. At lower frequencies (5 kHz and 10 kHz), a steep decline in σ is observed, suggesting higher thermal activation energy for conduction. As frequency increases, the decrease in σ becomes less steep, indicating reduced thermal activation of charge carriers. At 8 MHz, σ remains relatively stable, reflecting the limited mobility of charge carriers due to the inability to follow the rapidly alternating electric field. Figure 10b shows the dielectric constant (ε') as a function of temperature for various frequencies. The ε' increases significantly with temperature for all frequencies. At 5 kHz and 10 kHz, ε' exhibits a steep increase above 600 K, indicative of enhanced polarization mechanisms like dipolar alignment and interfacial polarization. Higher frequencies (1 MHz and above) show a more gradual increase in ε', suggesting less effective dipolar alignment with the rapidly alternating electric field. The substantial increase in ε' at 5 kHz, reaching around 6×10^5 at approximately 1100 K, underscores strong polarization and dielectric relaxation processes at lower frequencies and elevated temperatures. Figure 10c shows that dielectric loss (ε'') increases with temperature for all frequencies. At lower frequencies (5 kHz and 10 kHz), ε'' rises significantly above 600 K, indicating effective thermal activation of charge carriers and higher energy dissipation. At higher frequencies (above 100 kHz), the increase in ε'' is more gradual. The peak ε'' at 5 kHz reaches about 6×10^7 around 1100 K, highlighting strong ε'' due to conductive and polarization effects from magnetite and hematite. Increased temperature enhances charge carrier mobility, leading to higher ε'' through the alignment and reorientation of dipolar entities in the rock matrix. Figure 10d shows distinct peaks in the loss tangent (tan δ) at various temperatures for different frequencies. At 5 kHz, a prominent peak appears around 850 K, with tan δ values close to 10^5. As frequency increases, these peaks shift to lower temperatures (e.g., 830 K at 10 kHz, 800 K at 20 kHz), indicating a unique relaxation mechanism. This shift suggests that the specific properties

of the minerals influence the dielectric relaxation in these rocks. The pronounced peak at 5 kHz around 850 K likely results from dipolar alignment or interfacial polarization. As frequency increases, the relaxation process requires lower thermal energy, causing the temperature shift. The temperature impacts iron ore's electrical and dielectric properties, particularly after 550 K, by enhancing charge carrier activation and dipolar realignment, especially in magnetite and hematite. However, at temperatures lower than 550 K, it seems the electrical and dielectric properties are independent of temperature.

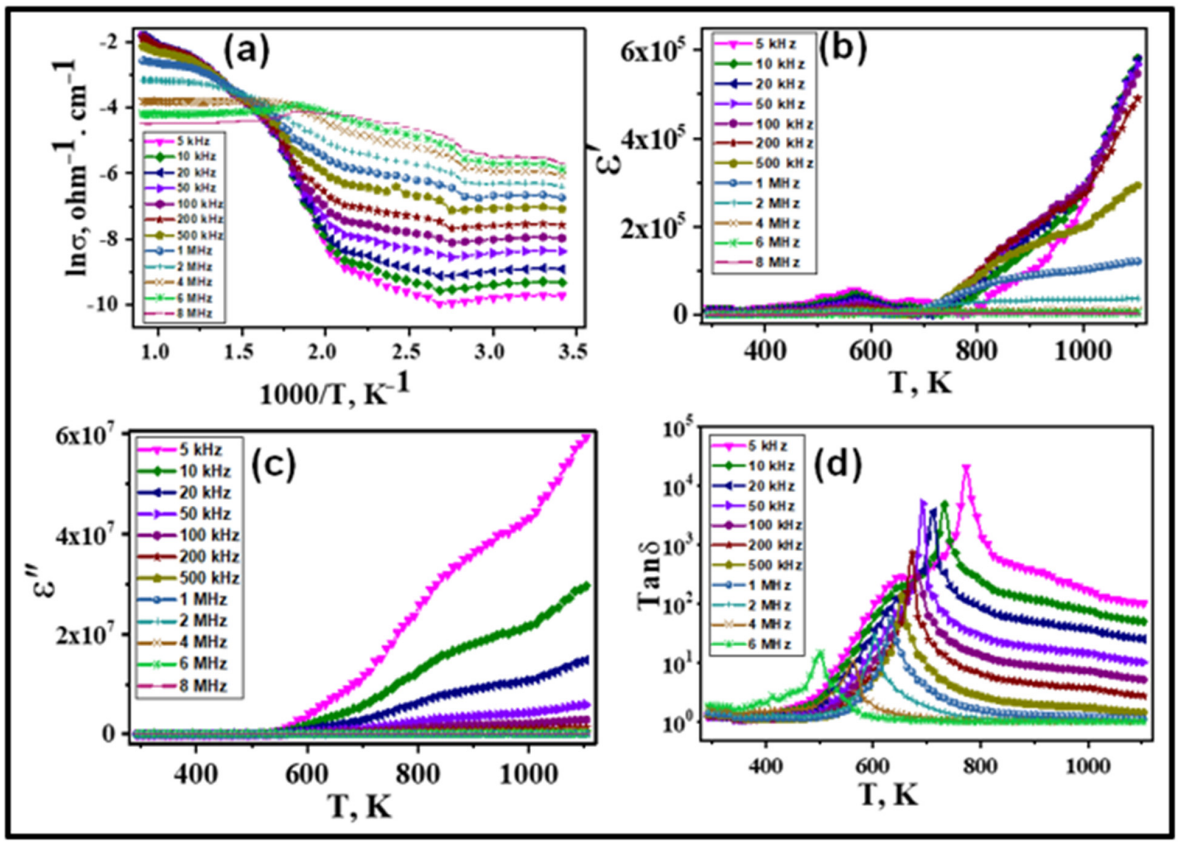


Figure 10. Electrical and dielectric properties of (**a**) electrical conductivity (σ) as a function 1000/T, where T is the absolute sample temperature, (**b**) dielectric constant (e′) (**c**), dielectric loss (e″), and (**d**) dielectric loss tangent (tanδ).

3.4. Effect of Pretreatment Methods on Work Index

The Bond work index of the investigated sample (sieve test size = 106 µm, average pack density = 2.4436 g/cm^3, P_{80} = 91.60 µm, F_{80} = 2742; G(g/rev) = 1.0234 m) is 17.5 KWh/ton. Microwave irradiation of material usually increases the material's temperature. Using 500 g representative samples (see Section 2.5), results indicated that average temperatures increased as the microwave residence time (1.7 kW, 2.45 GHz) increased (Figure 11b). Findings also indicated that the higher the sample mass, the lower the average final temperature reached after microwave treatment (Figure 11a). Based on the findings from Figure 11a,b, a lower sample mass of 125 g was considered for investigating the effect of microwave treatment on the selected iron ore sample (see Section 2.5). Results indicate that the average final temperatures of the investigated samples (125 g, 1.7 kW, 2.45 GHz) at 30, 60, and 90 s microwave irradiation times are 157.5, 258.7, and 302.3 °C, respectively (Figure 11c). As can be noted in Figure 12, the particle size distribution of the studied samples changes at different microwave residence times after grinding using the same milling condition. Figure 12 shows that the P_{80} improved after microwave treatment at 30, 60, and 90 s, leading to improved work indexes by 9.1%, 19.8%, and 6.9%, respectively (Figure 12). Firing and fusion of iron ore particles were noted during microwave treatment

at 90 s and beyond. This may be attributed to localized heating and intense, sudden temperature increase of iron ore particles. At 90 s microwave treatment time, the work index of the microwave-treated sample is higher than at 60 s microwave treatment due to greater amount of energy required to liberate fused particles, suggesting that further microwave treatment of the studied sample may continue to increase the work index of the studied sample. Therefore, microwave treatment of the investigated sample beyond 60 s is not desirable. As per the effect of conventional thermal pretreatment on the grindability of the investigated sample, the W_i of the studied sample slightly decreases with increasing temperature, with approximately 4.6% improvement in W_i after 400 °C (Figure 11d). This indicates that conventional thermal pretreatment has no significant effect on the grindability of the studied sample under the investigated temperature range. These results agree with the electric and dielectric properties of the investigated sample since the electrical and dielectric properties are independent of temperature at lower temperatures (Section 3.3).

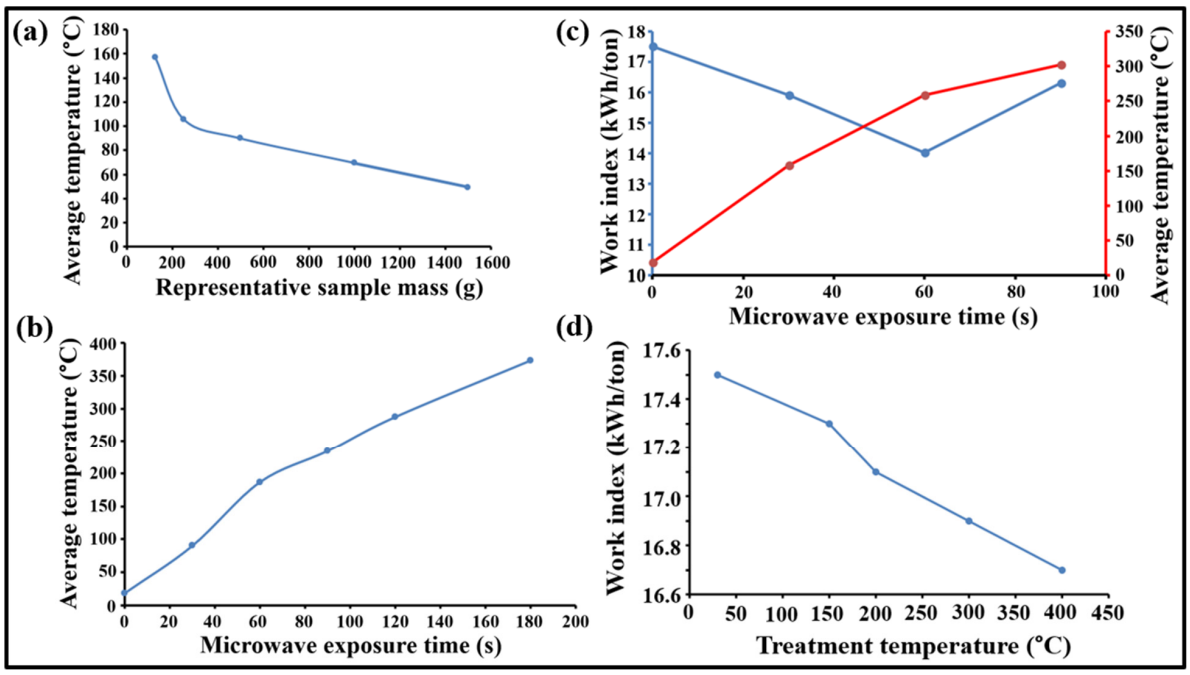


Figure 11. (**a**) Effect of sample mass on microwave treatment of the studied samples, (**b**) Effect of microwave treatment times on iron ore sample's (500 g) response to microwave treatment, (**c**) Work index of the studied iron ore sample (microwave treatment conditions; 2.45 GHz, 1.7 kW, 125 g batch treatment) and average temperatures after microwave treatment, (**d**) Effect of furnace pretreatment on work index of the investigated samples.

To better understand the appropriate sample mass for the mechanical pretreatment test, quartzite and iron ore representative samples were used for sample displacement tests after subjecting them to different applied pressures using a piston die machine. Results show that the higher the applied pressure, the higher the sample displacement (Figure 13a). At 14.86 and 19.81 MPa, the sample displacements nearly have very close values; therefore, 14.86 MPa was regarded as the optimum applied pressure. Due to limited iron ore samples, quartzite samples were used for the optimization of mechanical pretreatment (Figure 13b). Results indicate that the higher the applied pressure, the better the improvement in the W_i of the investigated samples (Figure 13b). Also, the higher the delay time, the better the improvement in the grinding behavior of the studied samples (Figure 13c). The reduction in W_i at 14.86 MPa with zero-time delay is almost the same as that achieved at 4.95 MPa with 30 min delay time (Figure 13b). For this reason, higher applied pressure without delay time was considered for the studied iron ore samples, as delaying ore grinding may affect the downstream process. Findings show that the W_i of

the studied iron ore sample after mechanical pretreatment at 14.86 MPa (zero-time delay) is 14.81 kWh/ton, equivalent to approximately 15.4% improvement in the Bond work index. This result suggests that the studied iron ore sample showed a good response to mechanical pretreatment. The combined microwave-mechanical pretreatment (1 min microwave irradiation time, 14.86 MPa, zero-time delay) decreased the W_i of the investigated sample from 17.50 kWh/ton to 13.54 kWh/ton equivalent to 22.6% improvement in its grindability. For the combined conventional thermal-mechanical pretreatment, findings indicate that at 400 °C and 14.86 MPa (zero-time delay), the grindability of the studied sample decreased from 17.50 to 14.14 kWh/ton, equivalent to 19.2% improvement in its Bond work index.

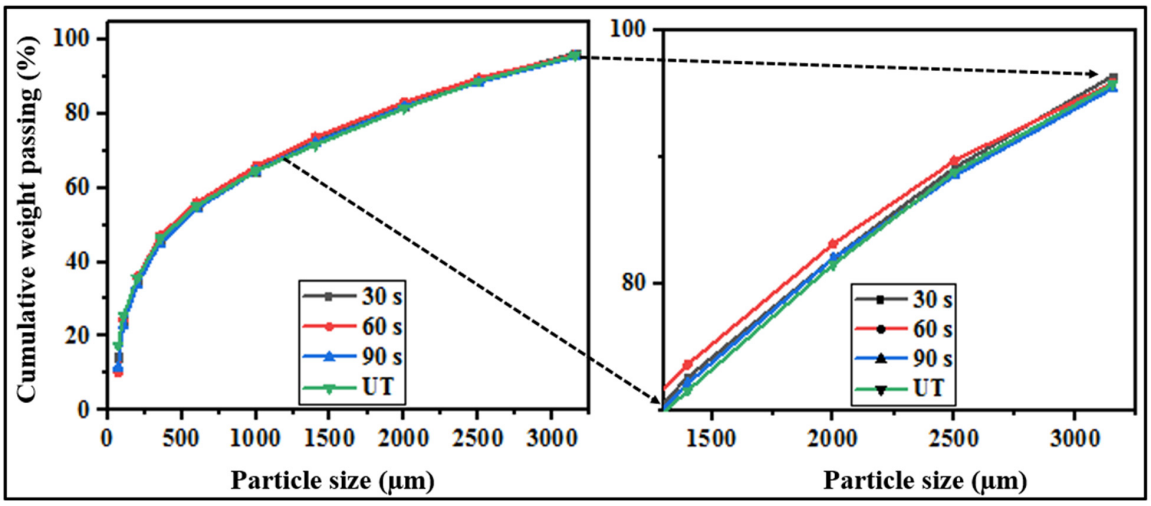


Figure 12. Particle size distribution of untreated (UT—0 s microwave) and microwave-treated iron ore samples after grinding operations.

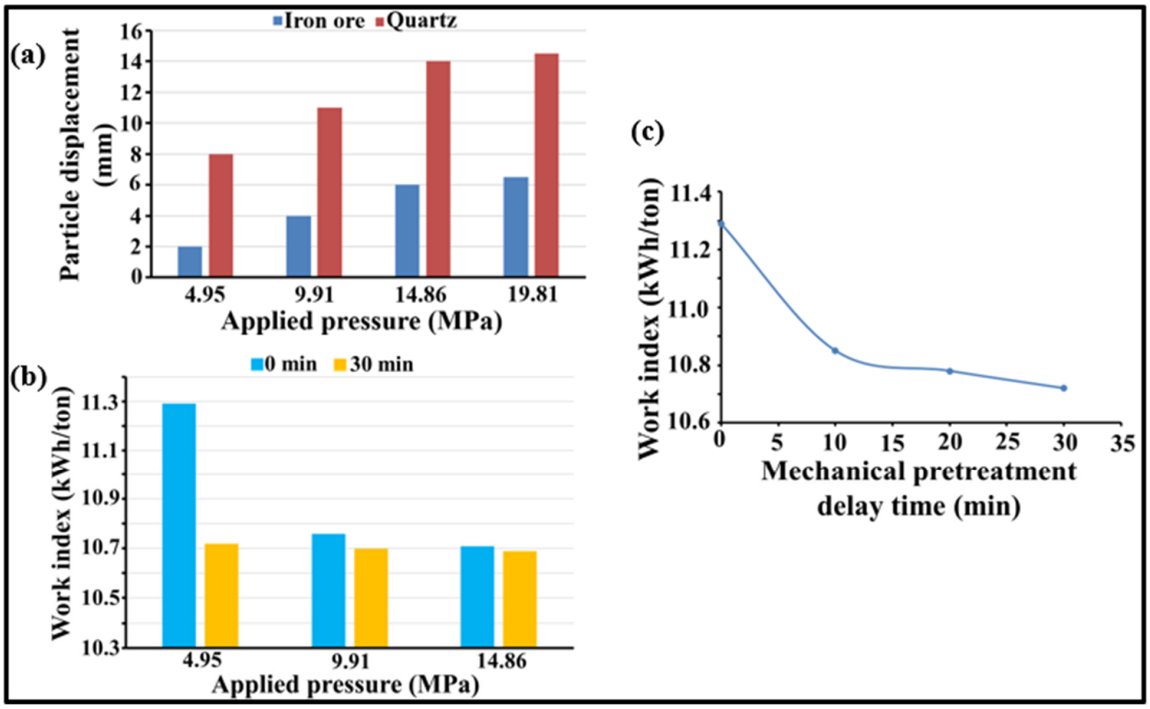


Figure 13. Optimization of mechanical pretreatment test using the quartzite representative samples (reference W_i = 11.49 kWh/ton); (**a**) particles displacement test; (**b**) mechanical pretreatment tests at 0 min and 30 min delay time, using quartzite representative samples; (**c**) effect of delay time on mechanical pretreatment.

4. Conclusions

This paper investigates hybrid thermal and mechanical pretreatments to improve the grinding behavior of fine-grain iron ore (hematite and magnetite interlock with diopside gangue). The mineralogy analysis of the representative sample using the XRD method indicated that the studied sample contain 37.3, 3.7, and 59.0% by weight of hematite, magnetite, and diopside, respectively. The electrical property of the investigated samples showed temperature independence at lower temperatures up to around 550 K, after which the electrical conductivity of the sample increased as the temperature increased. This result agrees with the findings of the grindability tests of pretreated samples using conventional heating since no significant improvement in grindability was achieved below 550 K. At 673 K, only a 4.6% improvement in grindability was achieved. The thermal pretreatment via microwave improved the grindability of the investigated sample up to 19.8% at 60 s microwave irradiation time. Mechanical pretreatment of the sample using a piston die machine at 14.86 MPa (zero-time delay) enhanced the sample's grindability by 15.4%. The hybrid thermal pretreatment was investigated in two ways: conventional-mechanical (400 °C and 14.86 MPa at zero-time delay) and microwave-mechanical (60 s and 14.86 MPa at zero-time delay). Findings indicated that the former improved the grindability of the sample by 19.2% while the latter enhanced the grindability of the sample by 22.6%. These results suggest that stand-alone mechanical pretreatment or microwave pretreatment may be more beneficial in improving the grinding behavior of the studied fine-grain iron ore sample. The results of the mechanical pretreatment obtained in this study may be used in a simulation of the HPGR system for grinding operations of similar iron ore. Future study should focus on economic feasibility of the proposed pretreatment techniques for industrial applications. Among the factors to be considered are input energy of microwave and energy required for mechanical pretreatment.

Author Contributions: Conceptualization, S.O.A.; methodology, S.O.A., H.M.A., H.A.M.A. and A.S.; formal analysis, S.O.A. and A.S.; investigation, S.O.A.; resources, H.A.M.A., H.M.A., A.A., K.L. and R.A.; data curation, S.O.A. and A.S.; writing—original draft preparation, S.O.A., A.A. and A.S.; writing—review and editing, S.O.A., A.S., H.A.M.A. and A.A.; visualization, H.A.M.A., H.M.A., A.A., K.L. and R.A.; supervision, H.A.M.A., H.M.A., A.A. and K.L.; funding acquisition, S.O.A., H.M.A. and H.A.M.A. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by the Deanship of Scientific Research (DSR), King Abdulaziz University, Jeddah, under grant No. DG-026-306-1441 and G: 570-135-1441. The authors, therefore, gratefully acknowledge the DSR's technical and financial support.

Data Availability Statement: The original contributions presented in the study are included in the article, further inquiries can be directed to the corresponding author.

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

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