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Microwave and Ultrasonic Pretreatment-Assisted Upgradation of Iron Ore of Karnataka Region from India

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Abstract

Essentially, size reduction is the very first and vital segment of mineral processing. Microwaves (MW) for comminution have piqued the interest of researchers and industrialists in recent years. The use of microwaves in processing industries is solely due to some of the unique benefits they provide. Because MW heating does not rely on temperature gradients but instead generates heat as a result of molecules interacting with microwaves and the resulting internal friction, this treatment can result in rapid and volumetric heating. Because of these factors, this treatment can significantly reduce process times while also improving product quality. The current study demonstrated the formation of intergranular fractures and their influence on comminution energy after MW treatment of Indian iron ore. Scanning Electron Microscopy (SEM) analysis confirmed that pretreatment with MW made the ore more brittle, making it more amenable to grinding, and a decrease in grinding energy of the pretreated ore was observed in comparison to the untreated sample. The X-Ray Diffraction (XRD) results demonstrated an increase in ore crystallinity following MW exposure, substantiating the pretreated sample's lower grinding energy consumption. Additionally, ultrasonication of MW pretreated ore was attempted to determine whether the combined treatment method could improve the ore quality, and the combination techniques were successful in increasing the iron content by 41% while decreasing the alumina and silica contents by 59% and 38%, respectively. The findings support the use of both MW irradiation and ultrasonication as promising methods of improving mineral quality.

Keywords: *Grinding Energy, Iron Ore, Microwave, Ultrasonication, Work Index*

1.0 Introduction

In the earth's crust, the precious minerals are often seen trapped within the gangue particles and are generally present in meager quantities. Mineral-to-gangue ratios in precious metals, for instance, such as gold may be as low as 0.1ppm¹. Comminution is a major step in mineral processing which promotes the dissociation of valuable minerals from those of gangue. However, mineral processing is known to be a highly energy-consuming process during extractive metallurgy, accounting for nearly 6% of global energy usage2 . Comminution alone consumes so much energy that it accounts for roughly 70% of the net energy input for mineral extraction³. According to

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another study, comminution processes account for about 1.5% of net energy consumption in the United States⁴. This is primarily due to lower comminution equipment efficiencies, with only about 1% of the energy input to this equipment known to be used for comminution⁵. Limited ore reserves, as well as low-grade ores combined with high comminution costs, could be a heavy burden on mineral processing industries, affecting metal commodity prices, especially given the ongoing increase in global demand for minerals. Furthermore, concentration with a higher degree of liberation would be required for subsequent beneficiation processes. This emphasizes the importance of researching energy-minimizing steps and alternatives during mineral dressing so that sufficient quantities and grades of mineral raw materials are produced.

Reduced comminution costs could be one way to improve process economics. Considerable research has been conducted in using microwaves to induce thermal breakage of minerals⁶, and MW treatment has proven to be economically viable when compared to conventional comminution7 . In contrast to conventional heating modes such as conduction and convection, which involve heating the entire mass of the material, MW heating involves selective and radial heating from the inside out via dipole alignment and particle rotations. The microwaves directly reach the materials to be heated, resulting in rapid heating of the material. These waves can cause volumetric heating because they can penetrate materials and deposit energy⁸. MW has a wide range of commercial applications, including food, pharmaceuticals, chemicals, paper, plastics, and so on⁹. Because different minerals have different MW absorbing capacities, this treatment has been investigated in mineral processing and metallurgy¹⁰⁻¹³ because it can induce thermal stress and cause rupturing of grain boundaries within the ore matrix due to differential heating of the material and corresponding differential stresses. It has also been discovered that, while many precious minerals absorb microwaves well, gangue minerals do not¹⁴. Some of the most important applications of these radiations for metallurgical applications have proven to be rock disintegration, improved liberation and recovery, and so on^{15} .

On the other hand, Ultrasonic (US) treatment has also been studied for reducing communication energy inputs16, but it is not widely used due to additional process energy requirements. It is, however, used in the beneficiation of low-grade ores¹⁵. US waves are pressure waves with frequencies greater than 20 kHz² and when they propagate through the liquid, they cause vibrations in the medium and the formation of a large number of micro-bubbles known as cavitation bubbles, particularly at solid-liquid interfaces. These bubbles expand and generate high temperatures and pressures, and at extremely high temperatures and pressures, they implode instantly, releasing strong shock waves and fracturing the solid boundaries in their locality¹⁷⁻¹⁹. In the case of ore processing, they aid in mineral liberation.

The current work is concerned with pre-treating an ore sample of Indian origin with MW irradiation in order to reduce energy expenditure during subsequent grinding operations. The iron ore sample was MW pretreated, and the grinding energy consumption was calculated and compared to that of the untreated ore. Furthermore, US treatment of the MW pretreated ore was performed to evaluate if this combined treatment assisted in upgrading the quality of the ore under study.

2.0 Materials and Methods

2.1 Experimental Setup

The experimental setup consisted of a 700W power output domestic MW oven (ONIDA MO20SMP11B) and a probe-type thermocouple with a multimeter capable of measuring temperatures up to 300º C. For US treatment, a probe-type ultrasonicator (frequency: 20 kHz; net power output: 2500 W) was used.

2.2 Sample

The iron ore sample used in the work was procured from one of the Indian mines located in the Chitradurga region of the state of Karnataka. The low-grade hematite ore was in the lumped form. Approximately similar-sized lumps with sizes ranging from 10-40 mm were selected for the study and subjected to MW treatment.

The sample in pulp form was used for ultrasonic treatment. The ore samples were ground, and a 20% (w/v) suspension in water was prepared. The pulp density was chosen based on previous research that demonstrated that US treatment is more effective on pulps with lower densities than those with higher density values¹⁷.

2.3 Microwave Pretreatment

Approximately 50 g of the lumped sample (containing almost equal-sized chunks) was subjected to MW treatment. A graphite crucible was used to place the sample because the MW-safe food-grade glass container failed to withstand the temperature used for the MW pretreatment of the iron ore sample. Using the graphite crucible, a treatment period of about 60-70 s was first tested. However, graphite being a strong absorber of MW, allowed only a little radiation to pass through the sample, failing to induce rapid dipolar orientation and thus uniform thermal breakage of the sample. As a result, a modified method of sample placement was adopted: the crucible was turned upside down, and the sample was kept on the flat surface of the crucible (bottom face) so that the majority of the sample could be exposed to radiation, except for the portion resting on the crucible surface. The samples were irradiated for different time intervals chosen at random, such as 60 s, 90 s, 120 s, and 180 s, using this new method of sample placement. Safety measures were taken to carefully remove the hot samples from inside the MW oven and clean the interior of the oven after the treatment of every sample.

A probe-type thermocouple with a multimeter was used to measure the temperature up to which each sample was treated. Following the sample pretreatment, each sample was removed from the oven and the thermocouple was placed in contact with the sample surface, with the other end connected to a multimeter to check the temperature. The multimeter showed a sudden increase in the temperature readings when initially held in contact with the sample, and then gradually slowed down. At one point, the increasing temperature values remained constant and then began to decrease. This constant temperature value, after which the temperature started to fall, was taken as the temperature of the sample, and the values of treatment time and temperature were plotted.

2.4 Grinding Test

Each of the MW-treated samples was subjected to size reduction using a ball mill (Size: 24" dia x 18" length; Drive: 4 HP, 415 volts, 3 Phase, 50 cycles, 1440 rpm). The ball mill was run five times for 3 minutes for each sample. After every run of the ball mill, the sample was taken out and sieved to 300 µm using a sieve shaker for 5 minutes.

The mass remaining after 3, 6, 9, 12 and 15 minutes of milling was then measured using a weighing scale to calculate the percentage mass retained. The same process was repeated for the MW untreated sample. Using this data, the respective values of retained mass percentage were plotted against the cumulative grinding time of MW-treated and untreated ore.

The sieve size through which 80% of the ground sample passes was noted down. The work index of iron ore was calculated using Bond's method, and the energy required for grinding the ore was calculated based on this. Finally, the grinding energies of untreated versus MW-treated ore were compared.

2.5 Ultrasonic Treatment

US treatment was performed on both the untreated and MW-treated samples, with one reference sample, i.e., the sample that was MW pre-treated for 180s, selected for ultrasonication. The untreated sample received US treatment for 300 seconds, whereas the pre-treated sample received US treatment for 600 seconds. The sample temperature was measured after US treatment, and the solid fraction was collected after sedimentation for 2-3 days, followed by filtration through a 20-micron Whatman filter paper. The residue was sun-dried to avoid any changes in chemical composition that would occur if dried using other thermal methods and then subjected to a series of characterization tests.

2.6 Analytical Methods

The chemical characterization of the ore sample before and after MW pretreatment was carried out using Fourier Transform Infrared Spectroscopy (FTIR). The transmittance percentage was recorded for wavenumbers ranging between 4000 cm^{-1} to 400 cm^{-1} . For topographic evaluation at the microscopic level, SEM analysis (JEOL JSM-IT300 with Energy Dispersive X-Ray Analysis (EDX) Unit was performed. In order to check if there are any significant changes in the microstructure of the treated ore, XRD analysis was conducted. The following specifications were used: Intensity range: 0-700 counts per second; Scan rate: 3º per minute; Range (2 theta): $0 - 90^\circ$.

To evaluate if US treatment can result in any change in the chemical makeup of MW-treated samples, Field Emission Scanning Electron Microscopy (FESEM),

EDX, and XRD analyses were carried out following this treatment.

3.0 Results and Discussion

3.1 Treatment Time Versus Sample Temperature

The time versus temperature data obtained during ore pretreatment is presented in Figure 1, which validates the stable interaction as well as adequate penetration of electromagnetic radiations into the ore sample. A reasonably linear relationship with a coefficient of regression of 98.8% was obtained between the tested variables, demonstrating the need to expose the ore to MW for longer durations in order to promote better MW absorption by the ore sample. The finding could be correlated well with the laboratory observation of sample surface spluttering as the treatment time increased. However, for the same reason, the ore sample under consideration was not treated for durations longer than 180 s, when in the lumped form. The optimal pretreatment time of 180 s in this case, which resulted in maximum energy transfer, may vary for other ores depending on factors such as the type and origin of ore, size, composition, throughput, MW power density, and so on. An increase in ore temperature with increased exposure time was also observed in the case of highphosphorous Egyptian iron ore²⁰. When another Egyptian iron ore was MW exposed, sample temperature increased with increased exposure time; however, in the same work

melting of the sample was observed to begin at 100 s, and total melting was observed at 150 s, so the MW treatment time was limited to 90 s^{21} .

3.2 Effect of MW Treatment on Sample Characteristics

To understand how MW treatment affects the intricate arrangement of elements and their bonding in the ore lattice, FTIR analysis of the treated ore samples was conducted. Figure 2 depicts the FTIR spectra of the untreated ore as well as MW-treated ore samples for time periods of 60 s to 150 s, respectively. The transmittance peak for the untreated sample was observed at 467.65 cm-1, and the percentage transmittance was 19.58%. When the ore sample was pre-treated with MW, the transmittance maxima changed; for different pretreatment durations, shifts in the peaks as well as variations in peak intensities were observed. MW treatment for 60 seconds produced a peak at 442.58 cm⁻¹ with a peak intensity of 23.81% , whereas treatments for 90 seconds, 120 seconds, and 180 seconds produced peaks at 546.72 cm⁻¹, 400.16 cm $^{-1}$, and 475.37 cm $^{-1}$, with peak intensities of 42.66%, 43.66%, and 61.02%, respectively. Because it is known that iron ore reacts primarily to infrared radiation with wavenumbers ranging from 750 cm⁻¹ to 400 cm⁻¹, the observations confirm the appearance of transmittance peaks at sample-specific regions before and after MW treatment. Additionally, the variations in peak positions during each treatment substantiate the role of microwaves

Figure 1. MW treatment time v/s ore temperature profile. Figure 2. Combined FTIR spectra of the untreated and MW-treated ore.

Figure 3. SEM images of the untreated and MW treated samples (**(A)** – untreated ore; **(B)**, **(C)**, **(D)**, **(E)** represent samples treated for 60 s, 90 s, 120 s, and 180 s, respectively).

in dislocating the bonds between various elements in the ore. An interesting observation was the increase in peak intensities with increasing pretreatment duration, which indicates the loosening of the ore matrix upon MW exposure, signaling the possibility of grinding the ore with less energy expenditure.

The results obtained are in accordance with the reported literature. The dissociation of the phosphorusbearing minerals from gangue was exclusively studied using FTIR during MW heating of Egyptian high phosphorous iron ore, and it was concluded that selective

Figure 4. XRD images of the untreated and MW-treated samples (**(A)** – untreated ore; **(B)**, **(C)**, **(D)**, and **(E)** represent samples treated for 60 s, 90 s, 120 s, and 180 s, respectively).

heating of iron oxides and gangue minerals after MW absorption facilitates this dissociation²².

Figure 3 shows the comparison of the SEM images between the untreated and the treated ore samples. This comparison suggests that after treating the ore with MW, the following changes occur: rupturing of the sample edges, an increase in the number of sharp particles, and thus an increase in the brittleness of the particles. These variations could be attributed to the differential heating within the ore body which leads to cracks forming along and across grain boundaries²³. Based on the fractures observed in the ore structure, it is possible to conclude that MW treatment improves ore crystallinity, grindability, and thus iron liberation. Similar studies, such as the one involving MW pretreatment of Indian coal²⁴, confirmed the presence of these types of intergranular fractures.

The production of micro-fractures along hematitegangue boundaries was observed in the case of oolitic iron ore from China, where the extent of fractures produced was found to be proportional

to the time of microwave treatment and the microwave power intensity²⁵. During the MW treatment of free-milling gold ore from Western Africa, cracks were observed along both intergranular and transgranular boundaries²⁶.

The electron microscopic observations were confirmed by XRD (Figure 4), which revealed a clear increase in peak intensities and a corresponding increase in crystalline properties of the ore (Table 1) with prolonged MW exposure.

The increase in crystallinity could be attributed to the induction of thermal stress in the ore as a result of MW exposure, which causes bond disruption between randomly spaced elements. This, in turn, indicates the

easy grindability of the MW pre-treated ore as compared to the untreated ore due to less resistance during grinding, and a positive correlation of MW exposure time with ore grindability. Additionally, crystal structure analysis (according to X'pert Highscore software) revealed that the ore sample has a rhombohedral crystal structure. The increase in crystallinity with MW exposure was also observed in the case of high-ash coal²⁴. Similar observations were made in the case of MW-treated Egyptian iron ore, where an increase in peak intensity was observed after MW treatment, and it was concluded that this observation is due to gangue mineral dissociation occurring at elevated temperatures as a result of MW $exposure²¹$.

3.3 Comparing Grinding Energies during MW Treatment

Because grinding is a common size reduction mechanism in mineral processing, the relationship between MW treatment and power consumed for comminution was investigated. Grinding energies of the MW-treated samples as well as the untreated ore were calculated and compared. However, prior to this, a weight fraction analysis was performed on both treated and untreated samples, and the percentage retention was plotted (Figure 5) to evaluate the efficacy of MW treatment in promoting efficient size reduction. The results show that ore is responsive to MW treatment. A clear decrease in percentage mass retention is observed with prolonged MW treatment, emphasizing the importance of MW exposure as well as the duration of exposure in fragmenting the ore. At the same time, a direct relationship between grinding time and mass fraction passed is visible. Overall, it is possible to hypothesize that prior MW treatment

Figure 5. Weight fraction analysis for the untreated and MW-treated samples.

can significantly reduce the amount of energy required to obtain powdered ore in its native form. For instance, while untreated ore retained approximately 85% of its mass when it was ground for 3 minutes and sieved, mass retention for the same grinding period decreased to 75% for the sample that received prior MW treatment for 90 seconds. Another source of evidence is a 25% difference in mass retained between untreated ore and ore treated for 180 seconds, which demonstrates the benefits of MW treatment in ore processing and quality improvement.

Following the fullsize analysis, grinding energy was calculated using Bond's work index method, which aids in ore classification based on the amount of energy required for size reduction. The work index and the corresponding grinding energy were calculated using Equations $(1)^{27}$ and $(2)^{28}$, respectively.

$$
W_i = 1.1 \frac{44.5}{P_k^{0.23} G^{0.82} \left(\frac{10}{\sqrt{P_{80}}} - \frac{10}{\sqrt{F_{80}}}\right)} \tag{1}
$$

$$
E = W_i \left(\frac{10}{\sqrt{P_{80}}} - \frac{10}{\sqrt{F_{80}}} \right)
$$
 (2)

The calculation was performed for a grinding time of 30 minutes and a grinding speed of 40 rpm in a ball mill, with the steel balls weighing 5 kg/ kg of sample.

The work index for the untreated sample was calculated to be 21.356 kWh/t and 15.11 kWh/t for the treated sample, resulting in a 29% reduction in the work index after MW treatment. Because the work index represents the energy expended in converting ore from its initial grain size to a specified reduced size²⁹, a decrease in the work index after MW treatment implies a corresponding decrease in grinding energy. Consequently, while the energy consumption for grinding untreated ore was 3.3 kWh/t, it was 3.1 kWh/t for the treated ore.

Additionally, the effect of MW treatment on dry grinding kinetics was investigated. For this analysis, the breakage of the materials was assumed to be of the first order in nature, and the specific grinding rates obtained for the untreated and treated samples are shown in Table 2.

The results show a 45% improvement in the specific grinding rate after MW treatment on the sample, and the effect of increasing the pretreatment duration on improving specific grinding rates can be clearly seen.

The results obtained are in concurrence with some of the reported literature: Prior to magnetic separation, MW pretreatment was used as a pretreatment method during the beneficiation of a low-grade banded iron ore sample collected from Southern India, enabling 85% iron recovery¹⁵. When compared to untreated ore, copper recovery from MW-treated South African carbonatite ore could be increased by 5 to 10%30. The microwave treatment increased the Handgrove index value of the lignite coal sample by about 23%31. Microwave pretreated high-ash Indian coal showed an increase in both the grinding rate and the rate of breakage, with a mean increase in the specific breakage rate of up to 15% when compared to untreated ore 24 . A similar observation was made for an Iranian iron ore sample, where the breakage rates were higher for the MW-treated samples than the

Table 2. Comparison of specific grinding rates for MW-treated and untreated ore

untreated samples, resulting in the lowest proportions of fines³². To avoid gold loss due to over-grinding of ore with conventional mineral processing practices, the ore sample was subjected to MW pretreatment prior to grinding, which not only reduced the Work Index by about 19%²⁶, but also improved gold recovery in the range of about 28% to 40%. A reduction in the grinding energy of the MW pretreated ore was also observed for high-phosphorous oolitic Egyptian iron ore 20 ; a comparison of the energy requirements of MW pretreated size reduction and size reduction with conventional heating pretreatment was conducted, and the reason for lower energy consumption in the case of prior MW pretreatment was attributed to intergranular fractures formed between hematite and gangue minerals³³. When the effect of modulated MW treatment on the breakage properties of copper ore was studied, the ore sample exposed to MW was found to be easier to break than the untreated sample³⁴.

3.4 Mineral Compositional Effects of US Treatment

In addition to determining the role of MW treatment in lowering grinding energies, the authors were also interested in determining if US treatment has any influence on upgrading the quality of ore under study. The changes in the morphological features and in the elemental composition upon US treatment are highlighted in the FESEM (Figure 6) and EDS (Figure

7) images, respectively. The elemental composition was analyzed using EDS, where the computation was done by imaging two different regions for the untreated and treated samples, respectively. The averaged elemental distribution over the two selected regions is displayed in Table 3. A clear and distinct increase in the atomic and mass percentage of iron is observed for the treated ore, i.e., for the MW-pretreated ore having subjected to successive US treatment and this demonstrates a positive influence of the combined treatment procedure on improving the iron content of ore. At the same time, the amounts of the gangue minerals, Al and Si are found to reduce by about 59% and 38% in their weight percentages, and 44% and 19% in their atomic percentages, respectively, following combined MW-US treatment.

These findings may be corroborated well with the one made when the two Indian ore samples (1:10 ratio pulp) were subjected to high-power ultrasound treatment¹⁸, wherein the iron content of the ore increased for 5 min US treatment, and the aluminum, silica, and phosphorous contents came down and was attributed to the implosion of cavitation bubbles between iron-gangue interfaces resulting in the disruption of bonds between them. A similar explanation may be given here as well. These observations were also accompanied by a reduction in the amount of oxygen and an increase in the amount of manganese, probably due to the reduction in the amounts of impurities.

Figure 6. FESEM images (**(A)** – untreated ore; **(B)** – MW treated ore subjected to US treatment).

Figure 7(a). EDS images of untreated ore at two different selected areas.

Figure 7(b). EDS images of MW+US treated ore at two different selected areas.

In order to draw further confirmation on the effect of MW-US combined treatment on ore composition, XRD analysis was performed and the plots are displayed

in Figure 8. Elemental analysis using EDS showed an improvement in the iron content and reduction in gangue minerals but to ascertain if this improvement is due to the

Figure 8. XRD images displaying the effects of different treatment types (**(A)** – untreated; **(B)** – MW treated; **(C)** – US treated; **(D)** – MW+ US treated).

effect of MW treatment or that of US, the sample treated with MW alone and US alone, respectively, was analyzed using XRD. The hematite content in the untreated ore was 26.1%; a slight increase of up to 27.1% was observed for US treatment (for 300 s), whereas for MW treatment (of the powdered sample form for 360 s), a reduction of up to 18.3%, and for the combined treatment, up to 22.6%, was observed. This means to say that while ultrasonication retains hematite, microwaves bring down its content slightly. On the other hand, while goethite content was 57.3% in the untreated ore, a percent increase was observed in the order of 62.2% for the combined treatment, 70.3% for US treatment, and 80.3% for MW treatment, suggesting that the penetration of US waves into the sample did not favour an increase in the content of goethite, while MW did. A significant increase of up to 23% was observed for MW treatment alone. Based on these results, it may be concluded that for the iron ore sample under study, for enhancement in goethite content, MW treatment may be adopted, whereas if hematite content has to be improved, then it is better to go with US treatment. If we look at the gangue minerals, the original quartz content of 16.6% in the untreated sample, drastically reduced to 2.6% with US treatment and to 1.3% with MW treatment, and interestingly, disappeared completely with the combined pretreatment approach, however, with a corresponding increase in the kaolinite content of up to 15.2%. On the whole, it appears that the combined treatment may not have to be followed for ore quality improvement; instead, MW treatment alone or US treatment alone may suffice, depending on the ore type, and the quality and level of upgrading required.

4.0 Conclusions

The FTIR, SEM, and XRD results show that the ore matrix disintegrates and crystallinity improves after MW treatment, which are the obvious reasons for the reduction in grinding energy. Because microwaves can be absorbed by many valuable minerals, similar results may be expected from a variety of ores. Microwave heating is advantageous over conventional heating because it eliminates the problem of waste heating. Accordingly, the extremely short treatment times compared to traditional heating methods make MW treatment more time-efficient, energy-efficient, and economically viable. Furthermore, MW treatment aids in the reduction of the wastage of valuable minerals by improving their liberation from ores. Along with thermal liberation properties, MW provides additional benefits such as reduced wear of the milling equipment. Implementing the following changes may result in further improvements in process economics: A domestic microwave oven was used in the current work. Grinding energies may be reduced further if a laboratoryscale microwave is used. Because the extent to which the sample is heated is dependent on the bulk density, it may also need to be changed. Increasing MW power and exposing ore samples to microwaves with high power densities for shorter residence times may produce better results, and this should be investigated. Some previous studies have also indicated an improvement in the magnetic properties of the ore after microwave treatment; in this regard, additional studies may be conducted to check these properties for the ore under consideration so that ore quality improvement can be beneficial.

To date, ultrasonic treatment has seen some success in upgrading ores. The combination approach containing microwave and ultrasonic treatment used in the current work proved to improve the overall iron content of the low-grade iron ore studied by helping in disintegrating and dislocating the gangue minerals. However, in upgrading the qualities of individual iron-containing minerals, such as hematite and goethite, individual treatments were better than the combined treatment method. To achieve even greater quality improvement, this combined approach may need to be further optimized.

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