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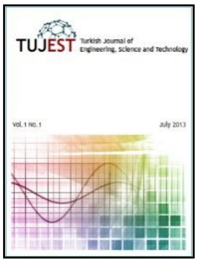

Prospects of Microwave Energy in Material and Mineral Processing

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Abstract

Microwave energy has gained worldwide acceptance as a novel method for heating, sintering and phase transformation of minerals and materials, as it offers specific advantages in terms of speed, energy / power efficiency, process simplicity, improved properties and produces the finer microstructures. This paper deals with a review on the advances in the microwave treatment of minerals. It shows the beginning of using the microwave energy as early stages of development for future processes for industries. Many different applications are considered, including fundamental heating rate studies on pure minerals, and its applications on ores, microwave assisted grinding, selective mineral liberation, possible exploitation in the area of extractive metallurgy, phase transformation, enhancement of magnetic and electrical separation, saving of energy, decomposition/ recycling of wastes. Conclusions are drawn on the need for further scope on fundamental and pilot investigations. This paper also shows that microwave energy is a clean and eco-friendly process for obtaining the value added products as compared to conventional method.

1. Introduction

Microwaves are a form of non-ionizing electromagnetic energy with associated electric and magnetic fields. It has a range varies from the infra red region of the spectrum to the short wave length region. The materials which couple with (absorb) the microwave radiation are termed dielectrics and contain dipoles. When the microwaves are applied to dielectric materials the dipoles align and flip around since the applied field is alternating. Subsequently, the material heats when stored internal energy is lost to friction.

Microwave ovens are designed and marketed for cooking of food and intended to supplement normal ovens. They had wide range of applications in mineral technology, metallurgy etc. In microwave cooking of food (organic matter) and microwave heating of object, aligning and flip flopping of molecules causes the materials get heated when stored internal energy is lost to friction.

2. History

The first US patent obtained for exposure of microwave heating to minerals was desulphurization of coal in 1978 by Zavitsanos [1]. It was not till 1984 that Chen et al [2] published the publications of the most pioneering paper related to relative transparency of minerals to microwave heat treatment. Since then , the applications of microwave heat treatment has been

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attempted and also reviewed by many researchers on different specialized fields such as desulphurization, selective heating, grinding, liberation [3,4]. In India, Indian Institute of Technology, Kharagpur was the first worked during early 1990's on coal with microwave energy [5]. Subsequently, the CSIR-IMMT, Bhubaneswar has reported the observations [6-8] on selective liberation and enhancement of grade from sulphide ore, bauxite and graphite ores. In the year 1995, the reduction of oxidized ilmenite was reported by Kelly and Rawson [9] to produce the metallic iron with leaching and microwave treatment. Subsequently, a few researchers reviewed on various specialized areas such as desulphurisation, selective grinding, liberation, leaching, reductions etc. During the year between 2011 and 2012, Srikant et. al in CSIR- IMMT found the wonderful technique for the mullite formation with the red sedimented placer sillimanite and most valuable product titania in form of slag from the red sedimented placer ilmenite with microwave treatment [10-11]. This paper deals with a review on prospect of microwave heat treatment in mineral and material technology and its application for various mineral industries.

Table 1. Minerals transparent to microwave heat treatment, microwave frequency 2.45 GHz, power 150 W, exposure 300 sec (Chen, et al, 1984)

Mineral Class	**Mineral / Compounds**
Carbonates	Aragonite, calcite, dolomite, siderite
Jarosite-type	Argentojarosite, zinc plant residues - synthetic natrojarosite / plumbojarosite
Silicates	Almandine, allanite, anorthite, gadolinite, zircon, muscovite, potassium feldspar, quartz, titanite,
Sulfates	Barite, gypsum
Others	Fergusonite, monazite, sphalerite (low-Fe), stibnite

2.1. Microwave Treatment In Minerals

Microwave heat treatment has wide potential application in the metallurgical industry although few research works has been published on the reactions of microwaves with minerals. The most important research work performed by Chen et al [2] who exposed forty minerals individually to microwave energy in order to understand their general behaviour and reported that the microwave heat treatment results can be divided into two groups; (i) very little or no heat is generated and the mineral properties are not affected because the mineral is transparent or totally reflective to microwave energy, (ii) heat is generated and the minerals will be either thermally stable or dissociate/ react rapidly. The results of his research work show that most silicates, carbonates, sulphates, some oxides and some sulphides, such as low iron sphalerite come under first group (Table 1) i.e they were transparent to microwave energy. However both synthetic and natural jarosite are not affected by microwave treatment. Metal oxides, haematite, cassiterite and magnetite fall under second group with thermally stable where as most sulfides, arsenides, sulfosalts and sulfoarsenides, and some oxides heated strongly, emitting fumes and fusing during microwave heating (Table 2) which also fall under second group.

Table 2. Results of microwave heat treatment on oxides and uranium minerals, microwave frequency 2.45 GHz, power 150 W, exposure 3-5 min (Chen, et al, 1984)

Mineral	**Power, W**	**Response**	**Product**
Allanite	>150	Does not heat	No change
Arsenopyrite	80	Heats, some sparking	S and As fumes; some fusion
Bomite	20	Heats readily	Some changed to bornite–chalcopyrite –digenite; some unchanged
Cassiterite	40	Heats readily	No change
Chalcopyrite	15	Heats readily with emission of sulfur fumes	Cu–Fe-sulfides or pyrite
Columbite	60	Difficult to heat when cold	Niobium minerals fused; most silicates not changed
Covellite / anilite (60% Vol)	100	Difficult to heat, emission of sulfur fumes	Sintered to single composition of $(Cu,Fe.)_9S_5$
Fergusonite	>150	Does not heat	No change
Galena	30	Heats readily with much arching	Sintered mass of galena
Hematite	50	Heats readily; arcing at high temperature	No change

Mineral	Power, W	Response	Product
Magnetite	30	Heats readily	No change
Monozite	>150	Does not heat	No change
Nickeline / cobaltite (3 Vol %)	100	Difficult to heat	Some fused; most unaffected
Pitchblende	50	Heats readily	Some fused, others unchanged
Pyrite	30	Heats readily with emission of sulfur fumes	Pyrrhotite and S fumes
Pyrrhotite	50	Heats readily with arcing at high temperature	Some fused; most unaffected
Sphalerite (High Fe)	100	Difficult to heat when cold	Converted to wurtzite
Sphalerite (Low Fe)	>100	Does not heat	No change
Stibnite	>100	Does not heat	No change
Tennantite	100	Difficult to heat when cold	Fused mass
Tetrahedrite	35	Heats readily	Fused mass of Ag–Sb alloy

The behaviour of minerals to microwave heating depends on their composition; for example, when Fe substitutes for Zn in sphalerite the resulting high iron sphalerite becomes microwave responsive. These test results indicate that microwave energy may find application in mineral treatment and metal recovery processes.

Walkiewicz et al [11-12] investigated microwave heating characteristics of various minerals and compounds quantitatively. The materials selected were irradiated in I kW, 2.45 GHz heater and the resulting temperatures and rates of heating determined are shown in Table 3. Thus it is confirmed that some minerals absorb microwave treatment readily and each mineral has selective heating rate of absorption while some others do not. In this entire investigation and careful studies, the problem of temperature measurement was overcome using a metal sheathed thermo-couple. The thermocouple was in constant contact with the sample.

McGill et al [13] discussed the effect of power level on mineral heating rate. Minerals similar to those utilised in previous studies were powdered and exposed to various microwave power levels ranging from 500 to 2000 W. All tests were performed with 25g samples for low density minerals.

It was observed that in general, an increase in power led to an increase in the heating rate of the mineral. However, this was only true, for the minerals which had previously demonstrated an affinity for microwave radiation. Low loss materials such as quartz and orthoclase did not heat effectively, regardless of the applied power.

2.2. Operating Microwave Energy In Extraction Of Value Added Products

Microwaves offer the possibility of enhancing the extraction / leaching of value added products, such as gold from auriferous ores [14-17]; enhancement of iron and titanium from ilmenite minerals and mullite from sillimanite minerals [18-19].

Haque investigated on extraction of gold from gold ores and observed that mineralogy plays an important role because some minerals absorbs microwave energy and may then react when hot with air or other substances, while other materials may be unaffected [16].

As a general observation, ore minerals respond favourably to microwave energy where as gangue minerals do not, and this fact could be advantageous in metallurgical processing although many practical problems remain to be solved yet. The various experiments performed by many researchers proved that Iron sulphides absorb microwave energy; heat rapidly and decompose quickly.

Table 3. Summary of Microwave heating results (Walkiewicz, 1988)

Mineral	Temperature Achieved, °C	Time, min
Chalcopyrite	920	1
Galena	956	7
Magnetite	1258	2.75
Orthoclase	67	6
Pyrite	1019	6.75
Quartz	79	7
Sphalerite	88	7

Roasting of gold ore in air, yielded more than eighty percent of the arsenic and sulphur were Volatilized as As2O3 and SO2, where as iron was oxidized to hematite [14-16]. Cyanidation yielded 98 % of the gold. In presence of inert gas nitrogen, the arsenic was converted to pyrrohotite (FeS). Cyanidation gave 89 % gold recovery. Microwave heat treatment of the concentrate with sodium hydroxide (NaOH) converted the arsenic and iron to soluble arsenates and ferrites and hence no Volatiles were produced. Ninety Nine percent recovery of gold was obtained by cyanide extraction of the leached process.

2.3. Microwave Energy On Mineral Properties

2.3.1. Surface area and grindability of minerals

Harrison and Rowson studied that on increasing the microwave power level; there is decrement of the work index. In the same study, the magnetic susceptibility of the magnetite ore was decreased after microwave heat treatment while magnetic susceptibility of the ilmenite increases [20]. It can be explained by the oxidation of magnetite to hematite which is less magnetic. In the case of ilmenite, the increase in temperature makes the alignment of the atoms easier giving rise to a more structured lattice. By irradiating various ores for five minutes, there were percentage increment in surface area and comparative work index.

Kingman et al [21] studied the grindability effect on the massive Norwegian ilmenite ore and the effect of varying the microwave power of the radiation. It was found that reduction in required grinding energy could be achieved.

2.3.2. Effect on particle size

The results from microwave heat treatment of minerals are always the particle size dependent. It was first investigated by Standish et al [22].

2.3.3. Effect on magnetic properties

The surface characteristics and magnetization of magnetic minerals alter after microwave radiation. It was first investigated by Florek et al [23] for chalcopyrite that the microwave heating increases the magnetic susceptibility of very weakly paramagnetic ore and enables its effective magnetic separation, which is impossible to achieve in its normal state and it will take longer time for conventional methods .

Kingman et al [24] investigated the effect of microwave heat treatment upon the mineralogy and magnetic processing of a massive Norwegian ilmenite ore. The results of his study indicate that short periods of exposure can cause fracture at grain boundaries, which leads to the formation of intergranular fractures. The fracture coupled with an increase in ilmenite mineral magnetization give rise to an increase in both concentrate grade and valuable mineral recovery. However, the study has also indicated that process efficiency can be affected with over exposure to microwave heat treatment.

Srikant et al [25] during his research work in beach sand and red sediment minerals with microwave energy observed that the characteristic relation between microwave power fed (Watt) and temperature absorbed (°C) for ilmenite ores with different magnetic strength indicates that the absorbance of temperature of ilmenite ores with different magnetic strength varied incrementally with the microwave input power fed (Watt).

2.4. Microwave Reduction Of Metal Oxides

Microwave reduction experiments were conducted on ilmenite and haematite minerals by few researchers. Microwave reduction process depends on the dielectric heating characteristics of materials. The investigation conducted by Standish et al [26] for microwave assisted carbothermic reduction of metal oxides concluded that if the metal oxide has a low loss factor i.e. poor receptor to microwave energy then adding carbon, which is a good receptor of microwave energy, plays the role of a microwave heating accelerator. The ilmenite are said to be very good dielectrics. However, once the ferrous iron is oxidized to ferric iron, its ability to form dipoles is lost and it will not heat without the use of reducing agent. Reducing agents are additives, which heat rapidly in presence of applied electric field. They conduct energy into the bulk sample so that electrons in non-dielectric materials can become more mobile. Due to this extra mobility, there is the formation of dipoles in the microwave field and the material can heat on its own. Carbon, conveniently, is a reducing agent and when mixed with fully oxidized ilmenite allows the mixture to dielectrically heat. At elevated temperatures many dielectrics couple to microwave energy to a greater extent [4]. This has the effect of creating the hotspots in samples and hence non-uniform temperature distributions. On these assumptions, Kelly and Rawson [9] considered the novel microwave reduction of pre-oxidised Ilmenite concentrates. The oxidation and reduction of iron in ilmenite concentrates between the ferric and ferrous states has been found to greatly increase its chemical activity. Two ilmenite concentrates were first oxidised in a conventional muffle furnace at 1000 °C. The ferric iron was then reduced back to ferrous iron by heating with a fine carbon powder under an inert nitrogen atmosphere. The reduction process was carried out in a variable power (0 – 1500 kW), 2.45 GHz microwave oven. Duplicate control samples were reduced at temperatures over the range 700-1000 °C. After reduction the effects of reduction on phase change, surface morphology and leachability were determined. It was shown that broadly similar results were obtained for both microwave treated and conventionally treated samples i.e. almost total reduction were achieved in a microwave oven over a very short period of time (typically 8 mins at 750 W compared to 8 hrs at 800 °C). The authors also suggested that the extraction of titanium apart from iron extraction from microwave reduced samples increased with the extent of reduction i.e. exposure time could be possible. Surface areas and thus porosities were also shown to increase with the extent of the reduction.

An attempt has been made on ferruginous bauxite with microwave heat energy by R Bhima Rao [7], for selective magnetization of haematite. The results of this important investigations revealed that a product containing 80% Al_2O_3 and 2.5% Fe_2O_3 with a recovery of 80% could be achieved from crude bauxite containing 55.7% Al_2O_3 and 5.6% Fe_2O_3 by a magnetizing roast in a microwave oven, followed by comminution and magnetic separation.

Microwave reduction of placer ilmenite concentrate experiment was investigated by Satya Sai Srikant et al [27] by using microwave domestic oven having IFB model 38SC1, 850 Watt. The results of the investigation indicated that at one minute of time the metallic iron formed contain 0.39% with no phase transformation. At three minutes of duration of time, a partial phase transformation of ilmenite was observed and the metallic iron contain is 1.65%. Interestingly at above nine minutes duration of time a distinct metallic iron phase containing 32% metallic iron is observed.

The limitation on use of domestic microwave oven is that the ilmenite sample must be heated with a lapse of every three minutes i.e after every three minutes of heating, the domestic microwave oven has to be switched off for a minute otherwise the microwave oven may damage because of metallic formation inside the microwave oven. Due to the limitations on the use of microwave energy with microwave domestic oven for complete reduction of ilmenite, the industrial microwave sintering furnace having model G N Technologies, 2.45 GHz, output 6 kW with steps 5W is used to achieve complete reduction of ilmenite for slag and metallic iron.

The results of initial experiments carried out on ilmenite for oxidation and reduction process with microwave furnace for 5 % and 7.5% of graphitic carbon as reducing agent and silicon carbide as susceptor, do not showing any significant change to form the titania products and metallic iron products. The optical microscopic observations for the reduced ilmenite samples with 5% and 7.5 % of graphitic carbon also did not shown any significant effect on phase transformation of minerals.

It was in the year 2012 only; Satya Sai Srikant et al [28] performed one important investigation with pre-oxidized ilmenite sample. The sample was heated in a microwave sintering furnace for 40 minutes in order to compare the heated sample from microwave oven. The authors found that the result of microwave heating on oxidized ilmenite with graphitic carbon in microwave sintering furnace produced titanium and titania phase as well as metallic iron phase from XRD analysis. The success of the process is explained by the fact that at higher microwave energy with increased exposure times, not only iron phase would be totally reduced with a faster conversion from Fe_2O_3 to Fe_3O_4 state but also a distinct titania phase were obtained in lesser time. This would produce the same results with conventionally reduced samples, however, with much more time.

2.5. Microwave Energy For Ceramics Industries

The mullite is a good, low cost refractory material widely used in ceramic industries because of the properties of high temperature strength, very high electrical resistivity, high thermal shock resistance, excellent thermal stability. The mullite is composed of large needle-like mullite crystals containing alumina. Also it has high resistance to most chemical attack, excellent stability in acid metal slags which are insoluble in most acids. Mullite not only provides the resistance to oxidation process and attack by furnace atmospheres but also provide the resistance to abrasion. Mullite is widely used in electronic packaging materials industry, refractory materials industries, heat resisting materials industries and heat exchangers as the engineering materials.

Generally mullite is occasionally found in nature, as its formation need high temperature and low pressure conditions. Artificial mullites can be made from kyanite, sillimanite and andalusite which are aluminium silicate minerals having the same chemical composition ($Al_2O_3.SiO_2$) but differing in physical properties. Thus the placer minerals which contain considerable amount of sillimanite ($Al_2O_3.SiO_2$) can be made use of mullite formation for applications in ceramics, low cost refractories and abrasives industries. The decomposition of sillimanite ($Al_2O_3.SiO_2$) with addition of Al_2O_3 and SiC to form to mullite may be expected as

$$2(Al_2O_3.SiO_2) + Al_2O_3 + SiC \rightarrow 3Al_2O_3.2SiO_2 + SiC$$

On heating the placer sillimanite between 1500°C and 1650°C with conventional heating process, it decomposes to mullite and silica [29] in such a way that their decompositions produce a mixture of the mullite 3:2 plus free silica.

Due to presence of rejected silica phase, the impurities become segregated at the grain boundaries. Use of conventional furnaces for preparation of mullite from sillimanite is universally known process. Several publications and patents are available in the literature on this aspect. Literature pertaining on the preparation of mullite using microwave heat source is very much restricted. The available literature is on other than sillimanite for formation of mullite from kaolinite or kaolin clay. The literature reveals that Elias and Ruth [30] observed the mullite phase in XRD analysis for the sample treated by microwave carbothermal reduction of kaolin. Ebadzadeh, et al [31] observed about mullite behaviour from the reaction sintering of clay and alumina heated in a microwave oven. They observed that after microwave processing, the minimum temperature required for the presence of mullite residual phase was 1250 °C.

However, Satya Sai Srikant et al [19], investigated the effect of heat treatment on placer sillimanite recovered from red sediment minerals for mullite formation in microwave sintering furnace, as all attempts have been made for mullite formation with conventional furnace on kaolin clay or fly ash using microwave oven only. The results of microwave power on mullite formation from sillimanite using SiC as susceptors are given in Table 4. It may be noted here the role of SiC in the microwave sintering furnace is as a heating agent because it couples quickly with electromagnetic radiation, creating heating owing to the Joule effect. The authors investigated that mullite is formed which was achieved at furnace temperature at 1384 °C and with a microwave power 1900 W having 10 % binder and the charged sample containing sillimanite (60%) and Al2O3 (40%) alongwith 60% SiC in the ratio of 3:2:3 respectively. The results from XRD analysis indicated that the mullite formation in both microwave furnace and conventional furnace were almost similar with reference to mullite phase concern. The results also evidenced that microwave energy is more efficient to produce mullite composite at 25 min than conventional furnace at 1300 °C / 3h duration.

Table 4. Mullite formation with microwave energy results from the mixture of sillimanite, alumina and SiC in the ratio of 3:2:3 with 10% binder (Satya Sai Srikant et al, 2012)

Mineral	**Power Fed, Watt**	**Temp, °C**	**Time, min**
Microwave	1900	1384	25
Conventional (Electric Furnace @ 5°C / min)	_	1300	180

The results from FESEM images for mullite formation from red sediment placer sillimanite using microwave heat treated clearly be seen that SiC in massive form and mullite in cluster structure, were distinctly observed in the morphological features of mullite formed from red sediment placer sillimanite using microwave furnace.

2.6. Microwave Energy For Coal

Various researches have been undergone for the effect of microwave radiation upon coal [1, 32]. The research activities identified are desulphurization of coal, drying of coal, heating rate investigations, and grindability studies. Desulphurization is an important process from environmental pollution point of view. Fuel gas cleaning processes have been installed in large scale combustion units. The removal of sulphur and ash for small scale units prior to combustion is probably more economical than fuel cleaning. The literature reveals a fact that under conventional methods, the heating of pyrite to obtain substantial high temperature to liberate the sulphur gases (100°C - 400°C) would result in simultaneous coal combustion. Whereas by using strong and powerful microwave energy (1kW – 6kW; 2450 MHz), the dielectric heating of pyrite present in the coal converts into pyrrhotite, the sulphur content is reduced and enhance the magnetic properties of paramagnetic pyrite which can be removed from coal by low gradient magnetic separation [32].

Rowson and Rice [34] and Hayashi et al [35] investigated the role of caustic leaching during the microwave desulphurisation of coals. Molten caustics (NaOH and KOH) were shown to be effective absorbers of microwave treatment and led to the accelerated differential heating of coal/pyrite phases. This was especially true in low pyrite grade coals where little heating would normally occur. Sixty percent reductions in total sulphur content are typically reported.

The production of pulverized coal feed for coal fired power stations is a highly energy intensive process, an estimated 4x109 kWh annual for the U.S alone. Harrison and Rowson demonstrated that a reduction of 30% in the comparative work index could be achieved by using microwave oven [650W, 2.45GHz]. The reduction in relative work index occurring because of cracking initiated around pyrite grains and superheating of water in the porous coal structure. It was observed that apart from reduction of work index, some additional desulphurization would occurred which further improved the economic and environmental benefits of the work [36].

2.7. Microwave Energy For Waste Management

All the industries adopting conventional process invariably generate waste material. To know the danger presented by the constituents of the waste, technologies are being investigated to minimize the waste generated and to provide safe handling, transportation, storage, destruction, removal or disposal of the hazardous waste. Currently, microwave energy is showing considerable potential in the management of a vast array of gaseous, liquid and solid wastes [37-38].

H2S is a very toxic gas produced during refining crude petroleum. Generally, hydrogen sulphide waste gas streams are treated by the Claus process, which is based on partial oxidation of hydrogen sulphide into sulphur and water. The Claus oxidation process requires a suitable oxidant mixture. The Kurchatov Institute in Russia, developed a process for the decomposition of hydrogen sulphide into hydrogen and sulphur by applying a microwave plasma (plasmatron).

The Argon National Laboratory, USA developed a process known as 'plasma-chemical waste treatment process' in which a hydrogen sulphide waste stream is passed through a microwave-generated plasma reactor where it decomposes into hydrogen and sulphur. The test results with this process can save the energy upto 40% with the decomposition of waste with this process in single pass only. More details of the process are available from Argon National Laboratory (Harkness, 1994).

Steel making slag usually contains 20 % iron. Hatton and Pickles [39] studied the laboratory scale microwave heating tests (1 kW, 2.45 GHz) with and without the addition of carbon or magnetite in order to investigate any modification occurred in the physical characteristics as well as to recover the iron from the steel making slag. The test results indicated that the coupling agent for addition of both carbon and magnetite increased the heating rate of the slag; 1000ºC with carbon, 800ºC with magnetite, compared to 650ºC without any addition. The amount of iron recovered increased with heating time and reached as high as 90 wt.%. Microwave heating altered the physical and chemical properties of the slag, saving the lots of waste generation.

3. Conclusions

A number of applications on the use of microwave technology are reviewed including fundamental heating rate investigations on pure minerals and its application on ores, microwave assisted grinding, selective mineral liberation, leaching/extraction, phase transformation, enhancement of magnetic and electrical separation, saving of energy, decomposition/ recycling of wastes. In general, these studies have been exploratory in nature and have also been performed on a laboratory scale; however, the results of the pilot scale tests mentioned reveal the encouraging results when microwave heat treatment process is compared to conventional process. To advance the application of microwave energy in the minerals industry, many more researches are to be required.

This review paper also shows that microwave energy is not only a clean process but also an eco-friendly process for obtaining the value added products. Also microwave energy has gained worldwide acceptance as a novel method for heating, sintering and phase transformation of minerals and materials, as it offers specific advantages in terms of speed, energy / power efficiency, process simplicity, improved properties and produces the finer microstructures. Although the quantity of research carded out in this field is limited the results published to date indicate that the future for applications of microwave energy within the mineral industries is encouraging.

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