

MICROWAVE ACTIVATED CRUSHING AND GRINDING OF TURKISH COALS AND SHALE FOR CLEANING AND DESULFURIZATION

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Abstract: The representative samples below 50mm are taken from the local coal area in Şırnak Province and other origin regions. As the coal samples and side rocks, Soma lignite, Tunçbilek lignite, Kütahya Gediz lignite, Şırnak asphaltite, Şırnak coal marl and coal shale were investigated. Heating with microwave of samples of 50 gr at time of 30-480 sec with 900W power in the Bosch laboratory type of Microwave Oven was carried out following roll crushing to minus 4mm and standard Bond Mill grinding. Microwave activated crushing and milling process was efficiently performed. Higher yields occurred about 52 % at 10-5 mm size fraction, 34% at grain size fraction of 3-1.5 mm, 12% at grain size of 0.5mm as grinding breakage. Microwave activated milling was also provided effective grinding with an increase in the Hardgrove Index about 43% for Şırnak asphaltite. Microwave activated milling also provided effective cleaning with a reduction of total sulfur content at 56% in the flotation and about 33% ash reduction in clean coal. As a result, the optimum microwave heat treatment process for coal cleaning plants was defined by making effective use in crushing, grinding and flotation, and the results are discussed.

Keywords: Microwave activation; Crushing, Grinding, Coal cleaning, Desulfurization, Asphaltite.

INTRODUCTION

Clean coal production and coal ash use is the main goal among various industries under ecological concerns (TKI 2009). High quality coal production so wished for the coal market in Turkey, deashing and desulfurization of coal needs low cost technological equipments and innovative techniques. By the coal mine production, the run-off-mine coal piles in the stockpiles field are dissociated or oxidized under atmospheric conditions during time passing months. In Southeast Anatolian Region, Şırnak coal production facilities located need the high amount of clean coal (Tosun a 2014). The microwave acted grinding of coal reduces the cost of grinding, produces clean coal. The effective alignment of microwave action in high capacity coal grinding plant will provide also the much desulfurization in coal flotation (Wheelock 1979, Yoon 1991). The coal and side rock shale and marl samples were economically heated by microwave oven with side rock stone in the porous structure of the coal and the developing of internal stresses in porous coal provided the much selective thermal crack formation, much possible following cleaning and desulfurization under 10mm size(Tosun b 2014).

Clean coal technology includes the crushing-grinding, screening, washing and flotation unit following a very fine 90 micron and 40 micron grinding is performed (Tosun, 2014). The design of the crushing-grinding and screening plant,

crusher and grinder selection is important for the ash reduction costs. Coal and side rock shale, which have the soft rock hardness. Also trace ash materials, marl, pyrite and limestone are needed to liberate with grinding prior to flotation. If the coal impurity pyrite minerals need to be separated with flotation at finer size, coal cleaning can be achieved by weathering and oxidizing effect of pyrite surfaces prior to flotation (Reimer&Franke 1991). Silica, silicate grains are difficult to grind. This cleaning method costs, negatively affect the quality and capacity. Coal properties are also important for mechanical breakage and pyrite surface type.

This type of microwave treatment may be advantageous in internal selective fracturing coal grains and provide selective surface oxidation of pyrite for coal desulfurization.

Microwave energy with frequency non-ionizing electromagnetic radiation in the range of 300 MHz to 300 GHz stopped. Include tri-band microwave frequency (UHF: 300 MHz to 3 GHz), super high frequency (SHF: 3 GHz and 30 GHz) and extremely high frequency (EHF 30 GHz to 300 GHz) (Walkiewicz et al. 1991). The microwaves penetrate the depth of the sample is in the form of electromagnetic energy increases the temperature of the sample volume, unlike conventional heating (Haque 1999, Gabriel et al., 1998).

Mineral leaching microwave thermal effect melting of the microwave roasting and sulfide

concentrates, refractory gold concentrates oxidation, activated carbon regeneration can be accomplished (Kelly & Rowson 1995, Vanwyk et al. 1998, Xie and Pickles 2000). Also be affected by microwave heating effect of calcinations of limestone rock (Sword, 2009).

Mineral grained rocks with different thermal expansion can take place easier on the texture microwave interaction (Jacob et al., 1995, Chen et al., 1984). Thus, the liberalization of fine ore and grinding View full size in energy costs could be reduced (Kingman et al., 1999, Marland et al., 2000, Veasey and Fitzgibbon, 1998). Microwave grinding technology to break the interaction was determined to be applicable successfully (Walkiewicz et al. 1991). Microwave interaction parameters of rocks, microwave penetration, expansion of different mineral grains, grain boundary cracking properties were examined. The temperature varies according to the thermal effects of microwave on mineral species (Walkiewics et al. 1988). Table 1 had the higher penetration of mineral grains of pyrite as given and has lead 1019°C like a temperature change.

Table 1, Mineral Microwave Thermal Interaction Values (Walkiewics et al., 1988)

Mineral	Maximum Temperature, °C	Time, min
Albite	69	7
Chromite	155	7
Chalcopyrite	920	1
Zinabar	144	8,5
Galenite	956	7
Hematite	182	7
Magnetite	1258	2,75
Marble	74	4,25
Molybdenite	192	7
Orthoclase	67	7
Pyrite	1019	6,75
Pyrotine	586	1,75
Quartz	79	7
Sphalerite	88	7
Zircon	52	7

Pore structure of the coal side rock, shale texture, contained pyrite at grain size in microns and determined the resistance to intergranular interaction with microwave. The grindability of these improved crack texture of side rocks and coal were examined with Bond grinding and HGI Standard test.

Microwave heating of coal samples containing pyrite of minerals may provide the inner thermal stress and interfacial cracks. In this work, following microwave treatment coals and the side rocks, marl and shale were ground to a size of minus 10mm to 200µm and especially, the effect of microwave radiation on grindability of those samples was investigated. Limestone

Soma lignite, Şırnak asphaltite, marl from Şırnak Coal Mine, shale taken from coal mine waste dumps were tested. Following 900W microwave radiation at 30-480 sec. in Bosch type microwave laboratory equipment, standard Bond and Hardgrove grindability were tested. The representative samples at each 10-5mm and 5-3mm fractions of Şırnak asphaltite, shale and coal marl bond work index increased to 23%, 21% and 12%, respectively and HGI raised to 46%, 24% and 14%. Bond work index of porous coal did not change. The optimum crushing and grinding method for clean coal production was discussed and designed.

The Standard Bond Grindability Test

The most widely known measure of grindability is Bond Work Index which was defined as the resistance of the material to grinding and quantified the specific work input (kWh/t) required to reduce the material from theoretically infinite size to 80% passing 0.106 mm (Bond 1952). The work index is subject to variations due to variations in the inherent properties of minerals and rocks, variations in the grinding environment and variations in the mechanism of energy transfer from the grinding equipment to its charge (Berry&Bruce 1966, Şahan 2012, Gökçen et al. 2012).

The standard Bond grindability test is a closed-cycle dry grinding and screening process, which is carried out until steady state condition is obtained (Toroman 2012, Gökçen et al. 2012, Şener 2012, Hosseinzadeh 2012, Eyüpoğlu 2012, Genç 2012).

In the design of grinding circuits in a mineral processing plant, the Bond method is widely used for a particular material in dimensioning mills, determining power needs and in the evaluation of performance. Its use as an industrial standard is very common, providing satisfactory results in all industrial applications. Despite having many advantages, this method has some drawbacks, such as being tedious and time consuming, and also requiring a special mill. Due to these difficulties, a number of easier and faster methods have been developed to determine the Bond work index (Armstrong 1986, Austin and Brame 1983, Şahan et al. 2012, Çayırılı et al. 2012).

Hardgrove Index Method

The HGI tests on parent coals and their binary and ternary blends were carried out using the standard Hardgrove Index method. For a comparative study, four standard reference

coals were also used. Gross representative samples, weighing 5 kg, were collected by engineers for each coal seam with 50 increments taken following the ASTM D 409-08 procedure. A representative sample was obtained using the coning and quartering technique following the ASTM D 346-04 method.

For the determination of the HGI values, each gross sample was crushed to 4.75mm (4 Mesh) in an impact crusher. The crushed sample was split into smaller lots of 250 g, using a standard method of sampling (ASTM D-2013-07). Samples each weighing 250 g was dried in an air-drying oven at 80°C for three hours until a constant weight loss was observed (ASTM D-3302-07a). Each air-dried sample of 4.75mm in size was placed on a 1.18mm (No. 16) sieve nested with a 600 mm (No. 30) sieve. The 1.18mm (No. 16) fraction was ground in a laboratory grinding mill until the whole material passed through the 1,18 mm (No. 16) sieve, followed by sieving through the 600 mm sieve (No. 30). Sieving of each sample was performed by a mechanical sieving machine for five minutes. The sieving procedure was followed by the sieving method (ASTM D293-93, 2004). The 0,600 mm (No. 30) fraction was discarded and the 0,600 mm fraction was saved for determination by the Hardgrove Grindability Indices by an HGI machine.

MATERIALS AND METHODS

Chemical Analysis and Polished Sections Study

Standard chemical and mineralogical compositions of the samples were determined by XRF. Fe, silica, Ca, and Mg were determined by means of chemical analysis. Samples firstly crushed in roll crusher from 40 mm to 10mm ground below 0.1mm in size and homogenized by grinding in mortar. as a representative powder samples of 5gr were dissolved with HF for silica content in platinum crucibles. The chemical compositions of Şirnak coal marl and shale is given in Table 2. The coal samples used in the experiments were analyzed by ASTM. The proximate analysis of Turkish lignites and Şirnak asphaltite is given in Table 3.

Polarizing microscopy (Zeiss) with the progress of micro-cracks pulling on photography, porous coal, shale texture, marl shows a heterogeneous texture (Figure 1).

Table 2, The values of chemical analysis of coal marl and shale in Şirnak Province

Component%	Şirnak Marl	Şirnak Shale
SiO ₂	24,14	48,53
Al ₂ O ₃	12,61	24,61
Fe ₂ O ₃	7,34	7,59
CaO	29,18	9,48
MgO	4,68	3,28
K ₂ O	3,32	2,51
Na ₂ O	1,11	0,35
Ign.Loss	21,43	3,09
SO ₃	0,20	0,32

Table 3, Proximate Analysis of Coal and Chemical composition of shale samples used in experiments.

Samples	Fixed Carb,%	Volatile Mat.%	Ash, %	Total S,%	Calor.V alue,kc al/kg
Gediz Lignite	34.55	25.6	25.5	4.5	3207
Şirnak Asphaltite	50.28	45.5	10.4	6.7	7820
Soma Coal	62.8	5.5	14.1	2.0	4820
Tunçbilek Coal	5.42	54.8	17.1	3.2	4560

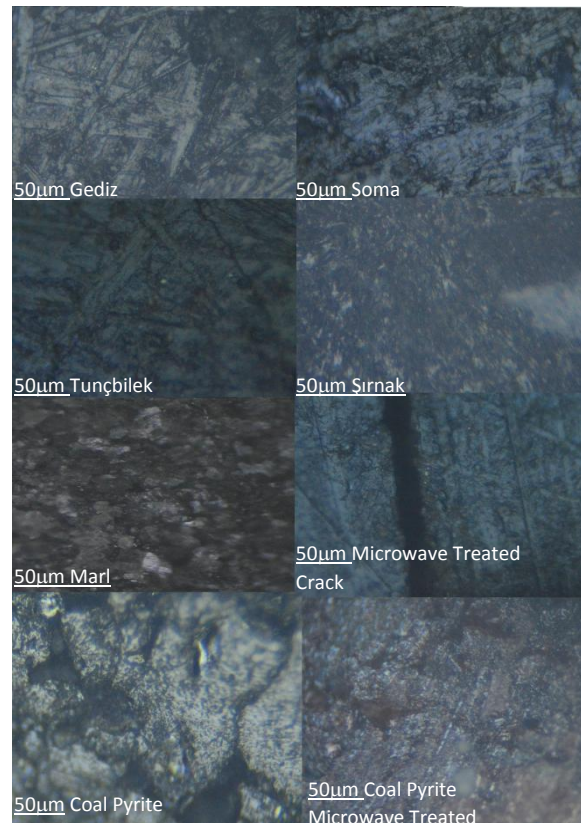


Figure 1, The Lignites, Şirnak marl, coal and Shale with micro and polished Sections 2min effect Microwave, Micro limestone marl, shale cracks Photography

The effect of the 2 min. microwave treatment was examined after the fracture and compared with the original as illustrated in Figure 1.

Particle size analysis

Kütahya Gediz lignite, Soma lignite, Tunçbilek lignite and Şırnak asphaltite, coal marl and shale were subjected to two-stage crushing in roll mill and to grinding below 4 mm. 10mm sized samples classified and were ready to microwave interaction test for different size fractions produced.

About 1 to 0.5 kg representative samples were sieved for a 20 min with standard sieve series. Weight distribution of collected following each sieving according to ASTM standard $\sqrt{2}$ series for each sample was determined by Gaudin-Schumann approach. Gaudin-Schumann approach for coal grain size distribution was found as much regressed for whole samples. According to this test, it was observed that approach provides a particle size distribution as illustrated in Figure 2.

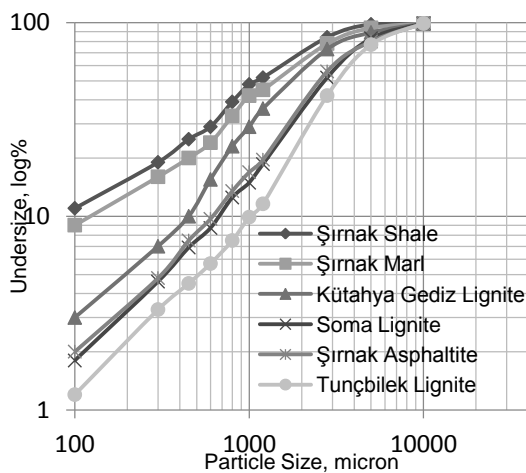


Figure 2, Coal, marl and shale samples of Gaudin Schumann Particle Size Distribution.

Fracture Rate, Standard and HG Bond Work Index Test

Microwave thermal effects of limestone, marl, has developed cracks in the shale layer of heterogeneous tissue and create fractures among the very fine particles. Grindability energy value was measured by grinding after the original sample, and microwave heating.

200 μ m under laboratory determined depending on the milling time and the amount 200 μ m six dimensions breakage rate was determined for each fraction of the original rock and microwave interactive fractions.

Standard Bond Work grindability index is calculated as below 200 μ m for each rock. Calculate G grindability values were determined by placing the Bond Equation. The grindability characteristics of the samples used in the tests is defined in Table 4.

HGI standard test device was used in order to determine the weight of under 75 μ m after 1min. grinding, so HGI value is determined in the amount and scale (Table 4).

Table 4, Coals, marl and shale samples of Bond and Bond Work Index HGI Grindability values.

Rock	Wi (Kwh/t)	HGI	Density (g/cm ³)	Porosit y %	Water Absor pt%
Şırnak Asphaltite	5.1	87	1.63	0,4	0,2
Tunçbilek Coal	6.9	52	1,56	11,7	9,9
Soma Coal	7,7	63	1,65	15,1	8,6
Şırnak Marl	6.7	47	2.64	2,7	1,9
Şırnak Shale	5.6	65	2.41	4,6	3,5

Interaction of Microwave Radiation on Coal samples

Samples of each coal types and marl and shale were treated in microwave radiation at ranging 30-480 sec with a laboratory type microwave of 900W power. A change at temperature was measured by thermocouples. As seen in the Figure 3, the explosion of shale sample was observed and the temperature was determined to be 600°C. In order to evaluate the impact of shale on five different size fractions of the treatment with microwave and heated for 3 minutes at 500°C for Şırnak shale samples. Bond breakage rate was determined as the ground weight % under 200 μ m by time (Figure 4). Microwave at 1, 2 and 3 minutes radiated samples were subjected to the HGI test.

HG index values in the microwave interaction as given in Figure 5 below the coal shale and Şırnak coal marl from 1.2 to 0.6 mm size fraction was determined for flotation.

-200 μ m Breakage rate values as given below in Figure 6 Turkish lignites, Sırnak asphaltite and marl, shale is determined for the fraction of 10-5mm size. 3min microwave interactions were found as highest effect on this fraction. Following microwave interaction breakage rate values increased at about 22% to 41% for Turkish lignites and 44% for Şırnak asphaltite as shown in Figure 7.

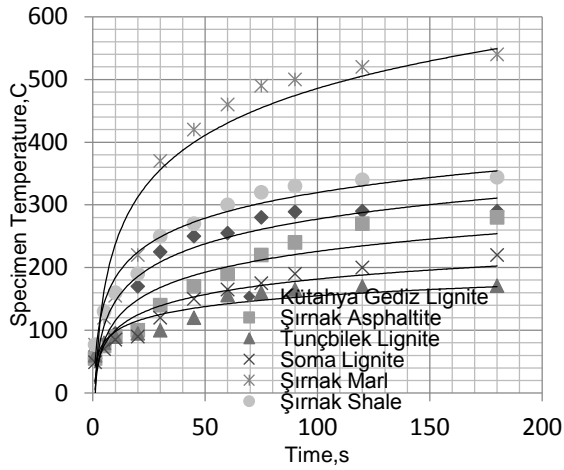


Figure 3, The temperature changes of Turkish lignites and Şırnak asphaltite, marl and shale followed microwave interaction

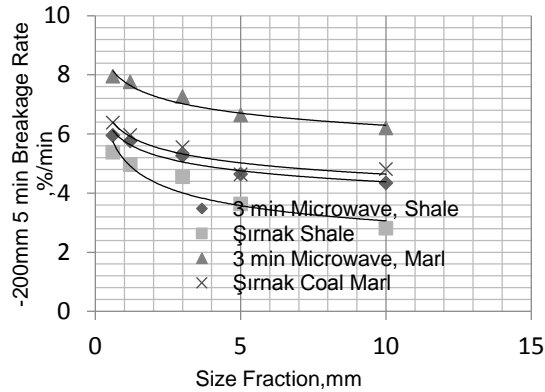


Figure 4, The rate of fracture shale 200µm under Microwave Interaction

HGI and Bond index values are given in the following Table 4 after microwave interaction.

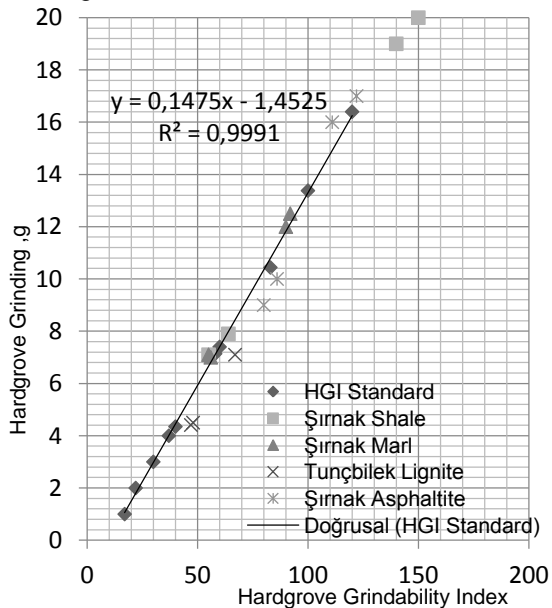


Figure 5, HGI Values of Original and 3 min Microwave interacted samples.

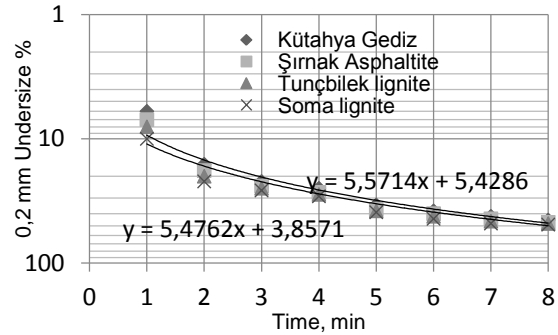


Figure 6, Breakage rate values to -200µm of samples.

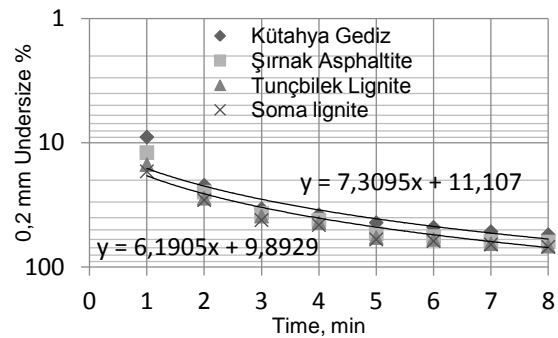


Figure 7, Brekage rate values to -200µm of 3 min. microwave interacted samples.

Flotation Experiments

-200µm 3 min microwave treated samples floated in 1500 ml cell with optimized collector (Kerosene) and frother (Ethyl hexane) dosages of 150ml/ton and 50ml/ton coal following 3min conditioning. Ash and desulfurization values for the different coal type as illustrated below in Figure 8. Also combustible yield values for the coals are shown in Figure 9.

RESULTS AND DISCUSSION

Bond grindability index and HGI were determined as 5.1 and 62 for Shale as given in Table 4. Şırnak asphaltite was found to be quite soft. Şırnak coal marl showed the hardest bond index at 10.4 and the Bond work index of Turkish lignites were changed from 8.1 to 8.4 identified as medium soft coals.

Şırnak marl showed a heterogenous texture with mineral content of sphalerite, pyrite and magnetite. Hence, following the microwave interaction at small size fraction of 1-2mm there was no like to be effective fracturing in rock grains due to elastoplastic feature (Figure 2).

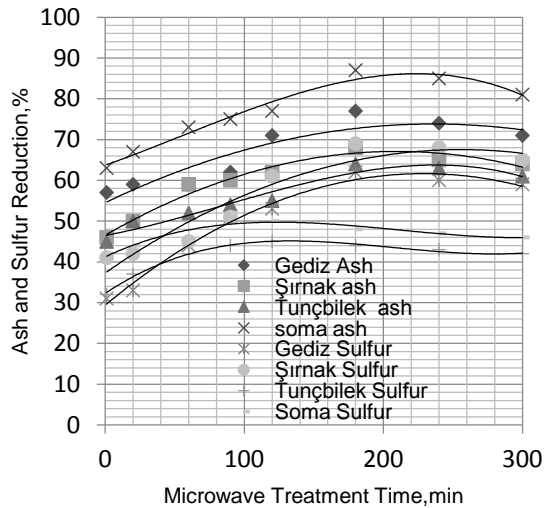


Figure 8, Ash and Sulfur Reduction Change of the microwave interacted samples.

At higher size fractions of 2-5 and 5-10 mm for Şırnak asphaltite, marl and shale low porosity caused high thermal expansion resulted by the formation of cracks. As illustrated in Figure 2 the cracks were observed on the line of reddish oxidized pyrite blowing and so swelling in the grain. Particularly, explosion effect of exposed to microwave of Şırnak shale and asphaltite increased 44% and 41% HGI grindability, respectively and breakage rate increased to 22 and 27%, respectively (Figure 7). Due to the volumetric thermal effect of microwave interaction on the coal grains containing pyrite the cracks developed in micro pyrite grain surfaces at higher levels. It was even determined that on the corners of particles microwave was greatly effective by expansion on the corners of coarse size of 10mm and over (Figure 4).

Another reason for this is that the lignite texture as maceral groups was more heterogeneous and even lignite particles in the coarse fractions were more mesoporous and microporous texture in the particles. Microwave interaction with non porous and homogeneous texture was observed to be much effective and it is seen that especially over heated pyrite micro grains in the coal changed much of the grindability of coal by providing high heat conduction.

Microwave exposure time with microwave power of 900 W, as shown in Figure 3, was sufficient as long at 1 and 1.5 min and showed a significant amount of interaction on grinding. About 5% moisture content of lignites, particularly the amount of water absorption ranged from 7-9% and in the Tunçbilek and Gediz lignite sample was observed to drying effect due to the evaporation of moisture in the lignite on exposure time of 1 minute and depending on pyrite content.

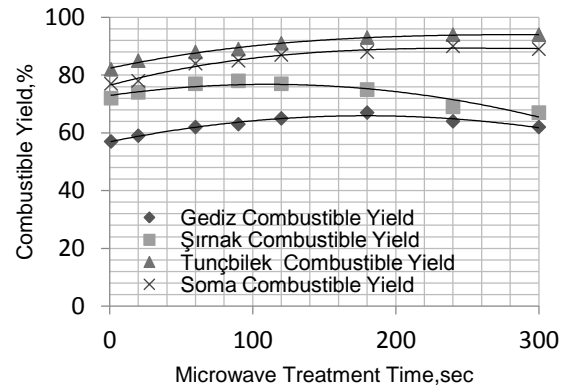


Figure 9, Combustible Yield Change of the microwave interacted coal samples.

Microwave power of 900 W with 60 sec to when raised, may develop due to thermal expansion of adequate fracture proved that 6% pyrite distribution in the asphaltite and shale and hard Turkish lignite was changed the temperature from 210°C to 670°C. The temperature in moist lignite samples were changed by drying effect, but porous texture avoided high temperature increase at short time. The evaporation of water absorbed in the pores of the sample was observed to release easily without causing fractures. Even the porous of lignite was not showed an advantage of the resulted crack formation over thermal expansion. Considering the ideal time to increase the interaction of microwave energy costs are defined as the interaction time 2min. The volumetric heating effect with a microwave interaction so that temperature rise over 300°C was observed which would lead to lignite such as combustion and even pyrolysis. Due to magnetite and pyrite in the Şırnak coal marl and Şırnak coal shale texture microwave decreased the Bond work index. The Şırnak coal marl and shale showed sufficient fracturing providing coming off oxidized pyrite surfaces prior to flotation units and HGI value increased from 52 to 83 and The breakage rate of 3.42%/min increased to 5.1. 900 W microwave exposure of 120 seconds had raised more than 7-8 parallel to the surface is flat intergranular fracture occurred in the Şırnak shale and marl. Shale is continued even after the effect of the explosion pose of 2 min duration.

There is a difference in the form of fractures created by the microwave. Surface cracks are generated more frequently. Especially brittle fracture type was seen as several cracks for marl rock below size of 10 mm in the flat prismatic shape with l/d ratio of 2-3. However, it is produced the fractures in large and deep longitudinal parallel to surface. Volumetric heating temperature is in the form of electromagnetic energy of microwave heating was higher than in the depths of coal. Therefore,

the heating of dust grains were minimal. Microwave interaction especially causing the central fractures (even as popping) significantly took place at the particles of 10-50mm size.

Tunçbilek and Soma lignite in particular following 3 min of the microwave interaction, showed efficient flotation manner by oxidized pyrite, and ash minerals such as radiation-absorbing mineral also been active during the microwave dielectric heating. 4-5% weight of pyrite in the lignite and Şırnak asphaltite in flotation tests sulfur reduction found to be increased to about 42%,65% from 29%,41% , respectively. The ash reduction in clean floating Tunçbilek lignite and Şırnak asphaltite ranged from 41% to 64%, the more microwave treatment affects on grinding even in the lignite or other type of coals one clean flotation coal product caused by dried activated carbon surfaces and oxidized reddish pyrite surfaces improved flotation ash reduction and sulfur reduction results. Even glassy silicate grains are exposed to less expansion microwave interaction, but high ash reduction might be caused by observed coagulation with electrical activation of silica particles under microwave heat radiation. Microwave interaction even finer size of 200µm of Şırnak asphaltite and Turkish lignites which containing about 4% pyrite content combustible yields in clean coal products remained low due to high ash contents and slime issue resulting cover over floatable coal particles. The low combustible yield was also the case even with high pore content of lignite and at the surface causing hydrophilic water or slime texture over porous lignite particles at even finer size under 200µm.

CONCLUSIONS

Grinding energy in the coarse grinding, especially in the range of 15 mm to 1 mm grinding to be done, was significantly decreased after microwave interaction. The conventional energy consumption without interaction in coarse grinding was about 2-3 kwh/t coal grinding as compared to Microwave interaction energy. Microwave interaction energy consumption was very low levels of energy and in coarse grinding was about up to 0.02 kWh/t coals. Therefore, the use of microwave radiation for drying and activation coal surfaces and even in the pyrolysis would be very beneficial, especially for the coals containing pyrite over 4%.

Due to the large size of the sample volume due to the increase in temperature and volumetric thermal stress changes more superficial crack

formation was observed. Bond work index of for 3 minutes microwave interaction with fallen 11-12% and 23% limestone and marl and shale HGI values were determined as the higher of 24% and 44%.

The porous lignites for the increase in the surface area although interaction affect of microwave radiation on lignite samples showed reduced heat conduction due to the air gaps between the particles and contact particle surface. The temperature in contact with the pyrite in microwave oven self combustion might be observed if uncovered to oxidizing atmosphere deteriorating flotation manner.

Because Şırnak asphaltite showed high bituminous texture and soft homogeneous pyrobituminous structure changed much with microwave affect over 2 min duration. But Şırnak shale and marl containing not bituminous textures, including iron minerals pyrites and magnetite showed considerable a high grinding with microwave interaction increase causing exploiting and popping even -10mm size.

Increasing microwave radiation provided a more effective flotation for coal cleaning and desulfurization following cracks in the thermal expansion, coming off fresh dried, more hydrophobic active coal surfaces and oxidized reddish hematite-pyrite surfaces. This fact improved easier grinding and combined with coal cleaning by effective flotation.

The ash and sulfur reduction in conventional flotation has increased by 22%, 19% for Şırnak asphaltite, 17%, 14% for Gediz lignite 19%,13% for Tunçbilek lignite17%,11% for Soma lignite, respectively.

Microwave interaction time of 2 min the sample was sufficient heat conduction and thermal expansion explosion may be at 1min exposure while at the same time and shale, high shale coal samples.

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