Effect of Microwave Heating on Vermiculite from Limpopo, South Africa

Antoine F. Mulaba – Bafubiandi[1,2,*], Boitumelo Seemise[1]

***Abstract*—**Vermiculite, as an industrial mineral, is often conventionally heated and used in packaging. It has the interesting property of expanding up to thirty times the original size when subjected to heat. This paper discusses the possible effects of microwaves heating on this alteration product of mica minerals such as biotite and phlogopite. The vermiculite materials used in this paper originated from Palabora Mining Company (PMC) in Limpopo province, South Africa.. Characterized with X-ray powder diffraction (XRD) for identification of mineral phases and Fourier Transform Infrared Spectroscopy for identification of different functional groups present in the sample, vermiculite was subsequently subjected to a multimode microwave oven heating at different power levels and durations. For comparison purposes, microwaves heating effect was discussed in the light of possible observed effect from conventional heating in a furnace at different temperatures and time intervals. During microwaves heating, X-ray diffraction showed a decrease in the d-spacing as the power was increased at low time intervals. For conventional heating at temperatures above 700°C, the d-spacing of the highest peak decreased as the time increased. FTIR analyses revealed a shift of the O-H stretching band for both microwaves and conventional heating. It was observed that the conventional heating at higher temperatures led to the reduction of bands in the O-H bending and Si-O regions.

***Keywords*—**Vermiculite, microwave heating, conventional heating, exfoliation.

I. Introduction

Vermiculite is a yellowish brown alumino silicate mineral with a generalized chemical formula $(Mg,Ca,K,Fe_2)_3(Si,AL,Fe_3)_4O_{10}(OH)_2O\ 4H_2O$ [1]. It forms from the hydrothermal alteration of mica minerals such as biotite and phlogotite. This trioctahedral mineral has a crystal structure of 2:1 i.e. two tetrahedral layers for every octahedral layer and expands up to thirty times the original size when subjected to conventional heat [2] (Brown, 1961). Vermiculite metallurgical processing includes milling, wet screening, gravity concentration, drying and exfoliation.

The vermiculite applications include being used as a shock absorber in packaging and molten metal insulation. It can also be used for seed germination. Exfoliated vermiculite is used for manufacturing of gypsum plaster and cementitious spray fireproofing. Aiming to investigate the possible effect(s) of microwaves on vermiculite mineral, this paper discusses the laboratory bench work as microwaves heating of vermiculite is conducted at varying time intervals and power levels. It also includes conventional heating at different durations and temperature levels.

II. Background

[3] Studied the mineral thermochemistry of bentonite and kaolin related to their possible application to the ceramic industry. Bentonite and Kaolin were fired at 950˚C and 1250˚C and XRPD analyses were then conducted on raw and fired samples to identify and study mineral phases. They then concluded that even though clay minerals are similar, their end products are not necessarily the same when fired at different temperature intervals due to their raw clay chemical and mineralogical composition.

When [4] conducted their investigation into the use of expanded agents such as vermiculite and perlite to minimize corrosion in flue gas systems. With the combination of the expanded vermiculite bulking agent, it was observed that good results were obtained using only 15 Kg./Hr. of the MgO, and 5 Kg./Hr. of expanded vermiculite, a total of 20 Kg./Hr. for the combination, compared to 40 Kg./Hr. when using only the MgO, a reduction of 50% of the magnesium oxide, and with greatly improved cleanliness of the metal surface when both additives were used in combination. In [5] it was revealed that the various raw vermiculite grades exhibited a rapid instantaneous adsorption of hexavalent Cr. Vermiculite grade 2 topped the list with 97.4 removal of Cr from 250mg/L of the equilibrium solution. When the effect of pH was assessed, it was noticed that in all grades of vermiculite, the pH of the equilibrium solution decreased as a result of the removal of Cr from the solution.

[6], reported that clay minerals have a common structure and that this is due to chemical composition and thermal variations in the range of near surface conditions. Further investigation by [6] indicated that maximum temperature range at which clay minerals occur can be given as 4-250˚C and that at higher temperatures clays are considered to be of

Antoine F. Mulaba-Bafubiandi is with the Mineral Processing and Technology Research Center, Department of Metallurgy, School of Mining Metallurgy and Chemical Engineering, Faculty of Engineering and The Built Environment, University of Johannesburg, POBox 17011 Doornfontein, Johannesburg, 2028, South Africa, He is also with the University of Mbuji-Mayi, Faculty of Applied Sciences, Mbuji-Mayi, POBox 225, Mbuji-Mayi, Kasai, Democratic Republic of Congo

Boitumelo Seemise was a B-Tech degree student at the .Mineral Processing and Technology Research Center, Department of Metallurgy, School of Mining Metallurgy and Chemical Engineering, Faculty of Engineering and The Built Environment, University of Johannesburg under the supervision of Antoine F. Mulaba-Bafubiandi..

metamorphic origin.

According to the investigation that was conducted by [7], the diffraction pattern of Mg-vermiculite looked more or less the same whether analyzed dry or after glycol solvation retaining its original d-spacing of 14.5Å. Smectite, unlike vermiculite expanded and produced a first order peak at about 17.7 Å after treatment with glycol.

[8] reported that the d-spacing of Mg-vermiculite structure changes from 14.36Å to 14.81Å when placed in water for over several days. The swelling was regular throughout the crystal and the increase in the interlayer water content was observed. Some clay minerals such as montmorillonite expand up to 18 Å when immersed in water.

[9] a molded article of vermiculite can be manufactured by radiating raw vermiculite with electromagnetic waves in the presence of urea or thio-urea. It was noticed that vermiculite expand when radiated with electromagnetic waves and the duration ranges from a fraction of a second to thirty minutes.

Investigation conducted by [10] revealed that treatment of montmorillonite with ethylene glycol produced a two layer interlamellar complex at d (0.01)= 1.71nm. It was then suggested that this method had the potential to be extended to measure the surface area of other clay minerals for which retention was restricted to external surfaces only.

III. MATERIALS AND METHODS

The mineralogical study of the material was conducted using X-Ray Diffraction (XRD) for determination of mineral composition and Fourier Transform Infrared spectroscopy (FTIR) for identification of chemical or functional bonds. Two full scoops were sampled from 1 ton of vermiculite ore sample. An approximate mass of 6g was then collected from a two full scoops’ sample and exposed to microwaves. The sample was heated at 1000, 800, 600, 400, and 200W for 3 min, 2 min 30s, 2 min, 1 min 30s, 1 min and 40s. After heating the temperature was measured using IR temperature measure by targeting a particular grain. During conventional heating, temperature and time were the two main parameters. An approximate mass of 15g was collected and placed in a metal plate which was preheated prior to heating at 300°C, 500°C, 800°C,900°C, 1000°Cand 1100°C for 5s, 3 min and 15 min. The samples from microwaves were milled using a coffee grinder to get them to the required size for XRD analysis. The samples were then mixed thoroughly and a mass of approximately 1.600g was measured and back loaded onto the sample holder. XRD analysis was performed on Phillips powder diffractometer and Copper Kα anode (40 KV – 40 mA) was used. Solid samples were milled with potassium bromide (KBr) to form a very fine powder. A clay mixture of less than 1mg in several hundreds milligrams of KBr was prepared and subjected to a very high pressure of between 6000 – 7000 psi to vitrify the KBr and leave the clay as a crystalline material. The IR spectra were produced by passing a multi-wavelength infrared beam through a finely dispersed sample pellet.

IV. FINDINGS AND DISCUSSION

Varying the microwave effective power from 20 % to 100 % and exposure time from 40 s to 3 minutes , table 1, the observed surface temperature varies between 49 ^{0}C and 174 ^{0}C

Microwaving the vermiculite at 1000W for 1 minutes dries the clay mineral as the d spacing decreases, Fig.1 and compared to Fig. 2.

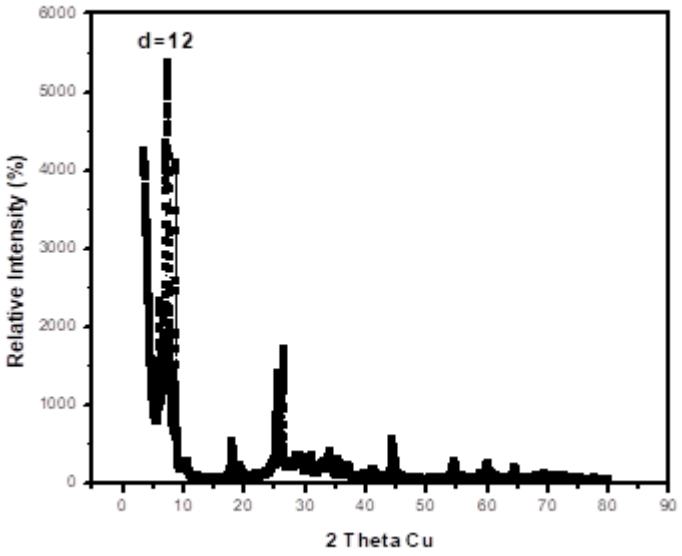

Fig.1. XRD pattern of raw vermiculite.. The observed interlayer d=12 A^0 decreases to 11.93 A^o as the clay mineral is heated with microwave, Fig. 2..

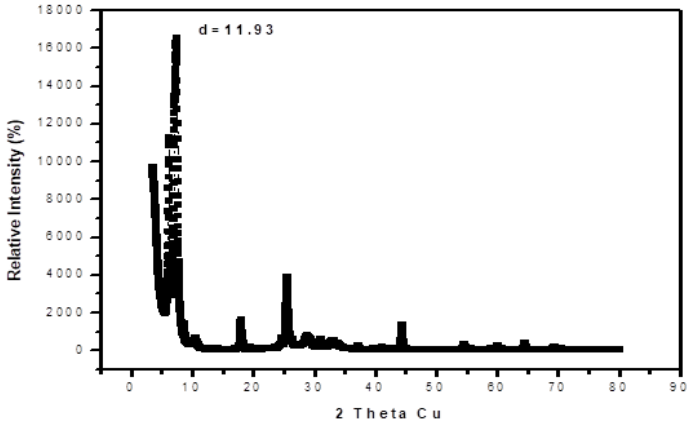

Fig.2: XRD Pattern after heating with microwaves at 1000W for 1 minute

The above figure is the XRD pattern of vermiculite after heating with microwaves at 1000W for 1 minute. The highest peak lies at 7.41 (2 theta Cu Kα) and has a d-spacing of 11.93Å. The fig.1 below is the XRD pattern after heating with microwaves at 800W for 1 minute.

TABLE I: SURFACE TEMPERATURES OF VERMICULITE HEATED WITH MICROWAVES AT DIFFERENT POWERS AND DURATIONS

Power Level (W)	**40sec**	**1 min**	**1min 30s**	**2 min**	**2 min 30 s**	3minutes
1000	162°C	116.°C	87.°C	80°C	99.°C	109^0C
800	108°C	174.°C	111.°C	128.°C	138.°C	103^0C
600	49.°C	103.°C	67.°C	95.°C	104.°C	106^0C
400	58.°C	69.°C	76.°C	69.°C	82.°C	83^0C
200	No Exfoliation	No Exfoliation	58.°C	58.°C	69 0°C	54^0C

The table 1 above indicates the surface temperatures of different samples exposed to microwaves at different power levels and time intervals. It was observed that at low power levels and lower durations, there was no exfoliation. It was expected that the surface temperature would increase proportionally to power and time but as it can be seen from this table 1, the data do not show any trend. An IR temperature measuring device was used to measure the surface temperatures by targeting a particular grain and reading off the temperature. It was therefore impossible to target the same grain throughout since different samples were used for this testwork.

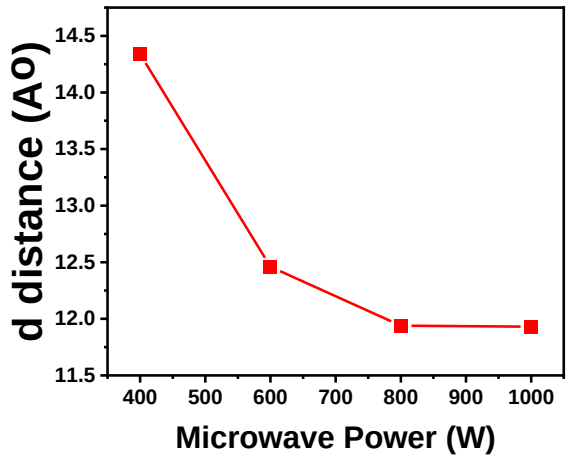

Fig.3. The vermiculite distance interlayer decreases as a higher the material is subjected toa higher magnetron power for the same duration. (here 1 minute).

The stronger the microwave power, Fig. 2, the more exfoliation is observed

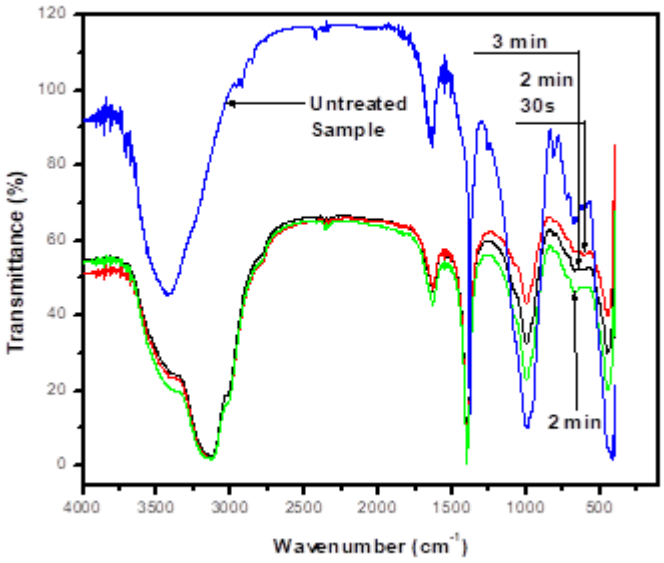

Fig.4. FTIR of microwave heated vermiculite. The OH band of the bending vibration has shifted towards 3000 Cm^{-1} showing an excess heating of the material leading to the water evaporation.

This confirms the internal heating characteristics of microwave heating. Increasing microwaving exposure as expected dries more the mineral

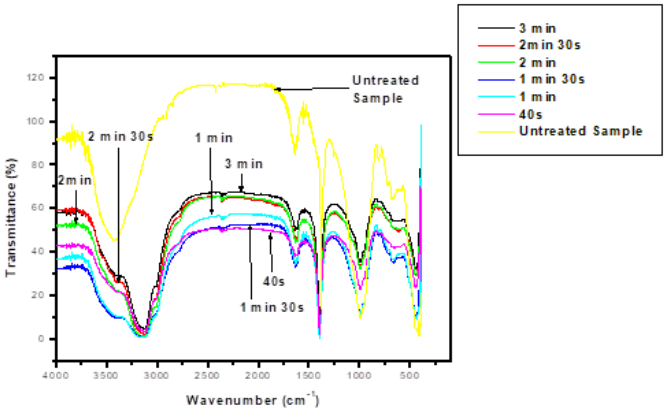

Fig.5. FTIR of microwaved vermiculite at varying exposure times. The internal drying is more pronounced as the heating time increases from 40 second to 3 minutes.

The conventional heating of vermiculite at 800°C resulted in a decrease of the d-spacing as the time decreased. The literature [1] states that vermiculite does not expand beyond 14Å as observed in the Fig. 6. but when heated up to 700°C the d-spacing collapses to 9.3 Å [1] The d-spacing dropped from 13.93 to 9.32 Å

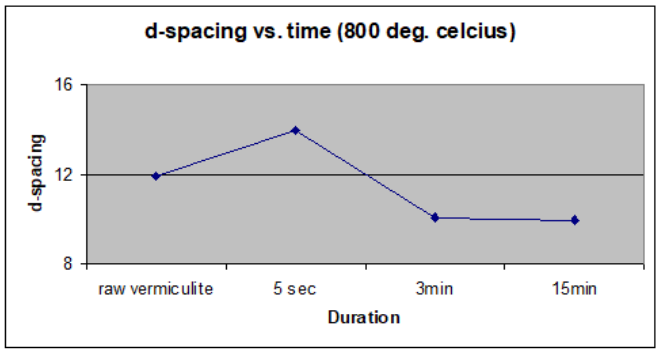

Fig 7. Responses on the responsible consumption and production goal.

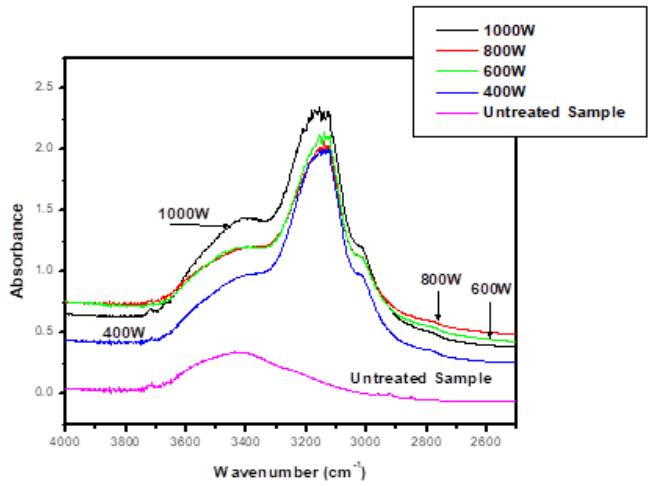

Fig. 8. O-H Stretching band after heating with microwaves at different powers for 1 min

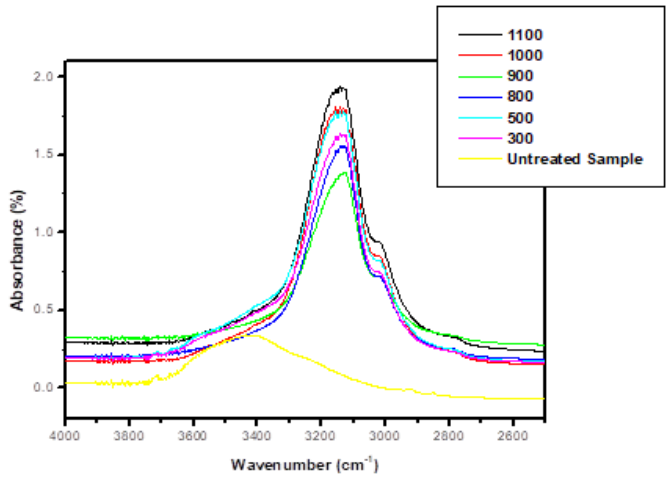

Fig. 9: O-H Stretching band after heating for 3 min at different powers

The above figure is the O-H stretching spectra after conventional heating at 1100, 1000, 900,800, 500 and 300°C. Similarly to microwaves heating, conventional heating also showed the movement and shrinkage of the broad O-H stretching bond. The only visible band is the absence of the band at 3700cm-1 and a development of a small band at 2750cm-1.

V. Conclusion

It was observed that as the microwave power decreases at lower durations, the d spacing increases from 11.93Å up to 14.33 Å. As demonstrated from the FTIR spectrum, the microwaving of vermiculite accelerates the water evaporation as the bending OH band shifts towards 3000 Cm^{-1}. ending

The X-Rays diffraction analysis of microwaves heated vermiculite showed decrease of the d-spacing at high powers and low durations. The conventional heating revealed that at high powers, the d-spacing decreased as the time was increased. The Fourier Transform infrared analysis revealed a movement and shrinkage of the O-H stretching vibration band for both microwaves and conventional heating. The decrease in the intensity of bands was even higher when heating at high temperatures. It was noticed that microwave heating at higher powers and temperatures showed the reduction in the intensities of O-H bending and Si-O vibration bands.

Contribution of authors

Antoine F. Mulaba - Bafubiandi initiated, conceptualized and supervised the research project. Boitumelo Seemise conducted the research work under the supervisor of Antoine F. Mulaba – Bafubiandi.

Declaration of interest

The authors have no financial nor personal interest in the content of the work here presented.

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