

Microwave Digestion of Gibbsite and Bauxite in Sodium Hydroxide

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Abstract:

It was hypothesized that bauxite digestion may be improved by using microwave heating as it has been shown in literature that some material processes have improved efficiency. To test this hypothesis, a set of digestion experiments were conducted using gibbsite, one of the major minerals in bauxite. Gibbsite was digested at various temperatures (50, 75, and 95°C) in either 1M or 6M sodium hydroxide solutions for 30 minutes using either a convection oven or a 2.45 GHz microwave applicator. Results show that microwave heating provided an increase of 5-7% in the digestion after 30 minutes and required around 1/10th the time to heat the solutions compared to conventional heating. Electromagnetic simulations show that preferential heating occurs at the solution surface creating a temperature gradient within the solution. Although vigorous stirring of the solution was used to minimize the temperature gradient, it could still be responsible for the observed difference in digestion. Digestion of bauxite itself yielded similar results to the gibbsite.

1. Introduction

Aluminum metal and its oxide are two important materials used in today's society. In 2015 alone, 120 million metric tons of aluminum oxide (Bray, 2017a) and 57.5 million metric tons of aluminum were produced (Bray, 2017b). These materials are extracted from bauxite ore through the highlighted digestion step in the process shown in Figure 1. Bauxite is made up of multiple minerals, which vary in concentration based on the region the ore came from. The ore typically contains one of three aluminum based minerals (gibbsite, boehmite, or diaspore) alongside other iron, titanium, and silicon based minerals such as hematite, anatase, quartz, and kaolinite (Sinton, 2006). During digestion the caustic soda reacts with the aluminum minerals via reactions such as the one for gibbsite shown below in Equation 1 (Heimann, 2010; Kaußen and Friedrich, 2016).

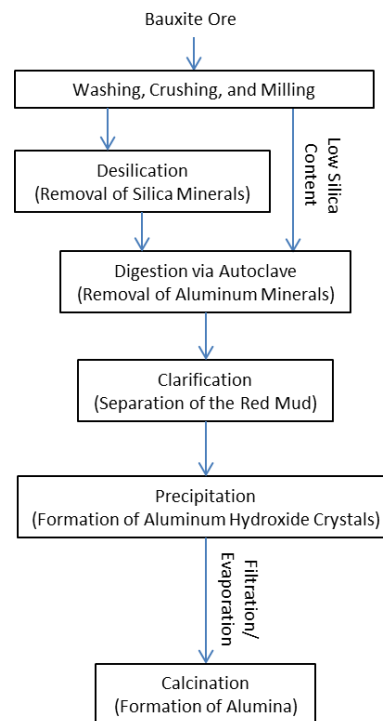
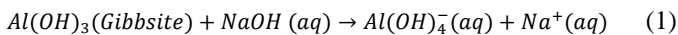


Figure 1: Overview of the Bayer Process

The goal of this research was to determine if microwaves would improve the digestion of gibbsite and bauxite by either reducing the reaction temperature or time or increasing the digestion. Microwaves have been used in applications ranging from solution preparation for inductively coupled plasma mass spectrometry (ICP-MS) to the drying and sintering of ceramic parts (Barclay, 2004; Clark, 2005;

Kingston and Jassie, 1988; “Microwave Digestion - EPA Method 3052 on the Multiwave 3000,” n.d.). Some mineral digestion processes have shown improvement when using microwave heating such as the digestion of chalcopyrite (copper ore) by ferric sulfate via selective heating of the ore (Al-Harashseh et al., 2009, 2006, 2005; Al-Harashseh and Kingman, 2004).

There has been some work using microwaves to pre-roast diasporic bauxite to improve the digestion but overall little research has been conducted investigating how microwaves could change the digestion for other types of bauxite (Le et al., 2017). For this research the digestion reaction and experimental parameters were based on information available about bauxite digestion conditions (“Controlling Alumina to Caustic Ratio,” 2008; Goyal, 2015; Griffing and Overcash, 2010; Heimann, 2010; Sinton, 2006). Changes were measured by examining the difference in the percentage of the material digestion that has occurred between microwave heating and conventional heating at specific temperatures and monitoring changes to the rate of heating for the solutions.

2. Materials and procedures

2.1. Material characterization

Before starting experiments, characterization of the materials was performed using a Horiba LA-950 particle size analyzer and a Panalytical X'Pert³ Powder X-Ray Diffraction (XRD) platform. Using this equipment the particle size range was measured and the phase of the aluminum hydroxide powder was confirmed to be gibbsite. Results showed that the majority of the gibbsite powder was below 10 microns and had a bimodal distribution with one peak at 0.3 microns and a second peak at 12.3 microns with a d₅₀ of 7.0 microns. The bauxite used in experiments was sieved to two different particle sizes, 38-53 microns and 1.2-1.7 mm.

In addition to the particle size and composition of the sample powders, the dielectric properties of the materials and containers were analyzed. The dielectric properties for the sodium hydroxide solutions, the gibbsite powders, and the bauxite powders were experimentally determined while the polymer properties were found in literature. Dielectric measurements of the gibbsite and bauxite were carried out using the cavity perturbation method (Von Hippel, 1954). A high performance coaxial probe (Keysight, N1501A) was used to measure the properties of the sodium hydroxide solutions. The coaxial probe technique is more suited to measure a high-loss liquid and provides a greater degree of accuracy than cavity perturbation when a significant volume reduction occurs due to evaporation at high temperatures. Uncertainties for the dielectric constant, ϵ' , are +/- 5% and loss tangent, $\tan\delta$, were +/-0.02 (Gregory and Clarke, 2006).

Based on their dielectric properties, polypropylene and PTFE were used as the reaction vessel and temperature probe sheath since they were nearly transparent to microwaves at the

frequency used. Gibbsite and bauxite were weak microwave absorbers at the 2.45 GHz frequency while both of the sodium hydroxide solutions were highly microwave absorbent. The depth of penetration (36.8% of the energy remains) for the solutions is less than 2mm. Therefore, most of the energy applied to the solution was absorbed in a thin outer layer of the liquid. Vigorous stirring was used to create a vortex, allowing more material to interact with the microwaves and create more even heating throughout the solution.

2.2. Electromagnetic simulation

Simulation for the reaction inside the microwave was undertaken to better understand the distribution of the electric field and power density. It was created using the Radio Frequency module in COMSOL Multiphysics software (V5.2, COMSOL Multiphysics, Burlington, USA), a commercially available software based on the Finite Element Method (FEM) (Jin, 2002). The simulation procedure involved constructing a tetrahedral mesh of the reactor and reaction mixture. At each intersection point, Maxwell's equations were calculated to derive the magnitude of the electric field and power density distribution (amount of power absorbed into the load), which together predicted the heating behaviour of the reaction mixture during processing and therefore the overall performance of the system. For this paper a simulation at 75°C with no stirring was completed for a 1M solution. This simulation was representative of the distributions at other temperatures that were tested as the only difference should be the decrease in depth of penetration as temperature and caustic soda molarity are increased.

2.3. Experiment variables and conditions

Temperature versus digestion experiments were conducted to measure how microwave heating affected the process. To simplify the analysis of the digested material aluminum hydroxide (gibbsite) was chosen to represent the aluminum minerals in bauxite. The gibbsite used was an extra pure grade material from Acros Organics (Lot A0371406). A set of experiments was conducted on multiple particle sizes of bauxite at 95°C to determine if the gibbsite results would be representative of actual bauxite in a microwave. The bauxite used in the experiments was taken from a mine in Western Australia. Aluminum concentration in the bauxite ore was determined using inductively coupled plasma mass spectroscopy (ICP-MS). Mineral liberation analysis (MLA) was used to show that the aluminum minerals in the ore were predominantly gibbsite while boehmite and silicon based minerals (quartz and Muscovite) account for roughly 3% of the digestible material as seen in Table A3. In the table, int Al and int Fe represent interstitial Gibbsite and FeO. The MLA method collects an XRD spectrum for hundreds to thousands of particles and matches it with the standards for the different minerals identified. The process useful for identify the ratios of different minerals present but is not as accurate as the available ICP-MS data for determining the total amount of aluminum in the ore. Because of this the MLA analysis was used to estimate

how much of the aluminum was from nongibbsite sources. Once those were removed, the total amount of gibbsite was calculated to be 54.9-55.5% gibbsite for both of the particle sizes used.

The experimental matrix for this research was constructed using the following two variables: solution temperature and solution molarity. Three tests were conducted for each variable combination for the gibbsite experiments and two tests were conducted for the bauxite experiments. Other experimental conditions such as pressure, time at temperature, stir speed, and microwave power were kept constant to limit the number of changing variables. Powder digestion was measured at three different temperatures: 50, 75, and 95°C. Two sodium hydroxide molarities (1M and 6M) were tested to determine how the digestion changed based on hydroxide concentration. Sodium hydroxide solutions were made using either powder from Consolidated Chemical & Solvents LLC or extra pure pellets from Fisher Chemical with an error of +/- 0.015g. The stirrer speed of 700 rpm was selected as it caused the solution to vortex.

2.4. Experimental design

At the start of each test, powder was added to a 15mL solution of sodium hydroxide contained in a 30mL polypropylene container. The initial ratio of alumina to free caustic soda (A/C) was 0.82:1. Pressure inside the container was released via a hole in the container lid. After the powder was added to solution, the container was placed inside the heating apparatus. Microwave heating was achieved using the Sairem Miniflow 200ss generator, capable of providing 200W of applied power at 2.45 GHz frequency, in a TE01 single mode cavity using a stub for impedance matching purposes. Conventional heating was completed in a Lab-Line Imperial V convection oven. Solutions were stirred using either a magnetic stir bar rotated by a stir plate (microwave heating) or an overhead stirrer with a PTFE Shaft (conventional heating). The solution temperature was monitored via an optical fiber with 0.2°C accuracy (microwave) or a type K thermocouple with 2.2°C accuracy (conventional) placed near the center of the container. Both temperature probes were protected by a 1mm thick PTFE sheath. Solutions were left at temperature for 30 minutes after heating was complete.

Before starting an experiment using microwave heating, the tuning stub on the instrument was used for impedance matching to minimize the reflected power to 10W or lower. The maximum forward power used was 75W, which was constantly modified by the system based on temperature measurements. During conventional heating, the oven temperature was initially set to be 25°C higher than the experiment temperature to accelerate the initial heating phase. This was in order to maximize the heating rate therefore enabling a better comparison with the microwave heating tests and to ensure that variations measured were not purely due to the fast heating rate. When the solution was within 20-25°C of

the selected temperature, the set point on the oven was lowered so the solution and oven temperatures would meet at the designated experiment temperature with a maximum of 2°C of overshoot. Once the conventionally heated solution was 3°C below temperature, the start time of the experiment was recorded.

After an experiment was complete the container was immediately removed from the heating apparatus and the powder was separated from solution via vacuum filtration using a rotary vane pump. Filtration was completed using either 8 micron pore cellulose paper or 0.8 micron pore nylon paper. Due to the difference in the filter paper pore size, a conversion factor between the two was created for data analysis. Once the filtration was complete, the remaining powder and filter paper were moved to an oven set to 60-70°C to dry overnight. The weight of the remaining powder was measured the next day. Total digestion was calculated using Equation 2:

$$\% \text{ Digestion} = \frac{\text{Initial wt Al(OH)}_3 - \text{Remaining wt Al(OH)}_3}{\text{Initial wt Al(OH)}_3} * 100 \quad (2)$$

To confirm that the sodium hydroxide was not converting the gibbsite to boehmite, XRD using a Bruker D8 Advance DAVINCI was completed on some of the remaining 6M powders. Figure 2 presents the XRD pattern from a 6M experiment along with lines indicating where the 100% peaks for gibbsite and boehmite should be if present. No boehmite was detected as only the gibbsite spectrum matched the sample peaks.

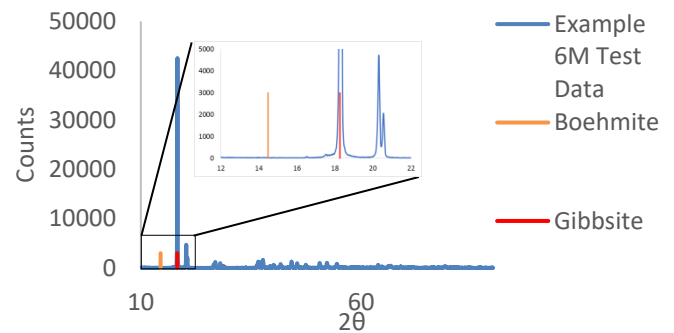


Figure 2: XRD Pattern of 6M Test Data with 100% Peaks for Gibbsite and Boehmite

3. Results and discussion

3.1. Microwave modeling

Figure 3 (A and B) shows the 3D model of the microwave reactor. This includes: variable sliding short used to maximize the amount of absorbed energy into the material and the metallic chokes used to attenuate the microwave energy from escaping outside the reactor and to assure inherent safety design and to comply with health and safety legislations (*Industrial, scientific and medical (ISM) radio-frequency Equipment. Electromagnetic disturbance characteristics. Limits and methods of measurement*, 2007). The

electromagnetic simulation in Figure 4a shows a high concentration of the electric field on the edge of the sample which is mainly due to the high dielectric loss of the hydroxide solution and therefore a skin depth heating effect (Horikoshi et al., 2018). This is also confirmed by Figure 4b where power density is dominantly high on the edge. High field concentration is observed on the interface between the sample and the vortex formed during the experiments. This is a normal behavior in such a scenario as the dielectric constant of air is 1.000536 and the medium much higher (140.78) (*Guide to Characterisation of Dielectric Materials at RF Microwave Frequencies*, 2003). A high field concentration is also observed at the poles of the magnetic stir bar. This would create an additional heating region around the magnet. To establish the

effect of skin heating, power density is plotted across the sample in the middle of the container as shown in Figure 5 A and B. The power density decreases from 13.17 MW m^{-3} down to 4.28 MW m^{-3} within the first 0.17 cm at 75°C . This corresponds to the penetration depth of the sample (0.17cm). Without stirring, the temperature at the surface of the solution would be at a much higher temperature than the bulk. During experiments the solution is rapidly stirred, minimizing the amount of time any particular volume of material resides in the region where the power and electromagnetic field are highest. Even so, it could be that the temperature gradient is not completely eliminated and this could account for the difference in digestion observed.

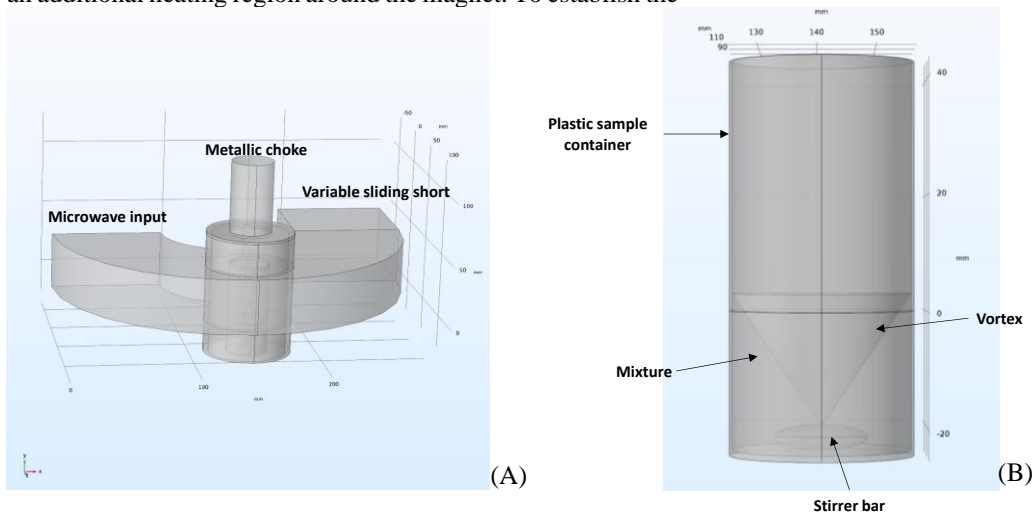


Figure 3: 3D Model of the Microwave System Showing the Reactor (A) and the Sample Container (B) with the Vortex and Stirrer Bar Modelled

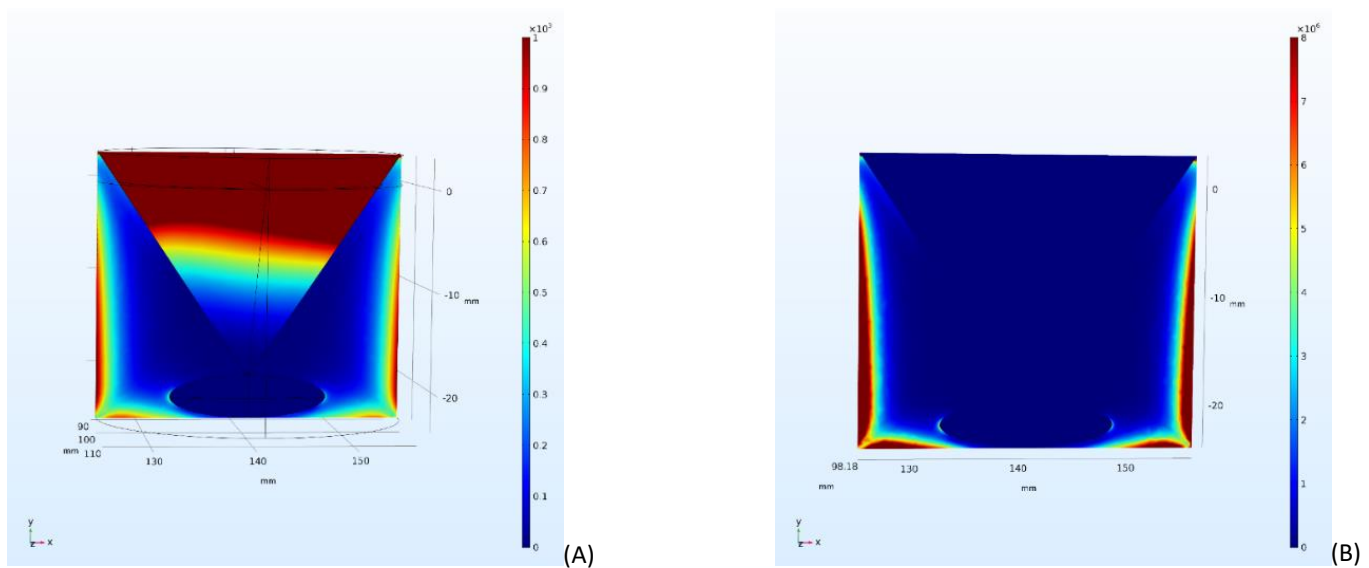


Figure 4: 2D Plot inside the Container: Electric Field Distribution (V m^{-1}) (A) and Power Density Distribution (B) Based on Electromagnetic Simulations

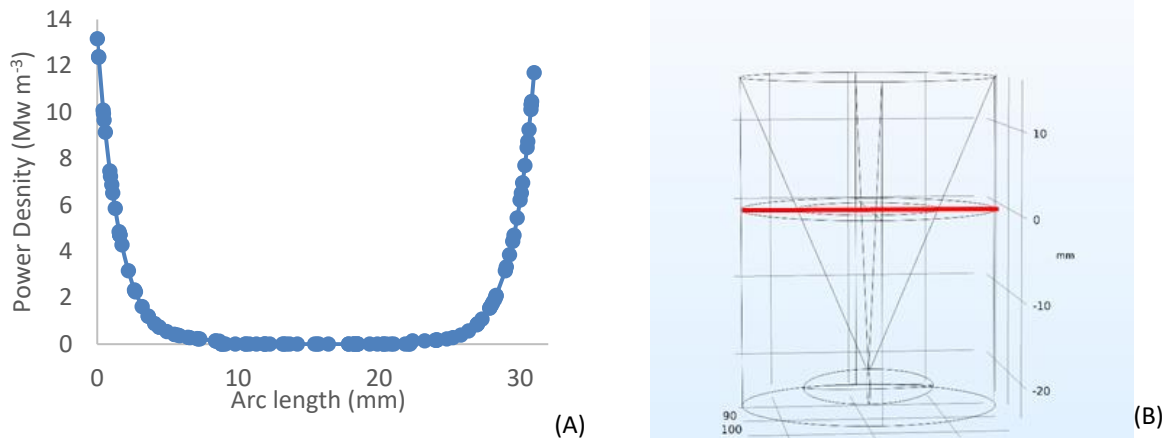


Figure 5: Power Density Distribution (A) across the Red Line through the 2D Plot (B)

3.2. Temperature vs digestion results

Figure 6 and Figure 7 display the percent digestion for 1M and 6M solutions and Table A2 in Appendix A compiles the results. The data shown in the figures and table is the average value of the three experiments conducted for each set of conditions. Included in the table with the average results are the standard deviation for each set of experiments. Experimental results with a difference of 10% or more from the average were removed from the data set and the average was recalculated. These anomalous values were thought to be due to fluid overflow during the filtration process that caused remaining material to go around the filter, lowering the nondigested weight and increasing the percentage digestion. Removing the results caused a ~5% decrease in the digestion and at least a 6% decrease in the standard deviation for the affected sets of data, indicating that this conclusion is most likely accurate. Standard deviation values for the data sets are within +/- 4% except for the 1M gibbsite at 50°C. Conventionally heated samples' standard deviations were smaller than their microwave counterparts. This was thought to be due to both a reduction in fluid overflow as proficiency in the testing procedure increased after the initial tests and due to the fact that most of the conventionally heated solutions were filtered with paper that had a smaller pore size.

There are several conclusions that may be drawn from these results. First, the total digestion increases linearly with temperature. This is shown by the linear trend line fits on the data series with high R² values (>0.99). The trend line equation and associated R² value for the percent digestion vs temperature results are provided in Table 1. Second, microwave treatment appears to have a positive effect on the digestion when compared to the conventional heating experiments. At most conditions the microwave experiments produce a 5-7% increase

in digestion over the conventional results after 30 minutes at temperature. 1M experiments seem to improve at higher temperatures while 6M experiments show similar improvement across all three temperatures.

Third, the molarity of the solutions affects the percentage of the material digested. The 6M solutions are shown to have a ~10-15% increase in digestion over the 1M solutions at 75°C and 95°C. As the temperature increases, the effect of molarity on digestion becomes more pronounced. Fourth, it is possible to estimate at what temperature complete digestion should occur using the equations in Table 2. This could be extrapolated to also determine the temperature required to completely digest different amounts of material. Finally, these plots and tables could also be used to estimate a reduction in temperature that could occur by switching to microwave heating. For example, the required temperature for digestion could be lowered ~7°C for 6M solutions by switching from conventional to microwave heating.

For the bauxite results (Figure 8) there are several points of interest. Trends in digestion are similar for both the gibbsite and bauxite, indicating that there is minimal effect from the additional minerals present in the bauxite ore. The amount of bauxite digestion is slightly higher than for the gibbsite for both conventional and microwave experiments. This is most likely due to the presence of digestible minerals other than gibbsite in the ore. Results also indicate that after 30 minutes of microwave digestion the effects of particle size are minimal (~2% difference in digestion) while there is slightly more of an effect during conventional experiments (~5% difference in digestion).

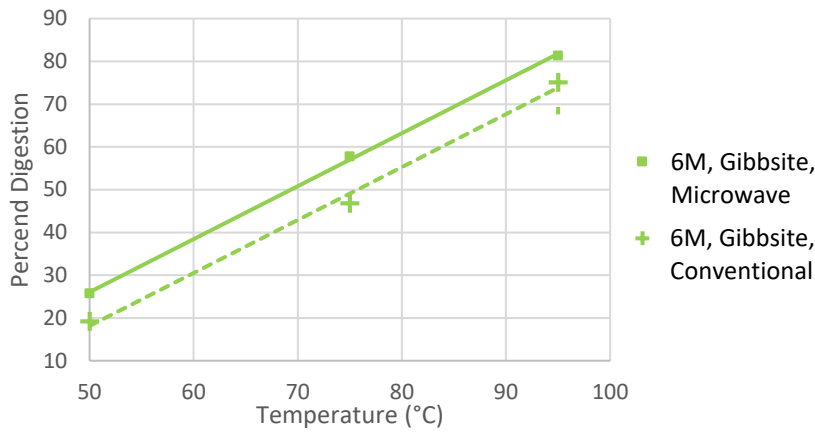


Figure 6: Average Percent Digestion for 6M Solution Experiments after 30 Minutes Soak at Temperature

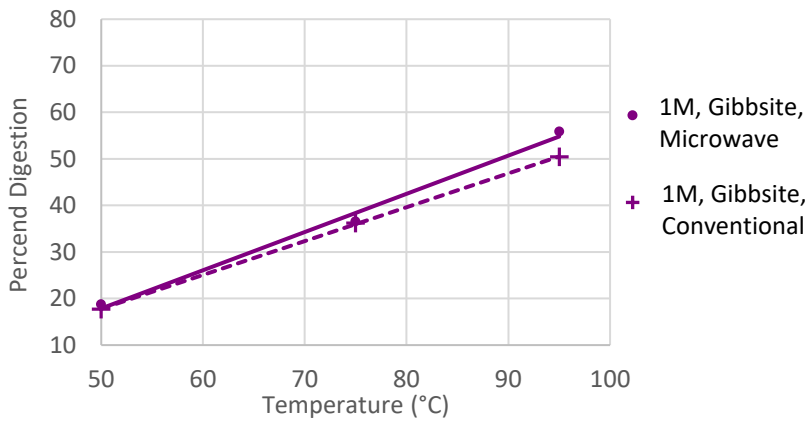


Figure 7: Average Percent Digestion for 1M Solution Experiments after 30 Minutes Soak at Temperature

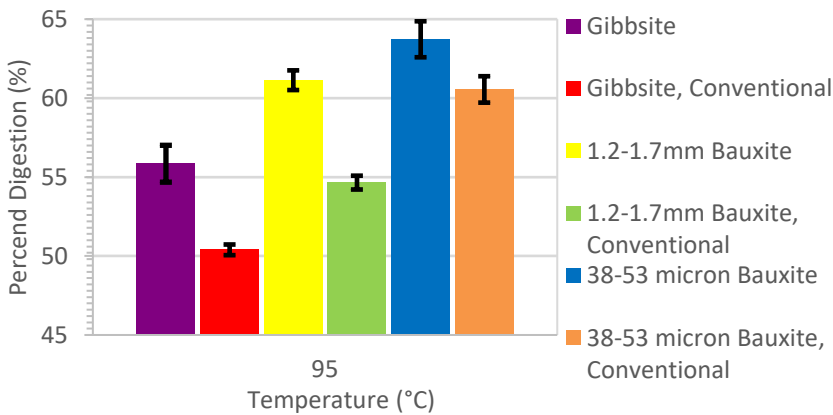


Figure 8: Average Percent Digestion at 95°C for Bauxite and Gibbsite in 1M Solutions Experiments after 30 Minute Soak at Temperature

Table 1: Trend Line Equation for Percent Digestion vs Temperature and Associated R² Value for Percent Digestion Data

Data Series	Trend line Equation	R ² Value
1M, Microwave	$y = 0.8207x - 23.159$	0.9923
1M, Conventional	$y = 0.7269x - 18.54$	0.9999
6M, Microwave	$y = 1.2383x - 35.859$	0.9994
6M, Conventional	$y = 1.2376x - 43.724$	0.9950

3.3. Solution Heating

Table 2 displays the average microwave and conventional heating times required to reach digestion temperature for each set of conditions. Microwave heating time was determined using the built in timer on the instrument and was accurate to the second. Conventional heating time was determined using a clock and is rounded to the minute. The results show that the microwave oven was able to heat the solutions around 10x faster than the convection oven set to an initial temperature higher than the experimental temperature. This indicates that if the oven was initially set to the experimental temperature, the time saved by using microwaves would have been even higher. Analyzing the temperature vs heating time data shows that for every 25°C increase in solution temperature, the required time to heat a solution was ~1.5x longer than before. This could lead to faster batch times for bauxite processing since less time is spent on heating the slurry.

Table 2: Average Initial Heating Time

Data Series	Temp, °C	Conventional Heating, minutes (STDEV)	Microwave Heating, minutes (STDEV)
1M, Gibbsite	50	9 (1.0)	0.83 (0.31)
	75	14 (1.7)	1.28 (0.10)
	95	21 (7.5)	1.61 (0.36)
6M, Gibbsite	50	8 (1.5)	1.05 (0.57)
	75	18 (3.8)	1.73 (0.11)
	95	22 (0.6)	2.50 (0.25)
1M, 1.2-1.7mm Bauxite	95	20 (0.7)	1.83 (0.04)

4. Conclusion

Using microwave heating instead of conventional heating results in a 5-7% increase in digestion and requires a tenth of the time to heat the mixtures to temperature. The digestion versus temperature trends observed were linear for all experiments with high R² values for confirmation. Results of the gibbsite digestion are 3-5 percent lower than the gibbsitic bauxite tested but the trends between both materials are similar. This difference is most likely due to the presence of digestible minerals other than gibbsite in the ore.

Electromagnetic simulation confirmed that a skin effect took place, causing heating to occur at the surface of the

solution which under static conditions would create a temperature gradient where the surface would be at a higher temperature than what was measured in the bulk. Although vigorous stirring most likely minimized the temperature difference between the surface and bulk, there could be enough of a gradient to explain the small increase with microwave digestion. At this time it is not possible to conclude whether or not microwave heating offers any advantages over conventional methods other than more rapid heating.

Future research should move experiments to a microwavable autoclave with an insulated chamber and use larger volumes of solution. Additionally experiments should examine how the digestion changes when going from a pure sodium hydroxide solution to a recycled sodium aluminate solution. These experiments would help determine if microwave heating would be beneficial under normal bauxite process conditions.

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Appendix A: Supplemental Material

Table A3: Mineral abundance in wt% for the two bauxite particle sizes used

Mineral	38-53 microns	1.2-1.7mm
Gibbsite	9.8	7.5
Boehmite	0.3	0.6
Hematite	4.9	5.0
Goethite	1.3	2.5
Illmenite	1.2	0.6
Muscovite	0.2	0.1
Quartz	1.3	0.7
FeO int 10% Al	14.1	11.6
FeO int 15% Al	14.7	15.1
FeO int 25% Al	4.6	5.6
Boehmite int	1.5	1.3
Gibbsite int 5% Fe	15.1	15.3
Gibbsite int 10% Fe	12.7	12.6
Gibbsite int 26% Fe	9.0	10.6
Gibbsite int 30% Fe	4.4	4.6
Gibbsite int 33% Fe	4.9	6.4

Table A2: Average Results of Temperature vs Digestion Experiments

Data Series	Weight Digested (g) (STDEV)			Percent Digested (%) (STDEV)		
	50°C	75°C	95°C	50°C	75°C	95°C
1M, Gibbsite, Microwave	0.142 (0.045)	0.2748 (0.003)	0.422 (0.0157)	18.71 (5.94)	36.52 (0.37)	55.84 (2.09)
6M, Gibbsite, Microwave	1.163 (0.164)	2.618 (0.126)	3.657 (0.210)	25.72 (3.65)	57.78 (2.88)	81.36 (3.88)
1M, Gibbsite, Conv.	0.134 (0.002)	0.273 (0.008)	0.380 (0.006)	17.71 (0.23)	36.20 (0.98)	50.39 (0.67)
6M, Gibbsite, Conv.	0.864 (0.090)	2.110 (0.146)	3.386 (0.048)	19.17 (1.97)	46.82 (3.29)	75.12 (0.94)
1M, 1.2-1.7mm Particle Size Bauxite	-	-	0.443 (0.006)	-	-	61.13 (1.06)
1M, 38-53 micron Particle Size Bauxite	-	-	0.460 (0.012)	-	-	63.73 (1.88)
1M, 1.2-1.7mm Particle Size Bauxite, Conv.	-	-	0.395 (0.005)	-	-	54.65 (0.83)
1M, 38-53 Micron Particle Size Bauxite, Conv.	-	-	0.434 (0.012)	-	-	60.55 (1.38)