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Mechanical and Thermal Properties of Microwave Synthesis Fly Ash Based Geopolymer for Building Applications

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Abstract: ***Fly ash is a major by-product from power stations. It is can be used to produce geopolymers materials at low temperatures in alkali solutions. Heat curing is used to improve the reaction and strength of geopolymers. Microwave is an innovative tool to heat the geopolymers and influenced the chemical reactions and enhanced the geopolymer strength. In this study the effect of microwave heating on the mechanical and thermal properties of geopolymers was discussed. This study discussed the compressive strength of room temperature and microwave heated geopolymers. The geopolymers were heated in microwave for 10, 15 and 20 minutes respectively. SEM results showed that the microstructures changed with increasing microwave heating time. Porosity and density results obtained showed a relationship with microwave heating. Higher compressive strength was obtained for the geopolymers with microwave heating compared to room temperature cured geopolymer. Also the results obtained showed that microwave synthesis geopolymers have good potentials to be used as blocks for buildings and as well for insulations for building walls.***

Keywords: Fly ash, Geopolymer, Microwave, Temperature, Compression

1. Introduction

Coal is nowadays considered globally as major source of producing energy. Coal powered generating plants produce in large quantities coal ashes comprising of fly ash and bottom ash. Approximately, the annual production of coal ash globally is about 600-800 million tons (Leiva, Arenas, Vilches, Alonso-Fariñas, & Rodriguez-Galán, 2015). The huge demand for energy both domestically and industrially caused more usage of coal and hence the generation of fly ash in large quantities, which should be disposed with safety, handle as a valued resources and as well avoiding polluting the environment (Strydom & Swanepoel, 2002).

These ashes are mostly dumped in landfills or used for construction purposes. A recent development in the utilsation of fly ash in construction industry, is in the manufacture geopolymer binder which serves as substitute for portland cement (Ul Haq, Kunjalukkal Padmanabhan, & Licciulli, 2014a). Although cement used as binder in concrete is universally accepted, however, to manufacture one ton of portland cement about one ton of carbon dioxide is produced to the atmosphere, because of the calcination of lime stone and fossil fuel combustions. (Zhang, Provis, Reid, & Wang, 2015).

The two main constituents of geopolymers are the source materials and alkali liquids solutions. The alumino-silicate are mainly the geopolymer sourced materials and must be rich in aluminium (AL) and silicon (Si), while the alkali solution can be mixture of sodium silicate/sodium hydroxide or potassium silicate/potassium hydroxide—(Haq, Padmanabhan, Zubair, Ali, & Licciulli, 2016). The activation of alkali material is the chemical reaction that cause sudden change of certain structures into frameworks that become compacted in a cementious nature. (Mustafa Al Bakri, Kamarudin, Bnhussain, Nizar, Rafiza & Zarina., 2012).

Geopolymers can be produce at both low and high temperatures. At low temperature it is produced using fly ash dissolved in high alkaline solution via process of hydrolysis. Heat curing is employed to enhance the reaction and geopolymer strength (Onutai, Jiemsirilers, Thavorniti, & Kobayashi, 2016). The common methods of curing the geopolymers are the room temperature and the conventional oven heat curing. However, a recent development is in the used of innovative microwave method in curing the geopolymer. This is because, with other methods, heating is not uniform sometimes it takes longer period of heating to reached the desired strength. However, the microwave energy penetrates the materials and developed heat all over the materials. This trend generates faster and voluminous heating that hastened strength development and boost specimens mcrostructure. (Graytee, Sanjayan, & Nazari, 2018). The microwave radiation heating depends on dissipation of internal energy related with molecular dipoles excited in electromagnetic fields, and this result in quick and increased uniform heating. (Zhuang, Chen., Komarneni, Zhou, Tong, Yang & Wang, 2016).

1.1: Source Materials

The raw fly ash obtained from Lafarge Cement Power Plant Sagamu, Ogun state, Nigeria was used for the experiment.

1.2 Characterisation of fly ash

The obtained raw fly ash was analysed for microstructural analyses using SEM using the Hitachi S 3400N and using the X -Ray Diffraction (XRD) using Bruker D8 instrument, while the X-Ray Fluorescence (XRF) using the Bruker AXS model S4 Pioneer instrument machine respectively.

1.3 Alkali liquid solutions

The sodium silicate solution of relative density of 2.13g/cm3 was thoroughly mixed with. liquid sodium hydroxide solution of 0.1M, density of 1g/cm3, in the ratio of 2: 1 to obtained the required alkaline solution.

1.4 Geopolymer preparation.

The mixing of the fly ash and the alkaline liquid solution in the ratio of 1.5: 1, was carried out in a plastic molds for seven to ten minutes to obtained the required homogenous paste. The resulting homogenous mixture was poured in 50 x50 x 50 mm3 plastic containers, vibrated, choked and allowed to settle.

1.5. Geopolymer microwave synthesizing and curing.

One set of the homogenous paste in the plastic molds was cured at room temperature. The other three sets were heated in the microwave at moderate microwave radiation power of 200W for 10, 15 and 20 minutes respectively, for the geopolymerisation process. An infra-red thermometer was used to register the heating temperatures of the respective heated geopolymers. After 24 hours, all the geopolymers were demolded and finally cured at room temperature for 14 days in preparation for compression test.

1.6. Geopolymer characterization

Both the room temperature and the microwave synthesized geopolymers were all analysed for morphology, using the Scanning Electron Microscopy (SEM) analysis.

1.7. Density and Porosity of geopolymers

In this case, the geopolymer specimens were dried in oven, where the oven-dry densities, absorptions and porosities of the geopolymers were evaluated according to ASTM C140-17b.

1.8 Thermal conductivity of geopolymers

The thermal conductivity tests were based on steady-state method, which consisted of having each sample in the middle of two metals discs and the heat losses calculated accordingly.

1.9 Compressive strength of geopolymers

The compression strength was conducted according to ASTM C109 16a standard, using the eco-smartz automatic compression machine. Three samples from each specimen group were tested to find the average mechanical strength value for each respectively, similar to other method employed in another research. (Bucham and Igbax, 2022).

2. Results and Discussion

2.1 X-ray Fluorescence Analysis (XRF) of Fly Ash

The figure table 1, below shows the XRF elemental analysis of the fly ash, and the results shows a content of low CaO content, with relatively higher content of SiO_2, Al_2O_3 and Fe_2O_3 content, with smaller percentages of magnesium, kalium, potassium, sodium and titanium oxide also present. The class F fly ash is characterized with inter particle speciation due to the crystallization of mullite from the aluminosilicate. The class F fly ashes because of their low in CaO, according to ASTM C618 are the most widely used in geopolymer synthesis.

Table 1: XRF analysis of Sagamu cement factory fly ash

Chemical contents	Fly ash (%)
SiO_2	60.12
Al_2O_3	24.29
Fe_2O_3	7.50
Ca0	1.32
K_2O	0.86
TiO_2	3.39
Cl	0.63
ZrO_2	0.11
K_2O	0.76
SO_3	0.80
BaO	0.22

2.2 X-Ray Diffraction (XRD) of fly ash

The figure 1, shows the XRD patterns of the fly ash. The predominant peaks were quartz (SiO_3) and mullite ($3AL_2O_3.2SiO_2$). The non-crystalline phases are the broad humps and spread from $2\theta = 28^0$ up to $2\theta = 70^0$C. The broad humps represent the amorphous phase in the fly ash, which was because of the presence of glassy fraction, and this can be linked to the type of fuel used. Furthermore, presence of the crystalline peaks of quartz and Mullite, have effect on the mechanical and physical properties of the geopolymer.

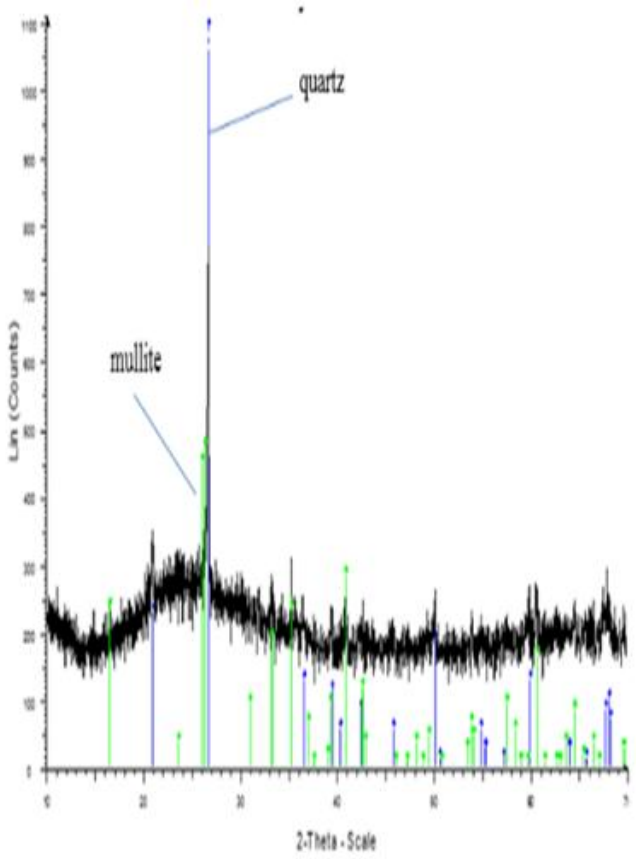

Figure 1: XRD analysis of Sagamu Cement factory Coal fly ash

2.3 Scan Electron Microscopy (SEM) analysis of Fly Ash

The microstructure of the source fly ash powder was studied using the Hitachi S-3400N. The figure 2, showed the fly ash consisted of series of fine spherical particles of different sizes mostly combusted carbons of diameters ranging from 1.51 um to 5.19 um. It consisted of some larger spherical particles co-existing with some other smaller particles.

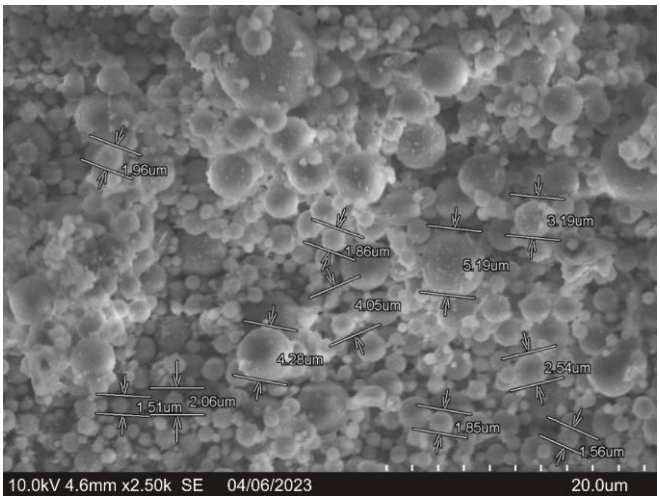

Figure 2: SEM micrograph of fly ash, 2500X

2.4 Scanning Electron Microscopy (SEM) analysis of geopolymer

The SEM the microstructural analysis of the fly ash geopolymers. is shown. The geopolymer G-00 has some few particles partially reacting with the alkali solution and co-existing with non reacting particles, (Zhang et al., 2015) thus reacting slowly figure 3. When 10 minutes microwave heat was applied on geopolymer G-10 it intensified the reaction, figure 4. . Applying more heating to 15 minutes, resulted in bi-directional attack on smaller particles which expanded from the inside out and vice versa thereby exposing smaller particles, G-15 in figure 5.

As more heating of 20 minutes was applied on geopolymer G-20, figure 6. This led to more dissolution and more alkaline solution penetration into the smaller particles and hence the spaces between the larger particles start accumulating with reaction products (Deventer, Provis, Deventer, Keyte, & Duxson, n. d.), More penetration of microwave heat energy caused formation of sodium alumino-silicate gel on the fly ash surface and in between the particles spaces. This accelerated the geopolymerisation reaction and finally forming dense and harder matrix with increasing strength.

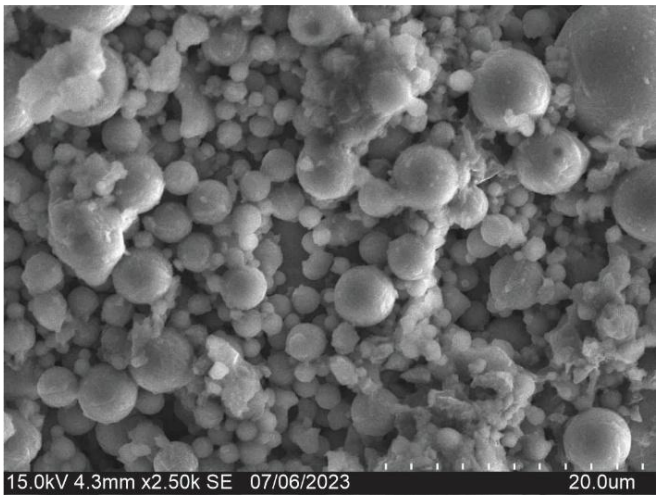

Figure 3: unheated (room temp.) geopolymer

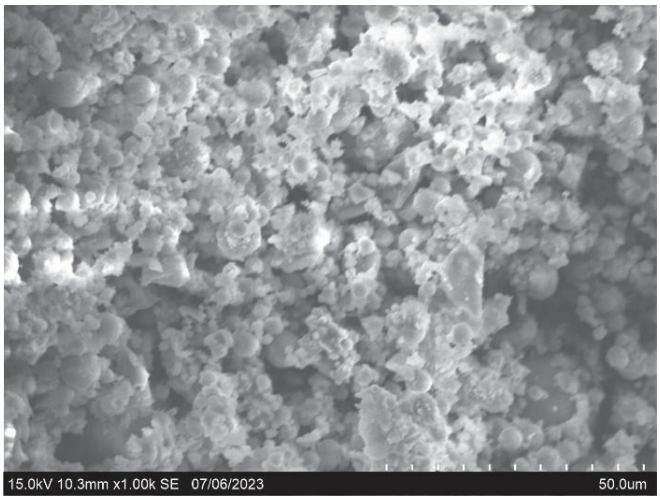

Figure 4: 10 min. heated geopolymer

Figure 5: 15 min. heated geopolymer

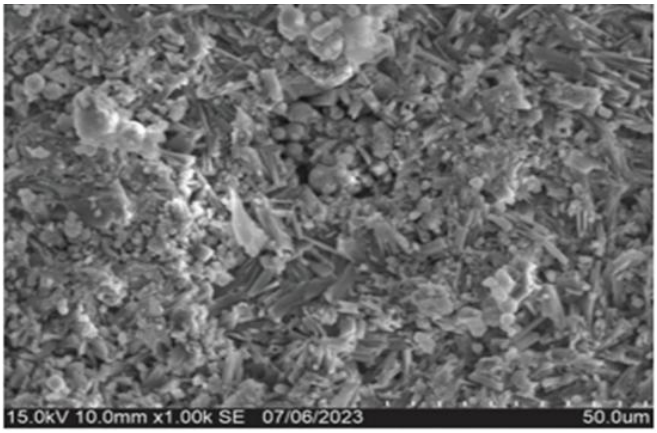

Figure 6: 20 min.

2.5 Porosity and Bulk density

The effect of microwave heating on porosity and density of geopolymer is shown in figure 7, G-00 (unheated) geopolymer at room temperature registered highest porosity of 15.2% with lowest density of 1654.36kg/m^3. Applying microwave heating for 10 minutes reduced the porosity to 11.4%, but with appreciable density increase to 1784.53kg/m^3. Moerover, increasing heating time to 15 minutes i.e. increasing microwave radiation heating, further reduced the porosity to 9.3%, and registered density increase to 1972.6kg/m^3. At 20 minutes microwave heating time, the porosity further decreased to 6.6% but with significance increase of density to the highest value of 2367.61kg/m^3.

It can be observed that with increase in microwave heat radiation time there is corresponding decrease in porosity but with increased in bulk density. Because increasing microwave heating decreased the porosity because of the released of gas as a result of microwave radiation (Ul Haq, Kunjalukkal Padmanabhan, & Licciulli, 2014b). Also the increase in heating overcame the activation energy. which reduced the porosity and resulted in more densification (Nyale, Babajide, Birch, Böke, & Petrik, 2013).

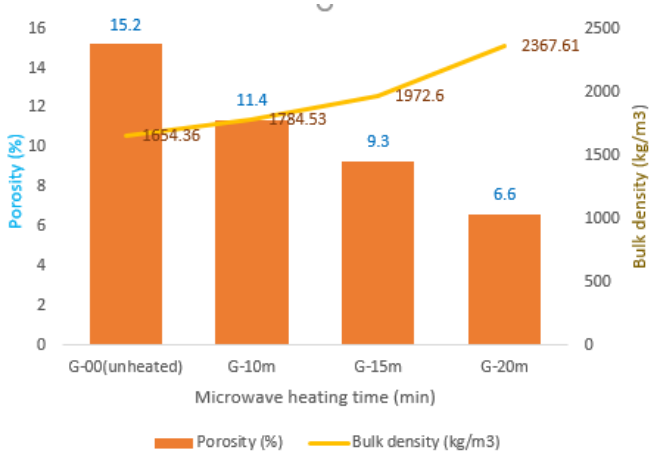

Figure 7: Relationship of microwave heating with porosity and bulk density

2.6 Microwave heating effect on porosity, bulk density and compression strength

The figure 8, shows the G-00 geopolymer at room temperature with registered porosity of 15.2%, bulk density of 1654.36kg/m3 and lower compression strength of 5.19Mpa. Applied microwave heating of 10 minutes on geopolymer G-10m reduced the porosity to 11.4% and increased bulk density of 1784.53kg/m3 and increased in compression strength to 5.74Mpa. Again heating the geopolymer G-15m for 15minutes further reduced the porosity to 9.3% and raised the bulk density to 1972.6kg/m3 and resultant high compression strength of 6.24Mpa. Finally heating geopolymer G-20m for 20 minutes in the microwave developed the lowest porosity of 6.6% and increased in density of 2367.61kg/m3 with the highest compression strength of 7.14Mpa. This is because the presence of microwave electric fields provides special interaction of the microwaves radiation with the materials resulting in lower porosity, reduced surface area and faster and more densification leading to higher strength of the geopolymers (Al-Bakri Abdullah et al., 2012) . Microwave radiation heat increased the densification and overcame the geopolymerisation activation energy (Chindaprasirt, Rattanasak, & Taebuanhuad, 2013).

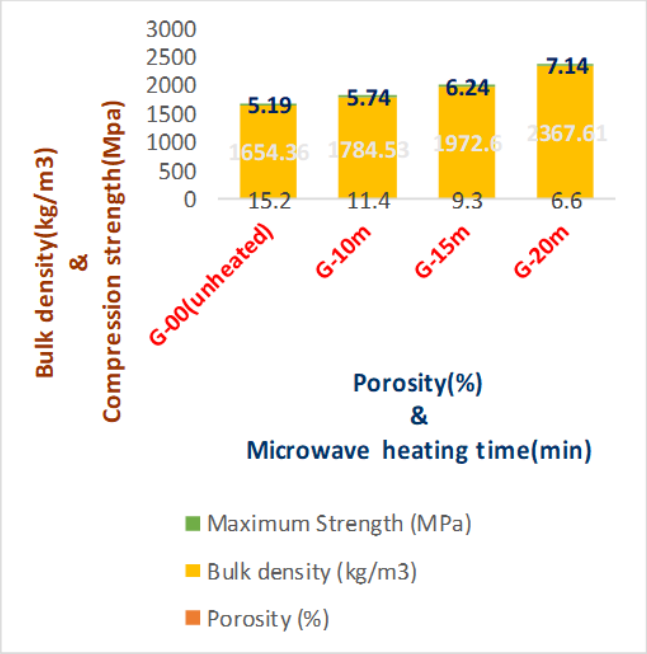

Figure 8: Microwave heating and porosity, density and compression strength of geopolymer

3. Effect of microwave heating on compression strength of geopolymers

The compression strength tests of the fly ash based geopolymer were conducted using the Eco-smartz automatic compression machine with a loading rate of 1.8KN /sec, after fourteen days of room temperature curing, according to the ASTM C 109 16a standard. This was carried out at the average of three tests. The detailed result of the compression strength values of the geoplymer specimens after microwave synthesis heating at 200 watts for 10, 15 and 20 minutes.

The table 2 below, showed a summary of the average compression strength values of the geoplymer specimens for the room temperature curing and the microwave heated geopolymers.

Table 2: Microwave heat and compression strength of geopolymers

Specimen name and heating time (min)	Maximum Strength (MPa)
G-00 (unheated)	5.19
G-10m	5.74
G-15m	6.24
G-20m	7.14

Also the result obtained is shown in figure 9. The compressive strength of 5.19 Mpa was registered for the G-00 (unheated) geopolymer at room temperature. This increased to 5.74Mpa for the G-10m geopolymer that was heated in microwaved for 10 minutes.

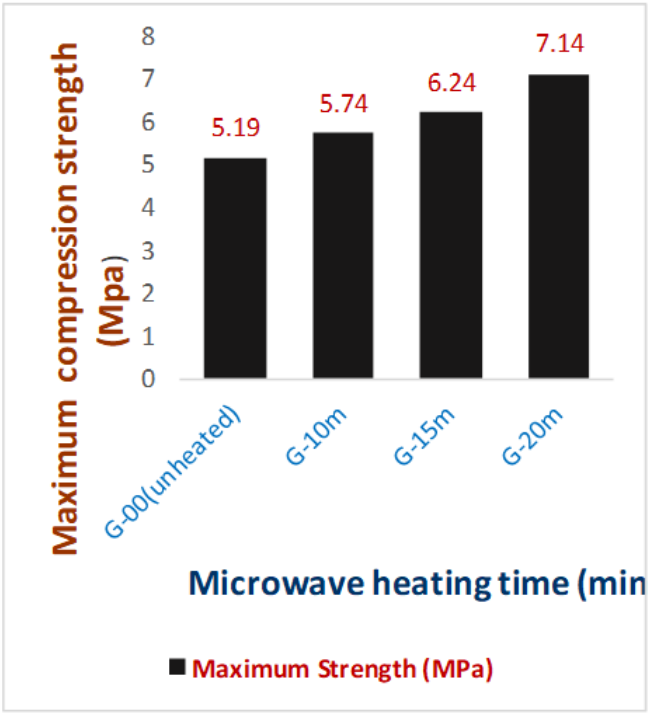

Figure 9: Compressive strength of geopolymers with microwave heating time

Again, the G-15m, which this time was heated in the microwave for 15 minutes yielded a higher compressive value of 6.24 Mpa due to the increasing microwave heating time and subsequently more radiation energy that enhanced the densification and reduced porosity. Finally, the G-20m geopolymer that was also heated in the microwave for 20 minutes registered the highest compressive strength value of 7.14 Mpa.

The high strength developed with the G-20m specimen may be due to the activation level which increases the formation of smaller finer pores and reduces the surface area and developed higher bulk densities. This can be attributed to the increasing microwave heating time that help in directly delivering sufficient energy to the materials through the interaction of the molecules with the electromagnetic fields. This led to formation of the G-20m geopolymer structure that was more hardened.

However, with the room cured geopolymer (G-00m) specimen the activation energy was not motivated and hence resulted in lower reaction rate and low strenght development within the period under investigation. It could be observed that with increasing time and corresponding additional microwave heat radiarion led to improvement in the increase in strength of the geopolymers. This is because microwave radiation heat increases the densification and overcame the geopolymerisation activation energy.

3.1 Thermal conductivity of geopolymers.

The thermal conductivities of the geopolymers were analysed based on steady state method employing the Lee disc thermal conductivity apparatus. The thermal conductivities of both room temperature (Unheated) and microwave heated geopolymers were respectively calculated

and the results are shown in summary, in table 3. Also the bulk densities are included in the table for discussion.

Table 3: Microwave heating time and resulting densities and thermal conductivities

Specimen name	Bulk density (kg/m3)	Thermal conductivity (W/Mk)
G-00 (unheated)	1654.36	0.089
G-10m	1784.53	0.110
G-15m	1972.60	0.128
G-20m	2367.61	0.176

The Thermal conductivity results were plotted against the density for all the samples as illustrated in figure 10. The thermal conductivity keep increasing as the density increases until it reaches the highest value of 0.176 W/Mk and this increase was proportional to incresase in microwave heating radiation time It can be seen from the plots the lowest thermal conductivity of 0.089 w/Mk was obtained at density value of 1654.36 Kg/m3 with the G-00 (unheated) geopolymer at room temperature curing.

Moreover, the density exhibited an increase to 1784.53 Kg/m3 due to the application of microwave radiation heat for 10 minutes on the geopolymer (G-10m) and the resulting thermal conductivity was 0.11W/Mk which pointed an increase in thermal conductivity from the previous value for the unheated geopolymer (G-00). After that, another microwave heating of the G-15m geopolymer, for 15 minutes registered an increase in the density value to 1972.6 kg/m3 and the corresponding thermal conductivity obtained for this geopolymer was 0.128W/Mk, which also demonstrated a significance increase.

The highest value of the thermal conductivity of 0.176 W/Mk obtained was when the density reached the highest value of 2367.61kg/m3 as the geopolymer G-20m was heated in the microwave for 20minutes. This result confirmed the fact that microwave radiation heat increases the density of geopolymers Furthermore, it can also be noted here that the thermal conductivities of geopolymers is depended on the densities of the geopolymers.

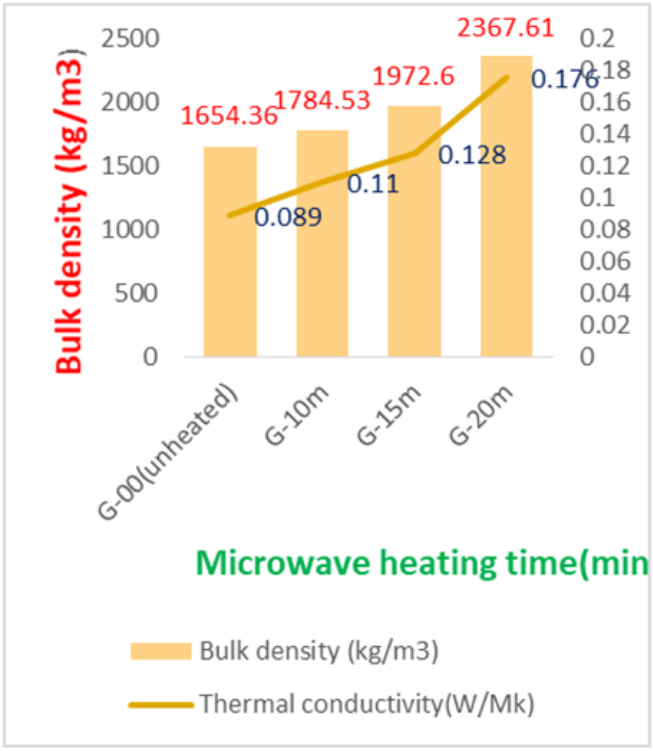

Figure 10: Microwave heating time relationship with density and thermal conductivity

The relationship of porosity with thermal conductivity for the different geopolymers heating is shown in table 4.

Table 4: Porosity and Thermal conductivity of geopolymers

Specimen name and heating time	Thermal conductivity (W/Mk)	Porosity (%)
G-00 (unheated)	0.089	15.2
G-10m	0.110	11.4
G-15m	0.128	9.3
G-20m	0.176	6.6

The figure 11. illustrates the increase of thermal conductivity with decreasing in porosity. This is because as the microwave heat activation energy reduces, the spacing between the particles and hence reduced the surface areas.

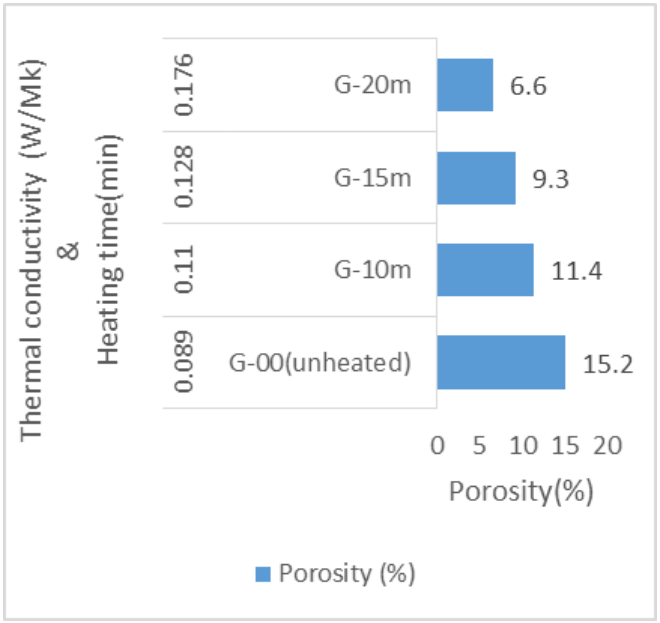

Figure 11: Porosity and thermal conductivity of geopolymers

4. Conclusion

Density

The density obtained in the present work range between 1654.36 Kg/m3 to 2367.61kg/m3 and these are higher than those obtained previously for brick and block of 1200Kg/m3 to 1600 Kg/m3 (Andini, Cioffli, Colangelo, Gricco, Montagnaro and Santoro, 2007). Then considering the current investigation, the results obtained showed that lightweight materials can be produced. It should be noted that for normal cementious materials the density ranges between 1750Kg/m3 to 2400Kg/m3 (Vijai, kumutha and Vishnuram, 2010).

Thermal Conductivity

Again the results obtained for thermal conductivity for G-00 (Unheated) and G-10m (heated geopolymers exhibited lower thermal conductivity compared to the normal materials such the blocks and bricks, which is comparable to the results reported by other researchers (Michael, Alengaram, Jumaat & Kim 2014).

It should be noted that thermal conductivity values of 0.128 and 0.178w/Mk obtained for G-15m and G-20m microwave heated geopolymers respectively, when compared with conventional building block and brick materials, exhibited higher conductivity. This is due to increase in particles densification and reduced porosity. However, this does mean microwave heating could not be employed, but the process could be adapted by modifying the parameters employed such as microwave power and heating durations, batch weight, temperature of specimens and sizes.

Compression Strength

The results of compression strength t of 5.74Mpa, 6.24Mpa and 7.14Mpa obtained from the tests of the G-10m, G-15m and G-20m heated gep9lymers respectively implies that these can be classified as Class II structural concrete with a compression strength between 3.5Mpa and 15Mpa (Michael, et al 2014).

The higher values of compression strengths of the heated geopolymers, showed an indication that microwave geopolymer heating technology has a potential to be used for the building industry.

References

[1] Al-Bakri Abdullah, M. M., Jamaludin, L., Hussin, K., Bnhussain, M., Ghazali, C. M. R., & Ahmad, M. I. (2012). Fly ash porous material using geopolymerization process for high temperature exposure. International Journal of Molecular Sciences, 13 (4), 4388-4395. https://doi. org/10.3390/ijms13044388

[2] Bucham, J. O., Igbax, S. I., & amp; A 2022. Hybrid Aluminium Alloy (Aa6064) Composite Mechanical Properties Investigation.

[3] United International Journal for Research & amp; Technology (UIJRT), 3 (8), pp.44-48.

[4] Andini, S., Cioffi, R., Colangelo, F., Gricco, T., Montagnaro, F. & Santoro, L. (2007). Coal Fly Ash as Raw Material for the manufacture of geopolymer-based products. Waste Management, 28 (2008) 416 423

[5] Chindaprasirt, P., Rattanasak, U., & Taebuanhuad, S. (2013). Role of microwave radiation in curing the fly ash geopolymer. Advanced Powder Technology, 24 (3), 703-707. https://doi. org/10.1016/j. apt.2012.12.005

[6] Deventer, J. S. J. Van, Provis, J. L., Deventer, J. S. J. Van, Keyte, L. M., & Duxson, P. (n. d.). Geopolymers : Structures, Processing, Properties and Industrial Applications.

[7] Graytee, A., Sanjayan, J. G., & Nazari, A. (2018). Development of a high strength fly ash-based geopolymer in short time by using microwave curing. Ceramics International, 44 (7), 8216-8222. https://doi. org/10.1016/j. ceramint.2018.02.001

[8] Haq, E. U., Padmanabhan, S. K., Zubair, M., Ali, L., & Licciulli, A. (2016). Intumescence behaviour of bottom ash based geopolymer mortar through microwave irradiation - As affected by alkali activation. Construction and Building Materials, 126, 951-956. https://doi. org/10.1016/j. conbuildmat.2016.08.135

[9] Leiva, C., Arenas, C., Vilches, L. F., Alonso-Fariñas, B., & Rodriguez-Galán, M. (2015). Development of fly ash boards with thermal, acoustic and fire insulation properties. Waste Management, 46, 298-303. https://doi. org/10.1016/j. wasman.2015.08.027

[10] Michael Yong Jing Liu, U. Johnson Alengaram, Mohd Zamin Jumaat, Kim Hung Mo (2014).

[11] Evaluation of thermal conductivity, mechanical and transport properties of lightweight aggregate foamed geopolymer concrete. Energy and Buildings 72 (2014) 238-245

[12] Mustafa Al Bakri, A. M., Kamarudin, H., Bnhussain, M., Nizar, K., Rafiza, A. R., & Zarina, Y. (2012). The processing, characterization, and properties of fly ash based geopolymer concrete. Reviews on Advanced Materials Science, 30 (1), 90-97.

[13] Nyale, S. M., Babajide, O. O., Birch, G. D., Böke, N., & Petrik, L. F. (2013). Synthesis and Characterization of Coal Fly Ash-based Foamed Geopolymer. Procedia Environmental Sciences, 18, 722-730. https://doi. org/10.1016/j. proenv.2013.04.098

[14] Onutai, S., Jiemsirilers, S., Thavorniti, P., & Kobayashi, T. (2016). Fast microwave syntheses of fly ash based porous geopolymers in the presence of high alkali concentration. Ceramics International, 42 (8), 9866-9874. https://doi. org/10.1016/j. ceramint.2016.03.086

[15] Strydom, C. A., & Swanepoel, J. C. (2002). Utilisation of fly ash in a geopolymeric material. Applied Geochemistry, 17 (8), 1143-1148. https://doi. org/10.1016/S0883-2927 (02) 00005-7

[16] Ul Haq, E., Kunjalukkal Padmanabhan, S., & Licciulli, A. (2014a). Synthesis and characteristics of fly ash and bottom ash based geopolymers-A comparative study. Ceramics International, 40 (2), 2965-2971. https://doi.

org/10.1016/j. ceramint.2013.10.012
[17] Ul Haq, E., Kunjalukkal Padmanabhan, S., & Licciulli, A. (2014b). Synthesis and characteristics of fly ash and bottom ash based geopolymers-A comparative study. Ceramics International, 40 (2), 2965-2971. https://doi. org/10.1016/j. ceramint.2013.10.012
[18] Vijai, K., Kumutha, R. and Vishnuram, B. G. (2010). Effect of types of curing on strength of geopolymer concrete. International Journal of the Physical Sciences Vol.5 (9), pp.1419 -1423, ISSN 1992-1950.
[19] Zhang, Z., Provis, J. L., Reid, A., & Wang, H. (2015). Mechanical, thermal insulation, thermal resistance and acoustic absorption properties of geopolymer foam concrete. Cement and Concrete Composites, 62, 97-105. https://doi. org/10.1016/j. cemconcomp.2015.03.013
[20] Zhuang, X. Y., Chen, L., Komarneni, S., Zhou, C. H., Tong, D. S., Yang, H. M., … Wang, H. (2016). Fly ash-based geopolymer: Clean production, properties and applications. Journal of Cleaner Production. https://doi. org/10.1016/j. jclepro.2016.03.019