# PREPARATION AND CHARACTERIZATION OF KAOLINITE CLAY FOR PRODUCTION OF NATURAL POZZOLAN

Zin Mar Aung[1], Mar Mar Swe[2]

## Abstract

Kaolinite clay has been modified with thermal treatment for application as pozzolanic material in cement. The thermal analysis of kaolinite has been characterized by using Thermogravimetric/ Differential Thermal Analysis (TG/DTA). In the result of TG-DTA, the endothermic peak (dehydroxylation temperature) has been observed at 542.95ºC. Kaolinite clay has been heat-treated at three different temperatures; 600 ºC, 700 ºC, and 800 ºC for two hours duration each. The phase analysis of kaolinite samples has been analysed by using X-ray Diffraction technique (XRD). The pozzolanic activity of metakaolinite samples has been revealed by strength activity index (SAI). Finally, the elemental content of the resultant metakaolinite sample has been analysed by Energy Dispersive X-ray Fluorescence (EDXRF). The XRD result has revealed that the original crystalline microstructure of kaolinite has transformed into an amorphous one after thermal treatment. According to EDXRF analysis, it has been found that kaolinite clay is suitable to be used as cement replacement. Based on the XRD and SAI test results obtained, it is revealed that metakaolinite produced from the treated temperature at 700 ºC for 2 hours was adopted as pozzolanic material for natural pozzolan production.

**Keywords**: Kaolinite, Pozzolanic Material, TG/DTA, XRD, SAI, EDXRF.

## Introduction

Ordinary Portland Cement (OPC) production releases massive $CO_2$ gases and generates $SO_3$ and $NO_x$ gases, which caused the greenhouse effect and incurred serious environmental impacts. On the other hand, large amounts of energy and virgin materials were consumed in cement production. To reduce environmental damage and exploitation of natural resources, the cement industry is actively looking for solutions. Hence, several research activities are directed towards partial or full substitution of Portland cement with the pozzolanic binder in some applications.

In recent years, the use of calcined clays as a pozzolanic material for mortar and concrete has received considerable attention. One of such materials is metakaolinite (MK), which is obtained by thermal treatment of kaolinite clays in the range of 600-800 ºC and mixed with lime or cement, MK acts as a highly reactive pozzolana. The production of calcined clay reduces the amount of energy used and $CO_2$ emissions because the activation temperatures of clay are lower than that of clinker and the decomposition of clays emits water vapor rather than $CO_2$.

Based on these backgrounds, an attempt has been made to utilize the kaolinite clay as construction materials to partially replace the use of cement in making concrete as pozzolanic material. Clay which is suitable to be used as a Pozzolanic material is very widespread and is readily available in almost all regions of the world. According to m geological surveys, kaolinite clay has been found in many areas of Myanmar. Kaolinite clay used in this study has been collected from Taungni Taung in Kyaukpadaung Township, Mandalay Division.

This research focuses on the effect of treated temperature of kaolinite on its microstructure, and the compressive strength of clay-based composite because the clay needs heat treatment before using as cement replacement material. Three tests were carried out to establish the pozzolanity of kaolinite. The TG/DTA analysis highlights the temperature at which the MK formation phase occurred. This does not indicate the exact treated temperature at which the MK with the excellent pozzolans is produced. The first two tests (XRD and SAI) help to identify the exact treated

[1] PhD candidate, Lecturer, Department of Physics, University of Magway
[2] Dr, Lecturer, Department of Physics, University of Yangon

temperature. The third test is the chemical analysis of the MK. While the first two tests particularly seek to determine the exact treated temperature that produces highly reactive pozzolana, the third test was carried out on samples eventually prepared under these conditions. The results of SAI and chemical analysis tests are compared with the ASTM (American Society for Testing Material) standard for calcined natural pozzolan since this is an internationally accepted standard.

## Materials and Methods

### Sample preparation of metakaolinite fine powder

Kaolinite clay was collected from Taungni Taung in Kyaukpadaung Township, Mandalay Division. The raw kaolinite was dried under the sun and ground into a fine powder using pestle and mortar. The kaolinite powder was then sieved with a 200 mesh to obtain a fraction with a particle size of fewer than 74 µm. The kaolinite sample was analysed by Thermogravimetric Differential Thermal Analysis (TG/DTA) before thermal treatment.

The fine kaolinite powder was treated at different temperatures of 600 ºC, 700 ºC, and 800 ºC for two hours each in a laboratory furnace. After treatment, metakaolinite obtained from treatment was allowed to cool down gradually in the furnace to the ambient temperature. Again the treated powder was ground and sieved with a 325 mesh to obtain a fraction with particle size of fewer than 45 µm. Finally, the obtained metakaolinite fine powder was characterized by X-ray Diffraction (XRD), and Energy Dispersive X-ray Fluorescence (EDXRF). Figure 1 shows the flow chart for the preparation of pozzolanic material.

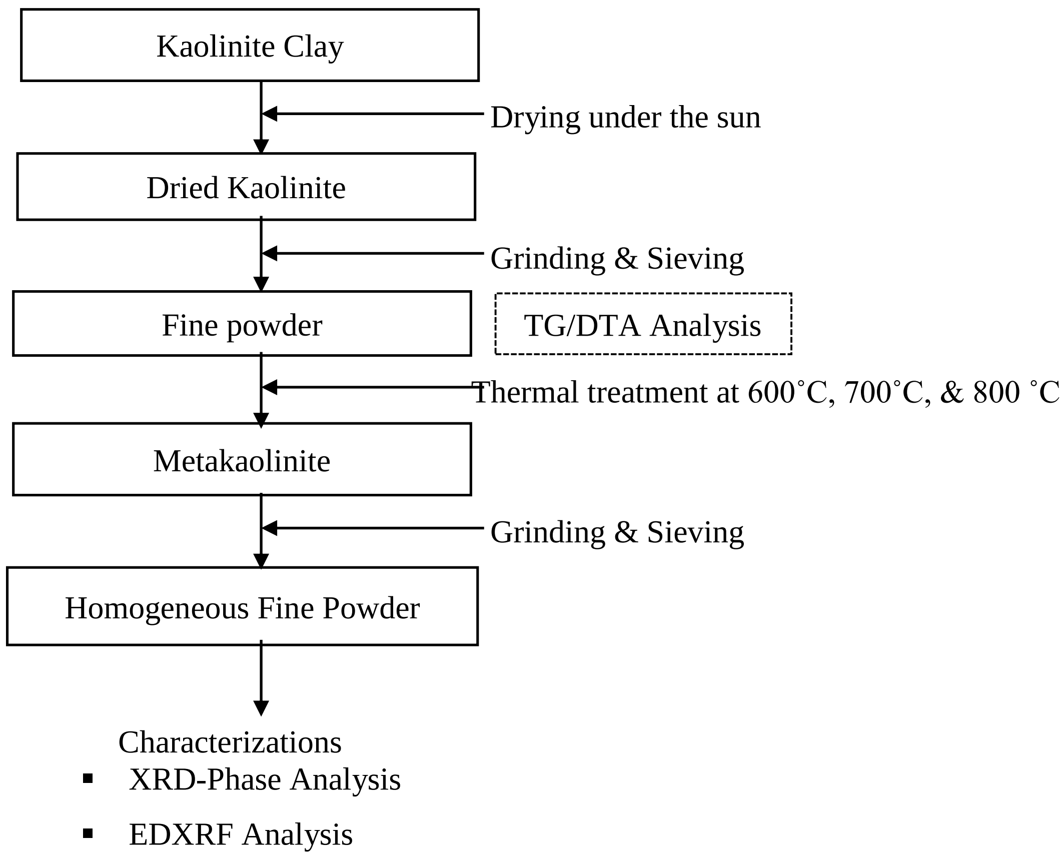



**Figure 1** Flow chart for the preparation of pozzolanic material

**Cement mortar processing for SAI test**

The SAI values of metakaolinite samples with various treated temperatures (600, 700, and 800 ºC) were also determined to support the result of XRD. Figure 2 shows the flow chart for the preparation of mortar cubes. The cement mortars were prepared in line with the requirements of ASTM C109/C109M-08. Table 1 shows the mixed proportions of various ingredients of the mortar specimen. The cement mortar cubes were prepared using a cement: sand ratio of 1:2.75 and a water: binder ratio of 0.48. These served as control cubes. Thereafter, metakaolinite obtained from each treated temperature was combined with cement at 20% cement replacement. This was used with sand in the same ratio to produce its respective mortar test cubes.

Mechanical mixing was used to combine all of the weighed ingredients. After mixing deionized water and cementitious material for 30 s, the sand was gradually added into the solution during the first 30 s of mixing. All of the materials were combined at high speed for the following 30 s, after which the mixer was turned off for 90 s: the material residues on the bowl walls were removed during the first 30 s, and then the mixture was allowed to stand. The mixer was restarted at high speed for an additional 60 s after the pause.

Then the cement mixture was gradually placed into the iron mold, which was made up of 50x50x50 mm cube specimens, at the end of the mixing period. The hand tamping method was used to finish the mortar consolidation in the mold. All of the cube compartments had a layer of mortar around 25 mm thick. In four rounds, the mortar in each cube compartment was tamped 32 times in roughly 10 s each round being at right angles to the other and consisting of eight contiguous strokes. The tamping pressure was exactly right to insure that the molds were evenly filled. After the first layer was tamped in all of the cube compartments, the remaining mortar was poured and tamped as specified for the first layer. After tamping, the mortar was brought to a flat surface and flushed with the top of the mold by sawing the straight edge of the trowel along the length of the mold.

The molds were then placed in a moist environment for 24 h. The specimens were then taken from the mold and cured for 28 days in saturated lime water. The cured specimens were then allowed to dry in the air for 12 h before being tested for their strengths. Finally, the compressive strength values of the mortar cubes were determined using the Compressive Testing Machine. The sides of the cubes were loaded uniformly with a compressive strength machine until fracture appeared. The maximum load in kN at which fracture occurred was recorded and used to calculate the compressive strength as

$$R_c = \frac{1000F_c}{A_r}$$

Where

$R_c$= compressive strength, MPa

$F_c$ = maximum load at which fracture occurs, kN

$A_r$ = area of a face of the cube, $mm^2$

For each crushing test, 2 cubes were used and the average was taken for the compressive strength.

The pozzolanic activity of the metakaolinite samples with various treated temperatures was revealed by strength activity index (SAI) test. The average strength for each temperature was compared with that for control to give the SAI. The metakaolinite sample with the highest SAI value is assessed as natural pozzolan with the highest pozzolanity. This result should agree with the result of XRD. Besides, the SAI for each temperature should satisfy a minimum of 75% as required for natural pozzolans.

**Table 1 Mixed proportions of various ingredients of the mortar specimen**

| **No** | **Materials** | **For Compressive Strength test** | **Remarks** |
|---|---|---|---|
| 1 | Specimen | 50 mm Cube | Materials required for 6 specimens |
| 2 | Cementitious materials ( Cement + Metakaolinite) | 500 gm | |
| 3 | Sand | 1375 gm | |
| 4 | Water | 242 ml | |

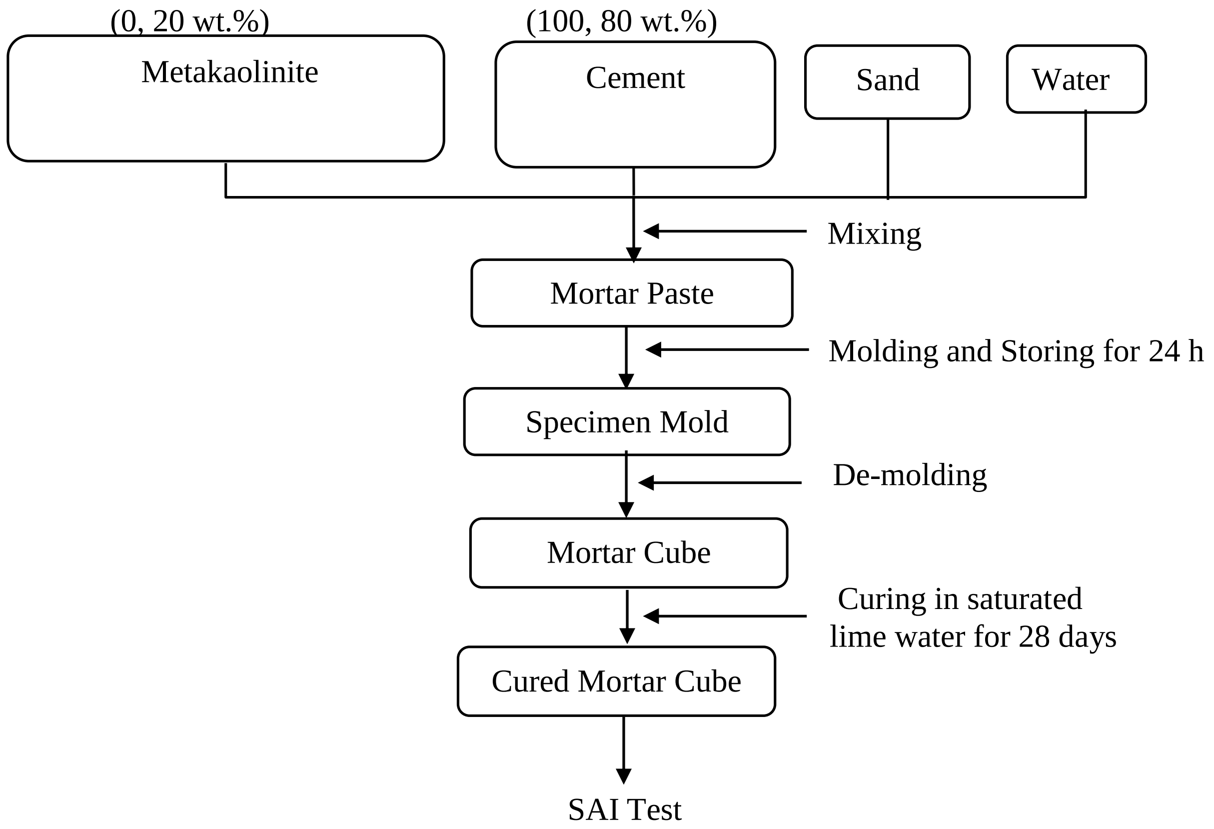



**Figure 2** Flow chart for the preparation of mortar cubes

## Results and Discussion

### TG-DTA Analysis

Thermal behavior of starting kaolinite clay was analysed using TG-DTA and shown in Figure 3. In the TGA curve, the material has a total mass loss of 13 % at 600 ºC. The mass loss was 2.11 % in the temperature range 37. 45-480 ºC, corresponded with the release of free or absorbed water from kaolinite surface. The second significant mass loss in the TGA curve was 10.86% in the temperature range 480-590ºC which was associated with dehydroxylation of kaolinite and formation of disordered material metakaolinite. The observed endothermic peak with a maximum at 542.95 ºC may be attributed to the dehydroxylation process.

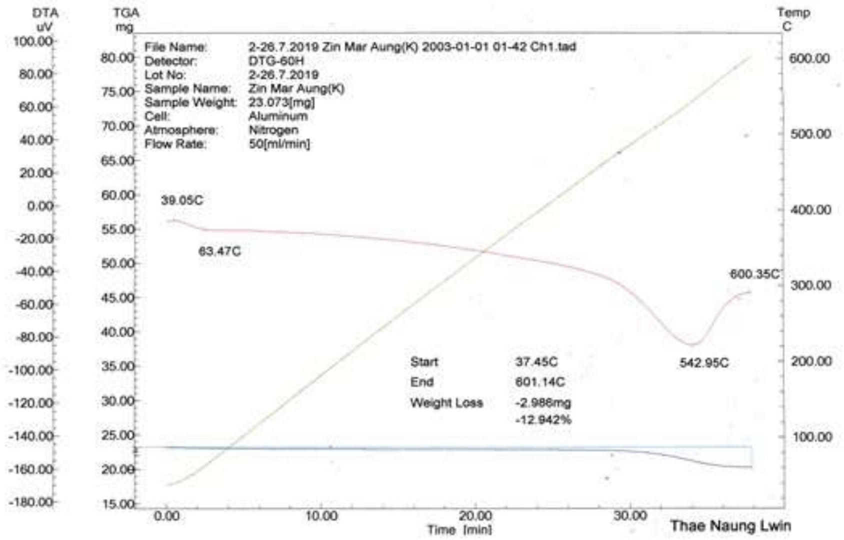



**Figure 3** TG/DTA curve of Kaolinite

## XRD Analysis

The XRD patterns of raw and treated samples with thermal treatment at 600 ºC, 700 ºC, and 800 ºC are shown in Figure 4. It is seen that the mineral constituent of the starting clay is kaolinite. Based on XRD result obtained, it has been observed that the original crystalline microstructure of the kaolinite has transformed into an amorphous one after thermal treatment. Importantly, much broader diffused peaks with maximum intensity at 2θ = 20.910 are observed at treated temperature 700 ºC. The amorphous structure is a characteristic of the excellent pozzolanic activity of pozzolan material. Therefore, the treated sample at 700 ºC may have potential application for pozzolan production.

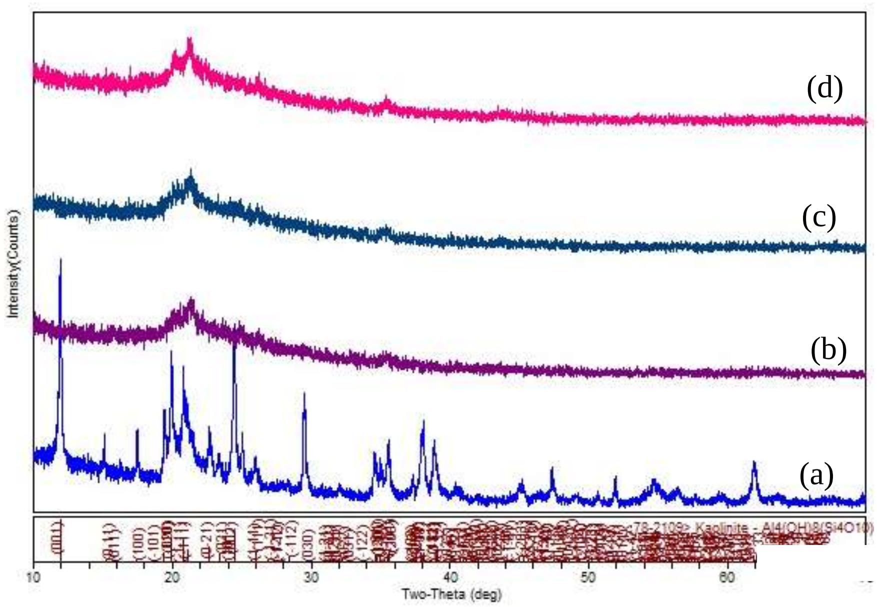
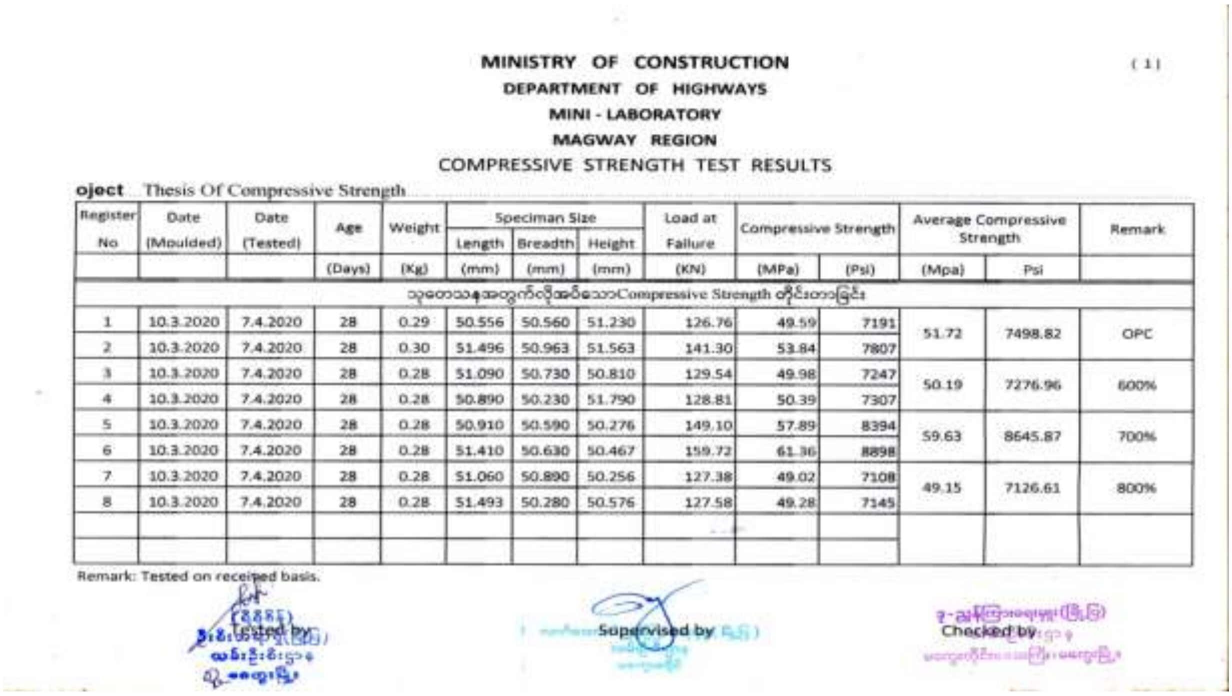



**Figure 4** XRD of kaolinite samples: (a) raw kaolinite; (b) thermally treated at 600 ºC; (c) thermally treated at 700 ºC; (d) thermally treated at 800 ºC

## SAI Test

The SAI test was used to support the result of the XRD test. The strength index provides the pozzolanic activity of MK samples with various treated temperatures (600, 700, and 800 ºC). The photograph of the Ministry of Construction's certificate was shown in Figure 5. Table 2 shows the compressive strength and strength activity index of specimens and the effect of treated temperature on the SAI of MK samples was also compared in Figure 6. According to the ASTM specification, the SAI for each temperature should satisfy a minimum of 75% as required for natural pozzolans. The results obtained show that all of the MK samples with various treated temperatures are assessed as pozzolans. The highest amount of SAI value was found in MK sample with the treated temperature at 700 ºC. The results obtained from the SAI test are in good agreement with XRD results.



MINISTRY OF CONSTRUCTION
DEPARTMENT OF HIGHWAYS
MINI - LABORATORY
MAGWAY REGION
COMPRESSIVE STRENGTH TEST RESULTS

( 1 )

oject Thesis Of Compressive Strength

| Register No | Date (Moulded) | Date (Tested) | Age | Weight | Speciman Size | | | Load at Failure | Compressive Strength | | Average Compressive Strength | | Remark |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Length | Breadth | Height | | | | | | |
| | | | (Days) | (Kg) | (mm) | (mm) | (mm) | (KN) | (MPa) | (Psi) | (Mpa) | Psi | |
| [illegible] Compressive Strength [illegible] | | | | | | | | | | | | | |
| 1 | 10.3.2020 | 7.4.2020 | 28 | 0.29 | 50.556 | 50.560 | 51.230 | 126.76 | 49.59 | 7191 | 51.72 | 7498.82 | OPC |
| 2 | 10.3.2020 | 7.4.2020 | 28 | 0.30 | 51.496 | 50.963 | 51.563 | 141.30 | 53.84 | 7807 | | | |
| 3 | 10.3.2020 | 7.4.2020 | 28 | 0.28 | 51.090 | 50.730 | 50.810 | 129.54 | 49.98 | 7247 | 50.19 | 7276.96 | 600% |
| 4 | 10.3.2020 | 7.4.2020 | 28 | 0.28 | 50.890 | 50.230 | 51.790 | 128.81 | 50.39 | 7307 | | | |
| 5 | 10.3.2020 | 7.4.2020 | 28 | 0.28 | 50.910 | 50.590 | 50.276 | 149.10 | 57.89 | 8394 | 59.63 | 8645.87 | 700% |
| 6 | 10.3.2020 | 7.4.2020 | 28 | 0.28 | 51.410 | 50.630 | 50.467 | 159.72 | 61.36 | 8898 | | | |
| 7 | 10.3.2020 | 7.4.2020 | 28 | 0.28 | 51.060 | 50.890 | 50.256 | 127.38 | 49.02 | 7108 | 49.15 | 7126.61 | 800% |
| 8 | 10.3.2020 | 7.4.2020 | 28 | 0.28 | 51.493 | 50.280 | 50.576 | 127.58 | 49.28 | 7145 | | | |

Remark: Tested on received basis.

Tested by Supervised by Checked by

**Figure 5** The photograph of the Ministry of Construction's certificate

**Table 2** Compressive strength and strength activity index of specimens

| Sample Name | Compressive Strength (MPa) | SAI (%) |
|---|---|---|
| OPC | 51.72 | - |
| MK-600 | 50.19 | 97.04 |
| MK-700 | 59.63 | 115.29 |
| MK-800 | 49.15 | 95.03 |

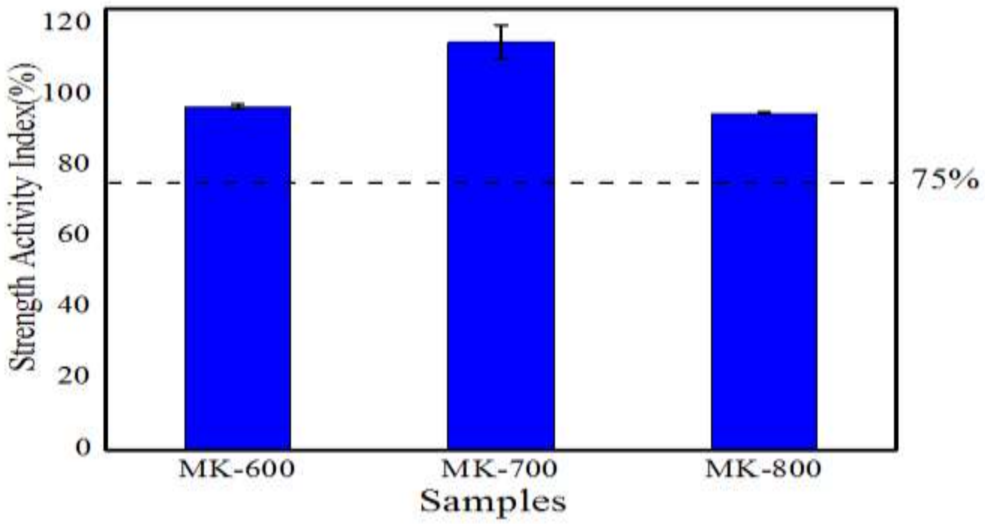



**Figure 6** Effect of treated temperature on strength activity index of metakaolinite samples (dotted line: reference for minimum requirement of natural pozzolans)

**EDXRF Analysis**

The chemical composition of MK at treated temperature 700 ºC is shown in Table 3. The result was compared with the requirements for classifying a material as a pozzolan as established by ASTM. The result of the chemical analysis showed that the resultant MK sample passed the ASTM requirements with the constituents of $SiO_2$, $Al_2O_3$, and $Fe_2O_3$ totaling 98.581%. This is more than the required 70% minimum. Another condition was seen to be satisfied as well signifying the suitability of the heat-treated clay as a pozzolan. Therefore, the EDXRF analysis has confirmed that kaolinite from Taungni Taung, Kyaukpadaung Township is suitable to be used as pozzolanic material.

**Table 3 The concentration of detectable oxides in metakaolinite sample**

| Oxides | Metakaolinite (%) | | ASTM C618-05 requirement for pozzolans |
|---|---|---|---|
| $SiO_2$ | 58.329 | 98.581 | $SiO_2+Al_2O_3+Fe_2O_3 \geq 70\%$ |
| $Al_2O_3$ | 40.048 | | |
| $Fe_2O_3$ | 0.204 | | |
| $SO_3$ | 0.762 | | 4% max |
| CaO | 0.106 | | |
| Others | 0.549 | | |

## Conclusion

This study investigates the effect of treated temperature on kaolinite clay for natural pozzolan production. In the result of TG/DTA, the endothermic peak (dehydroxylation temperature) has been observed at 542.95 ºC. Moreover, the XRD results clearly showed that the originally crystalline structure of kaolinite has transformed into an amorphous one after thermal treatment. Therefore, the heating time duration and treated temperature range play an important role in this study. EDXRF result has proved that metakaolinite is suitable for pozzolanic material due to its high content of silica and alumina. Based on the XRD and SAI test results obtained, it is concluded that metakaolinite produced from the treated temperature at 700 ºC for two hours may have the potential to be applied as pozzolanic material for natural pozzolan production.

## Acknowledgements

I would like to express appreciation to Dr Khin Maung Oo, Rector, University of Magway, for his kind permission and encouragements to carry out this work. I would like to express my profound thanks to Professor Dr Sandar Maung, Head of Department of Physics, University of Magway, for her kind permission and encouragements to carry out this work.

## References

Aliyu,U., Muhammad, B., and Nurabala, (2018), "Effect of Amorphous Silica Ash Used as a Partial Replacement for Cement on the Compressive and Flexural Strengths Cement Mortar." Morozov, IOP Conf Series: Earth and Environmental Science 140, 012124.

Gbenga,M.A., Olasunkanm ,A., and Adekitan, (2016), "Characterization of Ajebo Kaolinite clay for production of natural pozzolan." World academy of science, engineering and technology, International journal of civil and environmental engineering, vol.10, No.9

Ghassan,K.A., Mouin,A., and Panagiotis,G.A., (2013), "Natural Pozzolan as a Partial Substitute for Cement In Concrete." The Open Construction and Building Technology, 7, 33-42

Kaleeswari,G., Hanalakshmi,G., and Manikandan,N., March (2016), "Clay as a Partial Replacement of Cementitious Material in Cement." International Journal of Advanced Research in Biology Engineering Science and Technology (IJARBEST) Vol.2, Issue3.

Yang,H., and Jie, D., (2018), "Preliminary Investigation of Pozzolanic Properties of Calcined Waste Kaolin." ISSN 1392-1320 Material Science Vol.24, No.2.