



**Utilization of Dolomite with Different Percentage of
Fly Ash Replacement as Alkali Activated Materials for
Bricks Application**

by

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In the name of Allah The Most Gracious and The Most Merciful

With the Selawat and Salam to Prophet Muhammad S.A.W

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LIST OF SYMBOLS

-	Negative
%	Per cent
°C	Celsius
+	Positive
cm	Centimetre
kg	Kilogram
M	Molarity
m	meter
MPa	Mega pascal
°	Degree
θ	Theta

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LIST OF ABBREVIATIONS

Al	Aluminate
Al ₂ O ₃	Alumina Oxide
AlO	Aluminum Oxide
ASTM	American Society for Testing and Materials
Ca	Calcium
CaCO ₃	Calcium Carbonate
CaO	Calcium Oxide
CASH	Calcium Aluminate Silicate Hydrate
CO ₂	Carbon Dioxide
CSH	Calcium Silicate Hydrate
D	Dolomite
DSC	Differential Scanning Calorimetry
EDX	Energy Dispersive X-Ray Spectroscopy
FA	Fly Ash
Fe ₂ O ₃	Ferum Oxide
FTIR	Fourier Transform Infrared
GGBS	Ground Granulated Blast Furnace
H ₂ O	Water
ICDD	International Centre for Diffraction Data
K	Potassium
KOH	Potassium Hydroxide
M	Molar
MgO	Magnesium Oxide
MK	Metakaolin
Na	Sodium
Na ₂ SiO ₃	Sodium Silicate
NaOH	Sodium Hydroxide
NASH	Sodium Alumina Silicate Hydrate
OH	Hydroxide
OPC	Ordinary Portland Cement

SEM	Scanning Electron Microscope
Si	Silicate
SiO	Silicon Oxide
SiO ₂	Silicon Dioxide
TGA	Thermogravimetric Analysis
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

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Penggunaan Dolomit dengan Peratusan Berbeza Penggantian Abu Terbang sebagai Bahan Aktif Alkali untuk Aplikasi Bata

ABSTRAK

Sisa dolomit dari industri terutamanya kuari di Malaysia dan di seluruh dunia didapati mengalami peningkatan setiap tahun. Dolomit merupakan sisa atau bahan buangan daripada industri batu hiasan, industri simen dan industri dolomit itu sendiri. Objektif utama kajian ini dijalankan adalah untuk mengkaji potensi penggunaan dolomit sebagai bahan mentah alkali yang diaktifkan menggunakan larutan alkali khususnya dalam sintesis geopolimer. Berdasarkan rekod, masih tiada lagi kajian tentang dolomit sebagai sumber utama bahan mentah (60% hingga 100%) yang diaktifkan oleh larutan alkali atau geopolimer. Penyelidikan yang teliti terhadap penggunaan dolomit adalah amat perlu agar ia dapat digunakan dan membantu secara teknikal untuk kegunaan dalam industri pembinaan. Menerusi kajian ini, dolomit yang diaktifkan oleh larutan alkali, geopolimer berasaskan dolomit/abu terbang, dan bata telah dihasilkan dengan menggunakan proses geopolimerisasi. Abu terbang didapati berjaya menambahbaik reaktiviti serta kekuatan bagi geopolimer ini. Abu terbang telah dimasukkan sebagai pengganti pengisi disebabkan tindak balas dolomit yang rendah selepas penyiasatan awal dilakukan dalam kajian ini. Parameter yang digunakan untuk mencari kondisi campuran yang optimum dalam penghasilan bahan alkali yang diaktifkan merangkumi faktor kepekatan NaOH, nisbah pepejal kepada cecair (S/L) serta nisbah natrium silikat kepada nisbah natrium hidroksida ($\text{Na}_2\text{SiO}_3/\text{NaOH}$). Kesemua ciri-ciri alkali yang diaktifkan telah dinilai dan diperolehi menerusi ujian kekuatan mampatan, pembelahan Sinar-X (XRD), mikroskop pengimbas elektron (SEM), dan jelmaan Fourier inframerah (FTIR). Sementara itu bata daripada geopolimer berasaskan dolomit/abu terbang telah dihasilkan berdasarkan reka bentuk campuran optimum yang telah diperolehi menerusi kajian terhadap geopolimer berasaskan dolomit/abu terbang (D/FA) manakala nisbah bahan mentah kepada pasir bagi bata yang telah digunakan ialah 1:3. Prestasi bata D/FA dianalisis dari segi kekuatan mampatan, penyerapan air, dan daya tahan terhadap pendedahan kepada suhu yang tinggi. Daripada kajian ini, kekuatan mampatan yang optimum telah diperolehi melalui penggunaan kepekatan 22M NaOH, nisbah S/L 2.5, dan nisbah $\text{Na}_2\text{SiO}_3/\text{NaOH}$ 2.5. Daripada pencirian fasa menggunakan XRD, puncak C-A-S-H diperolehi daripada dolomit yang diaktifkan oleh alkali dan C-A-S-H + N-A-S-H bagi geopolimer berasaskan D/FA yang menentukan kehadiran gel geopolimer melalui proses geopolimerisasi. Kekuatan geopolimer yg dihasilkan juga didapati tinggi dan sesuai digunakan dalam sektor pembinaan. Ketumpatan untuk semua D/FA geopolimer juga boleh diklasifikasikan sebagai ringan menurut spesifikasi ASTM C55-11. Selain itu, bata yang dihasilkan menunjukkan peningkatan kekuatan dengan pengurangan nisbah D/FA dimana kekuatan maksimum mencapai 61.24 MPa. Bagi kadar penyerapan air oleh bata, julatnya bermula dari 6.89% hingga 10.34% di mana kurang daripada had yang ditetapkan (17%) bagi spesifikasi ASTM C90. Bata D/FA ini juga didapati mampu bertahan pada suhu sehingga 1000°C tetapi mengalami perubahan pada kekuatannya. Melalui kajian ini, bata D/FA boleh dikatakan memiliki ciri dan proses pembuatan yang lebih baik berbanding bata OPC.

Utilization of Dolomite with Different Percentage of Fly Ash Replacement as Alkali Activated Materials for Bricks Applications

ABSTRACT

The dolomite waste from industry especially quarry increased every year in Malaysia and worldwide. Dolomite basically is waste obtained from the decorative stone industry, cement industry and dolomite industry itself. The main objective of this study was to investigate the utilization of dolomite as alkali activated specially in geopolymers synthesis. Based on the records, there has been no study yet on dolomite as main source (60% to 100%) for alkali activated or geopolymer. These need meticulous investigations in order to be used widely through technically viable for highly performance of cements in construction industry. This study has been conducted to produce alkali activated dolomite and dolomite/fly ash geopolymer and bricks using the geopolymerization process. Fly ash was found to improve the reactivity and strength of the geopolymer. Fly ash was incorporated as replacement of filler due to the low reaction of dolomite after initial investigation on this study. The parameter used to find the optimum mix design of geopolymer paste production include the NaOH concentrations, ratios of solid to liquid (S/L) as well as sodium silicate to sodium hydroxide ($\text{Na}_2\text{SiO}_3/\text{NaOH}$) ratios. The properties of all the geopolymer were evaluated using compressive strength, X-Ray diffraction (XRD), scanning electron microscope (SEM), and Fourier transform infrared (FTIR). Meanwhile, for mix design of dolomite/fly ash geopolymer bricks, the best optimum mix design for the dolomite/fly ash (D/FA) geopolymer was used along with the dolomite/fly ash ratio while the raw materials to sand ratio for bricks used was 1:3. The performance of the alkali activated D/FA bricks were analyzed in term of compressive strength, water absorption, and resistivity towards high temperature exposure. From this study the optimum compressive strength was achieved at NaOH concentration 22M, ratio of S/L at 2.5, and $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio at 2.5. From the phase characterization using XRD, the existence of C-A-S-H peak were obtained from alkali activated dolomite and C-A-S-H + N-A-S-H peak for D/FA geopolymer which determined the existence of geopolymer gel through geopolymerization process. The strength of the D/FA geopolymer obtained in this study is also high and suitable to be used for construction applications. The density of all the D/FA geopolymer can also be classified into lightweight according to ASTM C55-11. Besides that, for bricks produced using D/FA, showed an increment in strength with the decreasing of D/FA ratio where the maximum strength was up to 61.24 MPa. As for the water absorption for the D/FA bricks, the range was 6.89% to 10.34% which less than the limit of (17%) of ASTM C90 specification. The D/FA bricks were also found to be able to withstand temperature up to 1000°C but had undergone a change in its strength. Based on the study, D/FA bricks were considered to have better properties and manufacturing process compared to the OPC bricks.

CHAPTER 1

INTRODUCTION

1.1 Research Background

Research works carried out so far in the development of alkali activated cement based binders has shown much interest and has already been investigated. The use of alkali activated cement as an environmentally friendly alternative to Portland cement is becoming more popular (Pacheco-Torgal et al., 2017). The alkali activation of waste materials (especially those coming from industrial and mining activities) has become an important area of research background due to its possibility to use these materials to synthesize inexpensive and ecologically sound cement like construction materials (Palomo et al., 1999a, Attanasio et al., 2018).

Geopolymers are amorphous to semi-crystalline three-dimensional silica-aluminate materials. They are prepared by mixing and activating the aluminosilicate sources with alkali liquid (sodium hydroxide and/or sodium silicate) and curing at a moderate temperature (Temuujin et al., 2012, Davidovits, 2018). Geopolymer usually divided into sodium alumina silicate hydrate (N-A-S-H), calcium alumina silicate hydrate (C-A-S-H), and calcium silicate hydrate (C-S-H). Recently discovered geopolymers are materials produced by alkali activation of aluminosilicate raw materials, which were transformed into a reaction product by geopolymerization, in a high pH environment and hydrothermal conditions at a relatively low temperatures

(which up to 120 °C). Geopolymerization reaction involves the dissolution and condensation process that occurs at room temperature. Considering the fact that the organic thermal insulation materials widely used today are flammable and the inorganic thermal insulation materials need complex processing conditions and high sintering temperature, which is high in manufacturing cost, the geopolymers material has good application potential as thermal insulation material in some situations (Feng et al., 2015). Due to the low energy requirements of production from common raw materials and their inflammability at high temperatures, these compounds (geopolymers) are attracting increasing interest as ecologically friendly fireproof building materials, sound and heat insulators and materials for encapsulating hazardous wastes for storage or disposal (Lyon, 1996, Khater, 2014).

Use of alkali activated and geopolymer terms normally classified by the calcium (Ca) and alumina (Al) content in source materials and system. The systems that contain high Ca content with less Al content were called alkali activated. In contrast, systems which contain high Al with less Ca content were called geopolymer. Figure 1.1 shows the classification of materials depends on Ca and Al content in the binder systems. Based on Figure 1.1, it can be clearly observed that geopolymer are alkali activated. However, not all alkali activated are geopolymers. The increment of Al content in the system will lead to a change from alkali activated to geopolymers region. Therefore, this has resulted in the application of geopolymer terms to the binder system.

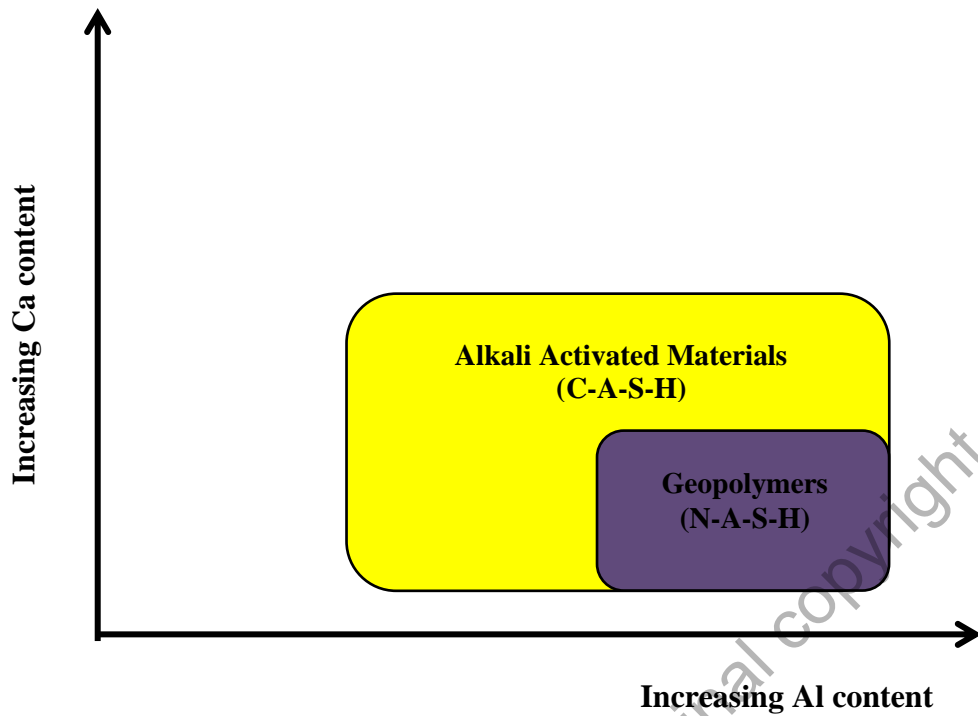


Figure 1.1: classification of materials depends on Ca and Al content in the binder systems (Deventer et al., 2012)

Various source materials have been utilized in the manufacturing of geopolymers such as fly ash, kaolin, metakaolin, slag and the newest is dolomite. geopolymers are classified as one of the green materials which contain many exceptional properties such as high compressive strength, low shrinkage, and excellent fire and acid resistant (Liyana et al., 2013, Glid et al., 2017). The source materials can also be combined in order to obtain desired properties for the geopolymer product.

Heah et al., (2012) in his study used kaolin as aluminosilicate source for geopolymer. They observed the influence of solids-to-liquid (S/L) and alkali activator ratios on kaolin-based geopolymers. They found that compressive strength was influenced by both S/L and $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratios and the geopolymer composites strength increased with ageing day. Both these ratio have also affected the workability of the mixes. Besides, the kaolin geopolymers produced good volume stability in water.

Mo et al., (2014) successfully studied the effect of curing temperature on geopolymerization of metakaolin based geopolymers. The results indicated that curing at elevating temperature will accelerate the polymerization, dissolution, reprecipitation processes of the geopolymerization reaction. The optimum curing temperature of geopolymers is around 60 °C at where the geopolymers samples present the best mechanical properties with the compressive strength of 97.95 MPa after been cured for 7 days.

The need for environmentally friendly construction materials (especially in the structure of the building and wall that insulate heat) to sustain the development is a critical environmental issue at this era in the construction industry. The production of bricks and concrete in industry is said to be one of the contributors of the global warming. This fact is due to the widely use of Portland cement as the main component in the production that was said to be responsible for about 6% of the CO₂ emission worldwide (Shaikh, 2013, Kroehong et al., 2018). Using geopolymers materials as concrete and bricks will be able to offer comparable performance to the traditional cementitious binders in a wide range of applications, but with the extra advantage of lower greenhouse emissions (Davidovits, 1994, Fernández-Jiménez & Palomo, 2009, Sofi et al., 2007, Uddin & Saraswathy, 2018).

Mishra et al., (2015) investigated the incorporation of lime along with fly ash in the geopolymer bricks. The study indicated that the compressive strength of the bricks decreased with the decreasing amount of lime with the same fly ash amount. It was also noticed that the water absorption of the lime-fly ash bricks increased as the amount of lime decreased.

In this study, dolomite has been utilized to manufacture alkali activated dolomite. The use of dolomite in geopolymers field is still new and still at the early stage of study. The dolomites were also utilized along with fly ash as raw materials to produce geopolymer bricks as an application for this study.

1.2 Problem Statements

The waste material from industrial has been increasing each year. Dolomite has a potential to be used as raw material for alkali activated. Unfortunately, it raises less attention from researcher due to lack of information about alkali activated dolomite. These lacks of information become the major barrier in the study of alkali activated dolomite. Dolomite also has low reactivity when used as alkali activated.

Previous researches show that dolomite was only used as replacement or filler for other raw materials in alkali activated (geopolymer). Dolomite previously used only as an addition to other geopolymer. The study done by Aboulayt et al., (2017) used dolomite as an addition to metakaolin based geopolymer. In the investigation, it was found that the incorporation of dolomite in replacement of metakaolin (up to 20 wt%) seem to give significant effect to performance of the matrix thus affect the strength of the geopolymer. Study of the influence of dolomite on the mechanical properties of boiler ash geopolymers paste was carried out by (Zarina et al., 2014). Result from the study shown that the addition of 4% of dolomite into geopolymer paste has the maximum compressive strength (7.3 MPa) compared to other filler percentage. However, the use of dolomite alone in alkali activated especially geopolymers was not

investigated in details. It is important to study the basic material to address how much dolomite can stand alone and in the production of alkali activated.

Dolomite with a comparatively lower reactivity requires sufficient time for interactions to occur and it is important for researchers to investigate the extent of geopolymerization by using dolomite. Since the reaction of dolomite is slow, characterization (microstructural analysis, phase analysis and functional group identification) of dolomite can be done with adequate time interval. Replacement of dolomite material by using other source material is also necessary to improve the reactivity thus led to the strength of the alkali activated geopolymer.

Alkali activated (geopolymer) can be applied in many fields especially in the construction sector. One of the potential applications is in the bricks production. Recently, there are some researchers conducted a study regarding the use of geopolymer in the bricks production. Some of them combined fly ash with other raw materials to produce geopolymer bricks. Among them, Laila et al., (2016) is one of the researchers who has successfully carried out a study on geopolymer bricks. The study used bottom ash along with fly ash as raw material for geopolymer bricks. The investigation found that the compressive strength increased with the increasing of fly ash amount. Another research on the geopolymer bricks made by Mishra et al., (2015) which incorporated lime along with fly ash in the geopolymer bricks. The study indicated that the compressive strength of the bricks decreased with the decreasing amount of lime with same fly ash amount. However, the use of dolomite incorporated with fly ash in geopolymer bricks has not been studied in details. It is also noticed that previous study on other raw materials incorporated with fly ash in geopolymer bricks

(with a ratio of 40% fly ash and 60% of other raw material) produced only acceptable strength and has shown slow setting time.

By using dolomite, it is possible to enhance the fundamental theory of alkali activated especially geopolymers field. The effect of modification raw dolomite by hybridizing it with other aluminosilicate source was also investigated to assess the possibility to improve the reactivity of dolomite towards geopolymerization reaction.

1.3 Objective

This research aimed to study the utilization of dolomite as alkali activated specially in geopolymers synthesis. The properties and characteristic of dolomite geopolymers at various mixing conditions were investigated. The objectives of the study are:

1. To characterize the alkali activated dolomite towards non-loading application based on strength.
2. To determine the mix design optimization of dolomite/fly ash geopolymer based on strength.
3. To determine the effect of different percentage raw materials replacement and different temperature exposure on geopolymer composites bricks.

1.4 Scope of Study

This study is focusing on the utilization of waste material from the dolomite industry, which is dolomite as major raw material for geopolymer. The fly ash was also used as a modification and replacement in the dolomite geopolymer. The characterization of dolomite and fly ash is analysed using X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), and Scanning Electron Microscope (SEM).

In order to develop dolomite based geopolymer, various mix designs were carried out and the optimum mix design is defined by geopolymer samples to contribute to high compressive strength. The parameter included in the mix design of the geopolymer paste is NaOH concentration, solid/liquid ratio, and sodium silicate/sodium hydroxide (NaOH) ratio. The characterization of D/FA based geopolymer was also evaluated using XRD, FTIR, XRF, and SEM.

In order to produce bricks using dolomite/fly ash materials, various ratios of dolomite and fly ash amount were used. The production of D/FA geopolymer bricks is carried out to study the strength properties of this application. The strength of the bricks were determined by compressive strength while the water absorption of the bricks was also investigated by using water up to seven days. The temperature resistant properties of D/FA geopolymer evaluated using temperature exposure in furnace.