



Calcined kaolin as cement replacing material and its use in high strength concrete



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HIGHLIGHTS

- Highly reactive metakaolin was produced by calcination of Malaysian kaolin.
- Optimum condition for the calcination is 800 °C (1472 °F) and 3 h duration.
- 15% is optimum content of produced metakaolin based on compressive strength.
- Higher mechanical properties of metakaolin concrete better than silica fume.
- The cost of metakaolin is about 33% lesser than commercially available silica fume.

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ABSTRACT

This experimental investigation has been carried out to examine the effects of temperature and duration on Malaysian kaolin to form highly reactive metakaolin. Metakaolin has been produced through the calcination process at temperatures 600–800 °C (1112–1472 °F) for the duration from 1 to 5 h. X-ray diffraction and loss on ignition are the usual process to identify the optimum temperature and duration of calcination required. In this study, to confirm the reactivity of metakaolin, compressive strength, splitting tensile and flexural strength of concrete at the age of 7, 28, 56 and 90 days have been investigated using locally produced metakaolin as cement replacing material. It was found that locally produced metakaolin enhance the mechanical properties of concrete and compressive strength is about 5% higher than silica fume concrete at the age of 28 days. This study divulges that calcination at 800 °C (1472 °F) for 3 h is the most suitable condition to convert kaolin into highly reactive metakaolin.

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1. Introduction

Kaolin is widely occurring white clay resulting from natural decomposition of feldspar and is mainly used in the manufacturing of porcelain, filler in the paper, textiles and as absorbent in medicines [1]. In Malaysia, around 11.2 million metric tons (25.2 million lbs.) of kaolin reserves have been identified in the states of Perak, Johor, Kelantan, Selangor, Pahang and Sarawak [1]. Production of kaolin has been recorded as 5,87,508 metric tons (13,20,836 lbs.) in 2007 which was 72% more than produced in 2006 from over 30 active kaolin mines [1].

When pure or refined kaolin is heated to a temperature between 600 and 850 °C (1112–1562 °F), metakaolin is formed [2–5]. Metakaolin is a highly reactive pozzolan, nonetheless its

physical and chemical characteristics greatly depend on the raw kaolin material used, temperature during calcination and finishing process (Rapid cooling after calcination) [6]. The earliest large-scale use of metakaolin in concrete dates back to 1960s; however, the variations such as mixing methodology, mixing proportions and the quality of metakaolin had delayed the detection of metakaolin as a mineral admixture till 1990 [6]. Several researchers [7–10] reported that the addition of metakaolin in concrete has no drawback. In 1991, Larbi and Bijen [8] demonstrated that metakaolin completely eliminates calcium hydroxide (Ca(OH)₂) or portlandite in a cement matrix. Though, metakaolin reduces the level of portlandite in concrete, pH remains stable above 12.5 [6]. Around two decades earlier, Martin [11] and Wild et al. [12] carried out investigations on metakaolin concretes. Martin [11] reported that the presence of metakaolin in concrete increases the compressive strength up to 110 MPa (16 ksi) with superplasticizer to overcome the higher water requirement [6]. While, Wild et al. [12] observed that the optimum OPC replacement with metakaolin is 20% (by weight) with superplasticizer dosage of 2.4% with respect to the

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binder weight. Metakaolin requires lesser dosage of superplasticizer as compared to silica fume [13] and enhances the mechanical properties [14] and durability of concrete [15]. Recently, Duan et al. [16] mentioned that the use of metakaolin has positive effects on the pore structure and interfacial transition zone (ITZ) enhancement of concrete at later ages in comparison to the silica fume and slag concretes.

In literature, several studies are available about the optimum temperature and duration of calcination of kaolin in the different part of the world [2–5]. In 2003, Shvarzman et al. [2] investigated the effect of degree of dehydroxylation on the pozzolanic activity of kaolinite. It was observed that 95% dehydroxylation achieved at the temperature 450–570 °C (842–1058 °F) and a moderate increase in the dehydroxylation was observed between 570 and 700 °C (1058–1292 °F). The activity strength index (i.e. ratio of cube compressive strengths of mortar of 20% admixture to 100% cement) of kaolinite depends on the degree of dehydroxylation and the amorphous phase; however, amorphous phase content up to 55% is sufficient for the kaolinite material to be considered as active pozzolan [2]. In 2009, the relationship between pozzolanic activity and degree of dehydroxylation was investigated by Bich et al. [3], who calcined the raw kaolin material between 650 and 850 °C (1202–1562 °F) for the duration of 1/2, 3/4, 1, 5 and 15 h. They [3] reported the achievement of the highest pozzolanic activity with more than 95% degree of dehydroxylation. Moreover, longer calcination time (5 or 15 h) decreases the pozzolanic activity [3]. In 2005, Badogiannis et al. [4] investigated the Greek kaolin and determined that calcination at 650 °C (1202 °F) and 850 °C (1562 °F) is sufficient to convert low alunite content kaolin and high alunite content kaolin into metakaolin, respectively. The differential thermo gravimetric (DTG) curve of kaolin of high alunite content showed three stages of mass loss between 700 and 900 °C (1292–1652 °F) [4]. In 2012, Güneysi et al. [5] reported similar results after investigating four sources of kaolin deposits in the western region of Turkey for the optimum temperature and duration of calcination. It was reported that high alunite content kaolin required thermal treatment to 850 °C (1562 °F) for 3 h and it had double endothermic peaks in differential thermal analysis (DTA) at 580 °C (1076 °F) and 780 °C (1436 °F) [5].

Since kaolin is naturally occurring clay, therefore chemical and mineralogical compositions are highly dependent on the parent rock, climatic conditions of kaolin formation and processes of cleaning impurities [1]. Kaolin sources in the Perak and Sarawak states are dominated by kaolinite and were formed by the weathering of granites and diorites [1]. The dominance of kaolinite is due to the high feldspar minerals in the parent rock, which results in more alumina in kaolin [1]. Therefore, kaolin from Perak and Sarawak states of Malaysia possesses more potential to be converted into highly reactive metakaolin. In Table 1, mineralogical composition of kaolin from Perak state is estimated through semi-quantitative phase analysis of powder X-ray diffraction (XRD) pattern of kaolin. The identified content of kaolinite, alunite and quartz in Malaysian kaolin are relatively different from earlier mentioned studies and it was observed that the content of alunite

in Malaysian kaolin is not very high (refer to Table 1). The alunite content suggests that the calcination at 650 °C (1202 °F) for 3 h might be suitable for Malaysian kaolin, as suggested by Badogiannis et al. [4] and Güneysi et al. [5]. Furthermore, based on dependency of dehydroxylation temperature on the particle size, it might be inferred that Malaysian kaolin will require lesser temperature for the calcination. The particle size of Malaysian kaolin is 2.5–4.5 µm (0.00009–0.00018 in.), which is relatively lesser than the kaolin previously studied [4,5], suggesting that low temperature will be required for dehydroxylation. Smaller particle size is also advantageous in terms of energy required for the exclusion of more OH-groups in the interior of the crystals and weakening of the bound OH-group surfaces and on the edges of the particles [17]. It is further observed that dickite content, a polymorph of kaolinite, is present in Malaysian kaolin, which is a not reported earlier mentioned studies [4,5]. Dehydroxylation of dickite is relatively different from kaolinite. The inner surface H-atoms of an octahedral alumina sheet of kaolinite are lost concurrently during the formation of metakaolin at 450–600 °C (842–1112 °F); however in dickite, the outer H-atoms are rather removed first [18]. Therefore, when kaolinite and dickite are calcined simultaneously, the outer H-atoms in dickite, due to their early escape, interfere the evading of inner H-atoms from dickite during formation of kaolinite and this requires further heat treatment to draw out the inner H-atoms compared to the temperature suggested based on alunite content. This is also confirmed by the differential thermogravimetric (DTG) analysis shown in Fig. 1. Despite the lesser content of alunite in Malaysian kaolin, there are quite a number of peaks between 600 and 800 °C (1112–1472 °F) as shown in Fig. 1. Also, TG curve suggests that the full dehydroxylation is almost occurring between 600 and 800 °C (1112–1472 °F). Henceforth, there is a need to study the calcination of Malaysian kaolin due to distinctive mineralogical composition, particle size, phase transformation and dehydroxylation temperature suggested by DTG–TGA analysis. This study is also conducted to develop metakaolin locally, which is highly reactive and cost effective.

In Malaysia, limited attempts have been made to investigate the calcination of kaolin and its use as pozzolan. In 2006, Huat [19] thermally treated the kaolin from Perak state at 750 °C (1382 °F) and used it to replace cement up to 10% and showed good agreement in concrete in terms of compressive strength [19]. The limitations of study [19] are that only normal strength concrete up to 40 MPa (5800 psi) was studied. The temperature and duration of calcination were not optimized for the higher degree of dehydroxylation or for higher pozzolanic activity. It is well established that compressive strength is an indirect method of confirming

Table 1
Semi-quantitative phase estimation results.

% Weight	Kaolin
Kaolinite/dickite	51.3
Bementite	13.3
Quartz	8.0
Alunite	6.8
Amorphous silica (SiO ₂)	5.9
Cristobalite	1.9
Others	12.8

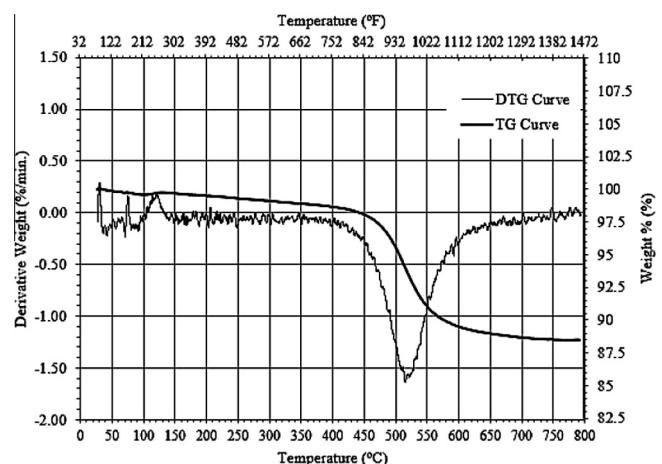


Fig. 1. TG and DTG curve of kaolin.

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