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21-23 December, 2018

Berlin - Germany

INFLUENCE OF THE CALCINATION THICKNESS OF KAOLIN ON STRENGTH DEVELOPMENT OF MORTARS

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Abstract

This research aims to improve local pozzolan material, kaolin, by investigating the effect of calcination thickness of kaolin to produce metakaolin with a high pozzolanic reactivity. Kaolin samples of the same mass were treated at 800°C for the same duration of 6 h by varying the calcination thickness of kaolin samples from 1 cm, 5 cm, and 10 cm in order to obtain metakaolins which were used to partially replace ordinary Portland cement (OPC) at the replacement of 20% by weight of binder. The results indicated that the optimal activation calcination layer was 10 cm. The XRD analysis confirmed pozzolanic activity of developed metakaolin. In addition, it was observed that the activated metakaolin had an appreciable influence on the increase in compressive strength of the mortars.

Keywords: Calcination, Compressive strength, Kaolin, Metakaolin, Thickness

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Introduction

Concrete is the most commonly used construction material worldwide and OPC, a major component of concrete, is the largest volume of construction material produced in the world and it will continue to grow as the result of continuous urban development all over the world (Adam, 2009) [1]. OPC production is one of the major reasons for CO₂ emissions into atmosphere. It is due to the use of fossil fuels, including the fuels required to generate electricity during cement manufacturing process. The use of pozzolanas for making concrete is considered efficient, as it allows the reduction of the cement consumption while improving the strength and durability properties of the concrete. Metakaolin when used as a partial replacement substance for cement in concrete, it reacts with Ca(OH)₂ one of the by-products of hydration reaction of cement and results in additional C-S-H gel which results in increased strength. Metakaolin is obtained by thermal activation of kaolin clay. This activation will cause a substantial loss of water in its constitution causing a rearrangement of its structure (Aiswarya et al., 2013) [2]. The energy generated by calcination of kaolin is derived from dehydroxylation of the kaolin by calcination according to the following formula (Abdelli et al., 2017) [3]:

Metakaolin is produced by number of researchers at different temperatures and durations. To obtain an adequate thermal activation, the temperature range should be established between 650°C to 800°C for 1 h to 12 h duration (Saand et al., 2016) [4]. At higher temperatures (>900°C), the metakaolin undergoes further reactions to form crystalline compounds, the end-products being free silica and mullite (El-Diadamony et al., 2017) [5]. In addition depending on the calcining temperature and clay type, it is also possible to obtain enhancement in strength, particularly during the early stages of curing. The very early strength enhancement is due to a combination of the filler effect and accelerated cement hydration (Wild et al., 1996) [6].

To reduce the production of cement to get reduction in CO₂ emission and reduction in overall construction cost, environmental friendly supplementary material is needed. In addition, the metakaolin has very interesting pozzolanic properties that can improve the performance of concrete. So the objective of this study is to develop the metakaolin as a pozzolanic material to be used in concrete from locally available natural material as supplementary cementing material.

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Materials and Methods

1. Materials

The materials used in this study are ordinary Portland cement (OPC) and kaolin. The kaolin was derived from Lampang, Thailand. The ground kaolin passed through 45 μm sieve. The chemical analysis of OPC and kaolin is shown in Tab. 1. SiO_2 is the major chemical component of kaolin with 64.36% and 25.61% Al_2O_3 . Moreover, the scanning electron microscope (SEM) and transmission electron microscope (TEM) photos of the kaolin as presented in Fig. 1 indicate that the kaolin has an irregular shape which is angular, square, and diamond shapes.

Table 1: Chemical analysis of the kaolin

Chemical compositions (Wt%)	OPC	Kaolin
Al_2O_3	3.51	25.61
SiO_2	18.09	64.36
SO_3	3.30	0.12
K_2O	0.63	6.64
CaO	67.5	0.60
Fe_2O_3	3.05	1.66

Figure 1: SEM and TEM images of the kaolin particles

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2. Methods

The study is divided into two parts:

In part 1, metakaolin is a product from dehydroxylation of a clay mineral, kaolin, which is very fine powder prepared by firing in a muffle furnace from room temperature up to 800°C for 6 h, the choice of this temperature being based on the work of Hensadeekul (1995) [7], by varying the calcination thickness of kaolin samples from 1 cm, 5 cm, and 10 cm. After thermal treatment, the metakaolin was left to cool to room temperature. Then, they were used to partially replace OPC at the replacement of 20% by weight of binder. While, the normal mortar (the control) was designed using a sand-OPC ratio of 2.75 by keeping the normal consistency for the mortar ranged from 105% to 115%. The best calcination layer which the highest compressive strength result was further used in part 2.

In part 2, to determine the factors influencing strength development in the metakaolin mortars, a substitution of 5%, 10%, 20%, 30%, and 40% by weight of OPC by the metakaolin was used.

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For the determination of compressive strength, cubic samples of $5 \times 5 \times 5 \text{ cm}^3$ were made. After casting for 24 h, the samples were removed from the molds and cured in water at room temperature for 1, 3, 7, 14, 21, 28, and 60 days. Depending on the nature of each type of material, kaolin, metakaolins and mortars were characterized using XRF, XRD, SEM, TEM, and compressive strength.

Results and Discussion

1. Characteristics of metakaolins

Each kaolin sample was thermally treated at temperature of 800°C for duration of 6 h using a muffle furnace to form metakaolin by varying the calcination thickness of kaolin samples from 1 cm, 5 cm, and 10 cm. The colour formation observed was as follows: the original kaolin, light brown colour; at 1 cm, light brown to reddish brown colour; at 5 cm, light brown to slightly reddish brown colour, and at 10 cm, light brown to brown colour. This was in consonance with the work of Salau et al. (2015) who studied about effects of temperature on the pozzolanic characteristics of metakaolin-concrete. Furthermore, SEM photos of the samples after exposure to 800°C are shown in Fig. 2. These specimens showed no noticeable change and were seen the same as before that had an irregular shape.

Figure 2: SEM images of the kaolin (a) unexposed (the original) and after exposed to 800°C at the calcination layer of, (b) 1 cm, (c) 5 cm, and (d) 10 cm.

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Figure 2: (continued) SEM images of the kaolin (a) unexposed (the original) and after exposed to 800°C at the calcination layer of, (b) 1 cm, (c) 5 cm, and (d) 10 cm.

2. X-Ray Diffraction Analysis (XRD)

The XRD patterns for the original kaolin (KL) and the specimens treated at 800°C for 6 h at the calcination layer of 1 cm (MK1), 5 cm (MK5), and 10 cm (MK10) are represented in Fig. 3. It can be observed that the XRD diffractograms of the samples were simple and similar. The major crystalline phases detected included quartz (Q). However, after exposure to a higher temperature of 800°C, kaolinite (K) became metakaolinite (M). The results are in good agreement with those reported by Abdelli et al. (2017) [3] who studied influence of the origin of metakaolin on pozzolanic reactivity of mortars.

Figure 3: XRD patterns for the original kaolin (KL) and the specimens treated at 800°C for 6 h at the calcination layer of 1 cm (MK1), 5 cm (MK5), and 10 cm (MK10).

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3. Compressive Strength

In part 1, calcination is needed to improve the performance of kaolin, converting it to metakaolin. Through calcination, kaolin will become reactive with $\text{Ca}(\text{OH})_2$ to enhance the strength of concrete. The results of compressive strength of the mortars with different calcination thicknesses are given in Tab. 2 and Fig. 4. The results revealed that the compressive strength varied from 8.45 MPa to 19.16 MPa with the highest value obtained from the metakaolin mortar at the calcination layer of 10 cm after 28 days. As expected, the compressive strength of the mortar specimens developed with the curing time.

Table 2: Compressive strength of the metakaolin mortars with different calcination thicknesses.

Thickness	Compressive strength (MPa)			
	1 day	3 days	7 days	28 days
1 cm	8.45	15.47	15.69	17.33
5 cm	9.26	14.59	15.81	16.50
10 cm	9.73	12.73	13.59	19.16

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Figure 4: Compressive strength development for the metakaolin mortars with different calcination thicknesses.

In part 2, the best calcination layer which the highest compressive strength result obtained from part 1, 10 cm, was used in this part. A substitution of 5% (MK5), 10% (MK10), 20% (MK20), 30% (MK30), and 40% (MK40) of OPC by the metakaolin at the calcination layer of 10 cm was produced. The results of compression test for varying OPC replacement are shown in Tab. 3, while Fig. 5 shows the strength performance of the mortars at different substitutions. The strength development of the blended mortars was relatively close to the normal mortar (the control). MK10 had the best strength performance among other samples. This can be clearly revealed with 10% replacement achieving compressive strength of 26.47 MPa compared to 25.30 MPa of the control at 60 days. The presence of metakaolin in the mortars led to a decrease in the strength values with the increase in metakaolin content for the first 28 days of curing. This shows the significance of pozzolanic effects as the strength increases with age or at longer period of curing. The compressive strength of mortar specimens with 20%, 30%, and 40% replacements was generally lower than the control at all test ages.

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Compared to 5% replacement, the amount of metakaolinite exists in the metakaolin blended mortars are probably too high. Likely, the quantity of Ca(OH)_2 , produced from the hydration of cement, is not enough to react with all the metakaolinite to produce extra C-S-H. The Ca(OH)_2 has been reduced to the minimum level while some metakaolinite are left out without any chemical reaction (Salau & Osemeke, 2015) [8]. The development of strength with age was consistent in all cases.

Table 3: Compressive strength of the metakaolin mortars with different calcination thicknesses.

Age (days)	Compressive strength (MPa)					
	Normal mortar	MK5	MK10	MK20	MK30	MK40
1	10.40	11.10	9.60	6.89	5.19	3.54
3	12.84	14.20	14.83	10.98	7.89	5.50
7	15.42	16.45	17.39	12.41	10.64	8.36
14	17.86	17.13	18.05	15.98	14.39	9.80
21	21.67	18.01	20.89	16.75	14.95	12.12
28	23.20	20.20	21.85	20.20	17.53	12.24
60	25.30	24.18	26.47	21.87	18.75	11.98

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Figure 5: Compressive strength development for the metakaolin mortars with different replacements.

Conclusions

From the above finding it may be concluded that:

- (1) Different colour formations are observed for different kaolin samples at different calcination thicknesses.
- (2) The optimum thickness among the range applied in this study is 10 cm.
- (3) Substitution of 10 wt% OPC by metakaolin exhibits the best strength performance in this study. The compressive strength values are comparable to the control.

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Acknowledgment

The author gratefully acknowledges Dr. Theerawat Sinsiri as esteemed adviser. I would also like to thank the financial support from the Faculty of Industrial Technology, Nakhon Ratchasima Rajabhat University and the Department of Civil Engineering, Suranaree University of Technology for providing facilities and equipment.

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