

COMPRESSIVE STRENGTH, FLOWABILITY, AND ULTRASONIC PULSE VELOCITY TESTS OF PORTLAND CEMENT-FLY ASH-CALCINED CLAY MORTARS

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Abstract

The cement production process significantly affects the environment and contributes to air pollution. Consequently, there is a growing emphasis on employing alternative materials or minimizing cement usage as a solution to address environmental issues and reduce air pollution. In this investigation, an examination was conducted by substituting Portland cement with a weight of 50% with both fly ash and calcined clay. The findings revealed that replacing Portland cement with fly ash resulted in a decrease in compression strength. However, concurrently incorporating calcined clay increased compressive strength. Higher compressive strength compared to the 50% Fly ash mix was achieved by adding 20 wt.% Metakaolin and 20 wt.% Calcined clay Lopburi due to their finer particle size. As a result, the pozzolanic reaction is better than Lampang calcine clay (30FA20CClam). Additionally, ultrasonic pulse velocity testing indicated that higher values in the specimen correlated with increased compressive strength.

Keywords: Compressive Strength; Ultrasonic Pulse Velocity; Flowability; Ternary Blend Mortar

Introduction

The use of pozzolan materials as a substitute for cement is widely adopted because Portland cement production plants emit significant amounts of carbon dioxide (almost 1 tonne/year cement produced) (Emetere and Dania, 2019). The airborne particles generated during the Portland cement production process are carried by the wind and settle in the proximity of the manufacturing facilities and also faraway places such as the countryside, green space, and relaxing places (Emetere and Dania, 2019).

Fly ash is recognized as an effective pozzolanic material for use in concrete, and several studies have verified its impact on the pore structure and physical properties of mortar (Chindaprasirt *et al.*, 2005). Fly ash has been employed as a substitute in proportions from 15% to 25% cement content. The specific quantity utilized varies significantly based on factors that depend on the intended application, fly ash properties, specification constraints, geographic location, and

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climate (Shvarzman *et al.*, 2003). Fly ash has been used as 30-50% for substantial structures such as foundations and dams to decrease the temperature of structural concrete. Moreover, the newest research has indicated that elevated dosage levels of 40-60% fly ash can be applied in structural applications, yielding concrete with desirable durability and mechanical properties (Choo and Newman, 2003). When kaolin is subjected to temperatures ranging from 600 to 850 °C (1,112-1,562 °F), it transforms into metakaolin (Badogiannis *et al.*, 2005). Metakaolin exhibits high reactivity as a pozzolan, however, its physical and chemical attributes are significantly influenced by the raw kaolin material, the calcination temperature, and the finishing process (especially rapid cooling after calcination) (Kim *et al.*, 2007). It is a pozzolanic substance capable of reacting with $(\text{Ca}(\text{OH})_2)$. This is a result of the hydration process of Portland cement (Numluk and Chaisena, 2012). Metakaolin contributes to the enhancement of compressive strength (Choo and Newman, 2003). The effect of metakaolin on its own in Portland cement (PC) - Metakaolin (MK) leads to a rise in the compressive strength of concrete, with the increase in MK content becoming more pronounced at 28 and 90 days also studied by Güneyisi *et al.* in 2014 (Güneyisi *et al.*, 2014). When metakaolin is used in combination with other pozzolanic materials for example fly ash, it has been created to have a useful impact on compressive strength (Güneyisi *et al.*, 2014). Metakaolin is the calcination of pure kaolinite clay. On the other hand, calcined clay contains metakaolin and other impurities (Chomyen *et al.*, 2019; Win *et al.*, 2023a) are more abundant in terms of resources (Shvarzman *et al.*, 2003). In addition, Güneyisi *et al.* (Güneyisi and Gesoğlu, 2008). studied the properties of self-compacting of the metakaolin - fly ash hybrid blend. The combined use of mineral admixtures effectively addressed the limitations observed in mixtures containing only binary blends of FA. Where the mix using 10% FA and 10% MK showed compressive strength surpassed those of the control mix.

Ultrasonic pulse velocity (UPV) is a nondestructive testing technique that can monitor concrete by transmitting ultrasonic pulses through the specimen and measuring the pulse velocity. A higher velocity signifies positive physical characteristics of the specimen, such as efficient compaction, enhanced strength, and higher density (Estévez *et al.*, 2020). Therefore, ultrasonic pulse velocity was found to have a good correlation with compressive strength. Güneyisi *et al.* (Güneyisi *et al.*, 2014) and Parsompech *et al.* (Parsompech *et al.*, 2022) investigated the relationship between compressive strength and ultrasonic pulse velocity.

Understanding the correlation between compressive strength and ultrasonic pulse velocity allows for the assessment of the strength and durability of a structure without causing any harm. Therefore, this study specifically examines the compressive strength and ultrasonic pulse velocity of specimens by replacing Portland cement at 50% by weight incorporating fly ash with calcined clay using three different types i.e. metakaolin (China source), calcined clay from Lampang, Thailand, and calcined clay from Lopburi, Thailand. because Thailand has a source of raw clay that is unique and has rare characteristics (Sudswong and Saengsoy 2022). Raw clay in Thailand contains metakaolin and impurities and the resource is available for calcining. Furthermore, the research investigated the relationship between compressive strength and ultrasonic pulse velocity, and this research also determined the ability of calcined clay in Thailand to replace Portland cement.

Materials and Methods

Materials and Mixture Proportion

Fly Ash (FA) was obtained from Mae Moh, Lampang Thailand, Metakaolin (MK) was supplied by Centasia Co., Ltd. Calcined clays (CC) was produced by calcining clay from Lampang and Lopburi provinces in Thailand at 750 °C. FA, MK, and CC were used to replace part of ordinary Portland cement (ASTM C150 Type I). The chemical compositions of Portland cement (PC), FA, MK, and calcined clay Lampang (CCLam) and calcined clay Lopburi (CClop) are given in Table 1. The morphology of materials used in this research taken from scanning electron microscopy (SEM; JSM IT-800) are shown in Figure 1.

The details of the mix proportions are given in Table 2. In this study, the water-to-binder ratio (w/b) was maintained at 0.5 and the sand-to-binder ratio of 2.5 was used in order to control the flow ability of the control Portland cement sample (105 ± 5 mm.) according to ASTM C1437 (ASTM C1437, 2001).

Table 1. Chemical Configuration

Chemical composition (%)	PC	FA	MK	CCLam	CClop
SiO ₂	18.89	37.26	51.30	55.58	46.75
CaO	64.45	19.46	0.05	0.45	3.93
Fe ₂ O ₃	3.13	12.76	0.42	1.20	11.58
Al ₂ O ₃	5.18	20.64	44.42	35.04	24.96
MgO	1.74	0.75	<0.01	0.26	
Na ₂ O	0.14	0.14	0.33	0.49	
K ₂ O	0.52	1.05	0.14	6.53	

The particle size of the pozzolanic material replaced with Portland cement was measured using Particle size Analyzer Laser, diffraction (Masteriser S, Malvern Instruments Limited), with the average particle size detected in the analysis being 0.05-900 μm . The table shows particle size distribution in Table 3.

The particle size distribution of Portland cement, fly ash, metakaolin, and calcined clay from Lampang and Lopburi were analyzed at diameters 10, 50, and 90 μm .

Methods

The specimens were cast into cubes measuring ($5 \times 5 \times 5 \text{ cm}^3$), following ASTM C109 (ASTM C109, 2009) guidelines. The hydraulic mortar was cured in lime-saturated water at $23 \pm 2^\circ\text{C}$ for durations of 7, 28, and 60 days. Compressive strength and ultrasonic pulse velocity were conducted using identical samples through the ultrasonic pulse velocity test employing the PUNDIT LAB equipment, then followed by the compressive strength tests.

Mixing method and flow

The samples were prepared according to ASTM C305 (ASTM C305, 1999). The binding components, comprising Portland cement, fly ash, and metakaolin or calcined clay were initially blended in a rotary mixer and mixed with water at a low speed ($140 \pm 5 \text{ r/min}$). After 30 seconds, fine aggregates were introduced into the rotary mixer, and the mixture was continued for 30 seconds. The mixing speed was then increased to a moderate level ($285 \pm 10 \text{ r/min}$) for 30 seconds, followed by 90 second pause of the mixer. After resuming mixing at medium speed for 60 seconds, the flow test was conducted following ASTM C1437 (ASTM C1437, 2001). A mixture was positioned at the center of the flow table, filled with mortar, and the mold before placing the table and measuring the flow after dropping the table 25 times in 15 seconds. The flow of the mortar was then measured based on its diameter.

Compressive Strength

The specimens were cast into a cubic mold with dimensions of $5 \times 5 \times 5 \text{ cm}^3$. After the curing process, the samples were investigated for compressive strength following the standard ASTM C109 (ASTM C109, 2009).

Ultrasonic Pulse Velocity (UPV)

UPV is a non-destructive examination that assesses the excellence of concrete, and mortar by measuring the speed of waves transmitted through

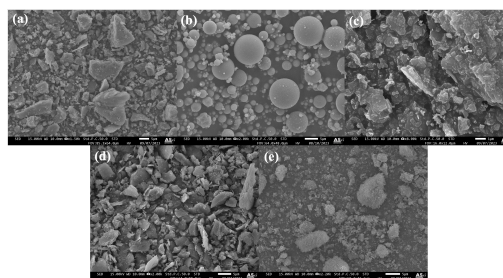


Figure 1. The morphology of (a) Portland Cement (b) Fly Ash (c) Metakaolin (d) Calcined clay Lampang, and (e) Calcined clay Lopburi

Table 2. Mixtures composition

Mixtures	PC (wt.%)	FA (wt.%)	MK/CC (wt.%)
100PC	100		
50FA	50	50	
40FA10MK	50	40	10
30FA20MK	50	30	20
30FA20CClam	50	30	20
30FA20CClop	50	30	20

Table 3. Particle size distribution

Sample	D10 μm	D50 μm	D90 μm
Portland Cement	4.36	20.84	116.37
Fly Ash	0.18	9.78	108.72
Metakaolin	0.15	2.04	5.08
Calcined clay (CClam)	2.58	6.05	14.30
Calcined clay (CClop)	0.19	9.43	39.45

the specimens within a defined time frame. The mortar of $5 \times 5 \times 5 \text{ cm}^3$ cubes was tested for pulse velocity and the transducer functioned at a frequency of 54 kHz following ASTM C597 (ASTM C597, 2009). The producer and acceptor were located on opposite sides of the cube.

Results and Discussion

Figure 2 illustrates the flow diameter of mortar specimens. It was noticed that the flow diameter decreased with increasing metakaolin, and calcined clay (40FA10MK, 30FA20MK, 30FA20CClam, 30FA20CClop), and then the flow diameter increased when replacing Portland cement with fly ash. Fly ash has the potential to decrease the water demand in mortar because of spherical and round surface appearance derived from fly ash enhances the flow characteristics and, consequently the workability of mortars (Jongwichaksana and Suwan, 2021; Win *et al.*, 2023b). Moreover, when

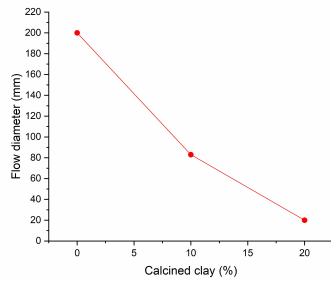


Figure 2. Flow diameter of mortars

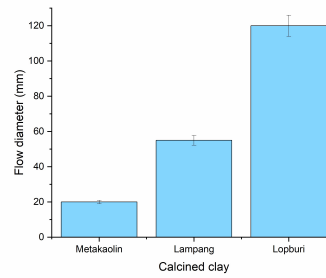


Figure 3. Flow diameter of Calcined clay

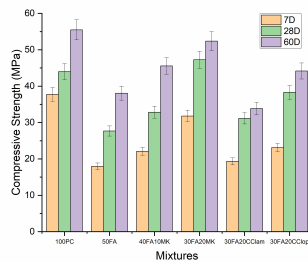


Figure 4. Compressive Strength of mortar specimens

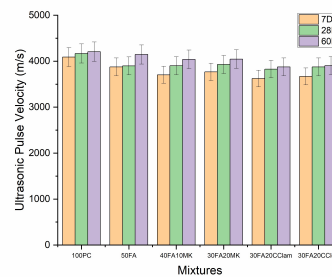


Figure 5. Average of Ultrasonic pulse velocity of mortars

using fly ash of sufficient quality, including low carbon content and high resolution, it will be possible to reduce water use in the system. Using an identical ratio of w/b in the mortar with an elevated content of fly ash (50 wt.%, 50FA) the flow is 220 mm, which is higher than the control mix of 110 mm (100PC). On the contrary, the inclusion of calcined clay in the mortar results in an increase in the water needed (Phung *et al.*, 2021). Figure 3 illustrates the flow diameter of calcined clay was then measured from its spread diameter. The addition of calcined clay in the mortar raises the water demand, and when the w/b ratio remains constant, increasing the calcined clay content from 10 wt.% (in the 40FA10MK) to 20 wt.% (in the 30FA20MK, 30FA20CClam, 30FA20CClop) in comparison to the 50FA mix results in a decrease in flow diameter. Specifically, the flow diameter for the 40FA10MK, 30FA20MK, 30FA20CClam, and 30FA20CClop is measured at 83, 20, 55, and 100 mm, respectively. However, it should be noticed that the combination of calcined clay with fly ash as a synergic can achieve a nominal flow similar to the control Portland cement such as in mixes of 40FA10MK (83 mm) and 30FA20CClop (100 mm).

Figure 4 illustrates the compressive strength of samples. It was noticed that the compressive strength is affected by different Portland cement replacement materials. The addition of metakaolin (MK) resulted in an enhancement of both the early-

age strength (7 days) and the 28 and 60 day strengths of mortars. Nevertheless, the progress in strength development was notably more pronounced during the initial 28 days. This observation aligns with findings from prior research, which indicated that the primary factor influencing the early-age strength development of the sample is the pozzolanic reaction of metakaolin (Wild *et al.*, 1996). Metakaolin is characterized as an amorphous phase composed of either $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ or the $\text{Al}_2\text{Si}_2\text{O}_7$ form (Shvarzman *et al.*, 2003). Therefore, the non-crystalline composition of $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ found in calcined clay can undergo a reaction with calcium hydroxide originating from Portland cement, resulting in the formation of calcium aluminum silicate hydrated gel, known as CASH (C_2ASH_8). This process refines the pore structure and contributes to the development of compressive strength, a phenomenon referred to as the Pozzolanic reaction (Frías *et al.*, 2000).

Figure 5 illustrates the average ultrasonic pulse velocity of the sample fly ash-calcined clay was added. The result shows that the UPV rises with increased time curing due to the hydration interaction of Portland cement and the pozzolanic reaction of fly ash and calcined clay is developed over time (Said-Mansour *et al.*, 2011). At a given age, the control mix (100PC) has the highest UPV and compressive strength at all curing times.

Figures 6 to 10 illustrates the correlation between ultrasonic pulse velocity and compressive strength of each specimen. It reveals a direct correlation of 50FA, 40FA10MK, 30FA20MK, 30FA20CClam, and 30FA20CClop with correlation coefficients (R^2) of 0.83, 0.81, 0.84, 0.82, and 0.94, respectively. The impact of the age of mortar on ultrasound pulse velocity is notable, as the strengths of mortars experience significant variations based on the quantity of calcined clay in the mixture. The ultrasound pulse velocity rises as the specimen ages. Utilizing ultrasonic pulse velocity as an indicator of strength is highly reliable (Bilir *et al.*, 2015).

From the experimental results in the flowability. Ultrasonic pulse velocity including the compressive strength, it is evidence that when

Portland cement was replaced with fly ash and metakaolin (30FA20MK), the compressive strength was higher than that of Fly ash (50FA) but at the same time, because the price of metakaolin is quite expensive, it will affect the cost of production, which is another limitation. It is evident that in addition to the proportion of 30FA20MK when replaced with calcined clay from Lopburi (30FA20CClop) has a compressive strength higher than that of Fly ash (50FA), although lower than that replaced with metakaolin. Including the amount of Calcined clay in Thailand, it is easily found and is higher in quantity than metakaolin. But calcined clay is less expensive to produce than metakaolin, so it is more suitable to be used to replace Portland cement in actual use.

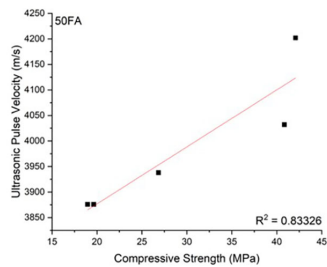


Figure 6. The correlation between UPV and compressive strength of 50FA

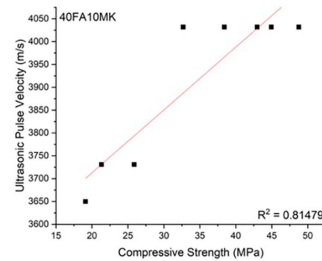


Figure 7. The correlation between UPV and compressive strength of 40FA10MK

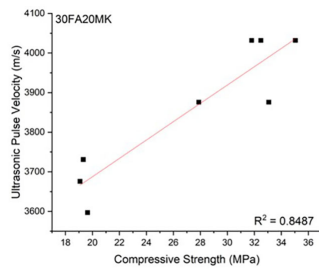


Figure 8. The correlation between UPV and compressive strength of 30FA20MK

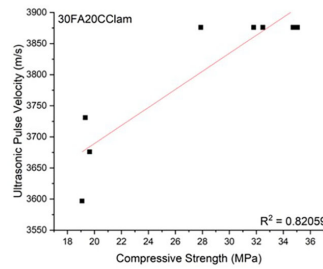


Figure 9. The correlation between UPV and compressive strength of 30FA20CClam

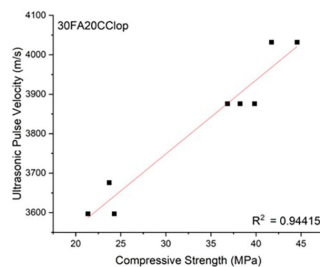


Figure 10. The correlation between UPV and compressive strength of 30FA20CClop

Conclusions

- At all ages, 40FA10MK, 30FA20MK, 30FA20CClam, and 30FA20CClop mixtures exhibit an increased compressive strength compared with the mix with 50FA at the same Portland cement replacement level. On the other hand, it was observed that the compressive strength increased due to the pozzolanic reaction with an increase in calcined clay content compared to a mixture containing only fly ash.

- The ultrasonic pulse velocity increased as the curing time increased (from 7, 28, and 60 days), and the raising of calcined clay increased from 0, 10, and 20 wt.% (50FA, 40FA10MK, 30FA20MK, CClam, and CClop, respectively) because calcined clay could fill pores and pozzolanic reactions. That is increased compressive strength. The findings indicated a clear correlation between the compressive strength and ultrasonic pulse velocity of 50FA, 40FA10MK, 30FA20MK, 30FA20CClam, and 30FA20CClop. Where R-square (R^2) is 0.83, 0.81, 0.84, 0.82, and 0.94 respectively.

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