# An investigation of palm oil fuel ash and limestone powder for use in sustainable high strength concrete construction

OP. Sanit-in<sup>a</sup>, OT. Charoensuk<sup>b</sup>, OS. Dueramae<sup>c</sup>, OA. Abdulmatin<sup>d</sup>⊠, OP. Ratanachu<sup>d</sup>, OW. Tangchirapat<sup>b</sup>, OC. Jaturapitakkul<sup>b</sup>

a. Faculty of Engineering, Kasetsart University Kamphaeng Saen Campus, (Nakhon Pathom, Thailand)
b. Faculty of Engineering, King Mongkut's University of Technology Thonburi (KMUTT), (Bangkok, Thailand)
c. Faculty of Engineering, Rajamangala University of Technology Krungthep, (Bangkok, Thailand)
d. Faculty of Engineering, Princess of Naradhiwas University (PNU), (Narathiwas, Thailand)
i. akkadath.a@pnu.ac.th

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**ABSTRACT:** This study deals with the production of concrete with low CO<sub>2</sub> emissions. For this reason, two materials, namely palm oil fuel ash (POFA) and limestone powder (LP), were used as mineral additions to replace conventional cement as much as possible. The results showed that concrete containing 60 wt% POFA could achieve the high strength requirements of ACI 363R from an age of 28 days. At a replacement level of 70 wt% of admixtures, the use of 10 wt% LP + 60 wt% POFA was a good mixture as it had the highest concrete compressive strength, had a minor effect on shrinkage strain and reduced heat generation by approximately 50% compared to cement concrete. Additionally, the use of POFA and LP is a good choice to produce environmentally friendly concrete, which can reduce the CO<sub>2</sub> emissions of concrete by about 44–62% compared to cement concrete at similar strength.

KEY WORDS: Palm oil fuel ash; Limestone powder; High-strength concrete; High-volume pozzolan; Low CO, concrete.

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**RESUMEN:** Investigación del empleo de ceniza de la combustión de aceite de palma y filler calizo en la fabricación de hormigones sostenibles de alta resistencia. En este estudio se aborda la producción de hormigón con bajas emisiones de  $CO_2$ . Por este motivo, se utilizaron dos materiales, a saber, ceniza de aceite de palma (POFA) y filler de piedra caliza (LP). Estos materiales se añadieron para llevar a cabo el reemplazo del cemento convencional tanto como fuera posible. Los resultados muestran que el hormigón que contiene un 60% en peso de POFA podría lograr los altos requisitos de resistencia de ACI 363R, a partir de los 28 días de edad. Al ampliar el nivel de sustitución al 70% en peso de aditivos, con la mezcla de 10% de LP + 60% de POFA se obtendrá una mayor resistencia a la compresión del hormigón, ya que este tenía un efecto menor en la deformación por contracción y de igual manera reducía la generación de calor en aproximadamente un 50% en comparación con el hormigón convencional. Además, el uso de POFA y LP es una buena opción para producir hormigón ecológico y lograr una aportación con el medio ambiente ya que reduce las emisiones de  $CO_2$  del hormigón en aproximadamente un 44–62% en comparación con el hormigón convencional de resistencia similar.

PALABRAS CLAVE: Ceniza de aceite de palma; Filler de piedra caliza; Hormigón de alta resistencia; Puzolana de alto volumen; Hormigón bajo en CO,.

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## **1. INTRODUCTION**

The second most popular material after water is concrete (1), which usually contains ordinary Portland cement (OPC) as a cementitious material. OPC is a material that emits  $CO_2$  into the atmosphere directly (through the calcination of limestone) and indirectly (through heating in a kiln that burns fossil fuels) during production (2), while the concept of low-carbon products is a popular topic of discussion worldwide. Therefore, researchers and the industrial sector have focused on reducing the amount of OPC in the concrete mixture by using pozzolans e.g., fly ash (FA), palm oil fuel ash (POFA), bagasse ash (BA) and rice husk ash (RHA) as a partial substitute for OPC to reduce environment impact.

Currently, the waste materials of the palm oil plant e.g., palm oil residues and palm oil fuel ash (POFA) have been investigated for construction products from the many research (3-5). However, palm oil residues can be used as fuel for power manufacturing in palm oil plants. At the same time, a by-product of POFA is produced, which is disposed in the vicinity of the palm oil plants without the utilization. Palm oil production is one of the top agricultural industries in Thailand. In 2022, the production of palm oil fruits amounted to 18.6 million tons (6). Tanchirapat et al. (7) reported that the palm oil residues produced after oil distillation make up about 50 wt% of the crude palm oil. After combustion of palm oil residues for power generation, POFA was estimated to be 5 wt% of palm oil residues. Thus, Thailand probably produces about 0.46 million tons of POFA annually, and this quantity is probably increasing every year. In general, POFA consists of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> (together about 55-70%), which is the main chemical composition of pozzolan. The strength activity indices are in the range of 78–115% depending on the fineness and chemical composition of POFA (5). When using ground POFA mixed with OPC in mortar or concrete, the compressive strength is generated not only by the pozzolanic reaction but also by the filling effect due to the fine particles of the ground POFA. Jaturapitakkul et al. (8) reported that considering the compressive strength of 50.8 MPa at 28 days of the mortar containing ground POFA with a median particle size  $(d_{so})$  of 12.3 µm, up to 30% of the strength came from the pozzolanic products and 8% of the strength from the filling effect. Zevad et al. (9) studied high-strength concrete containing POFA as a complementary binder and found that high compressive strengths of 98 and 96 MPa at 28 days could be achieved with a proportion of 20 and 40 wt% ground POFA with d<sub>50</sub> particles smaller than 3  $\mu$ m and a water-binder (W/B) ratio of 0.27 in the concrete mixtures. The compressive strength could be enhanced to 104 and 106 MPa at 90 days for concrete with ground POFA of 20 and 40 wt%, respectively, which resulted in a greater strength than OPC concrete at the identical W/B ratio. Furthermore, many researchers have confirmed that by using suitable POFA in the concrete mix, similar or better mechanical and durability properties can be achieved than with OPC concrete (10-12).

In Thailand, the construction industry is transitioning from ordinary Portland cement (Type 1) to hydraulic cement for general construction (Type GU) which usually contains additives such as limestone powder (LP). LP is a mineral material whose main component is calcium carbonate (CaCO<sub>2</sub>). LP is one of the alternative complementary cementitious materials that can partially replace OPC. Although LP is an inert material, it could act as a filler material, resulting in a denser transition zone of the paste-aggregates (13). Furthermore, the use of LP in appropriate dosage and particle size could act as an accelerator during early hydration, which does not negatively affect the concrete properties (14-16). From earlier study (17), it was clear that the fine particles of LP augmented the nucleation effect, which could stimulate the degree of hydration reaction and precipitation of hydration products, resulting in increased strength at an early age. However, concrete with an overdose of LP led to a reduction in compressive strength. Ramezanianpour et al. (18) recommended that the use of LP in the concrete mixture should not exceed 10 wt%, otherwise the compressive strength of the concrete containing LP would be less than OPC concrete without LP. Similarly, Bentz et al. (19) concluded that OPC can be replaced by LP up to a maximum of 10% by volume to obtain acceptable performance of the concrete.

There is also some research showing that pozzolanic material could be applied together with LP to partially replace OPC. For example, Wang (20) investigated the degree of reaction from the ternary materials of OPC, FA and LP as a binder and reported that the degree of reaction of paste with OPC: FA: LP in a ratio of 65:30:5 wt% was higher than that of paste with OPC: FA of 65:35 wt%. The result was consistent with the study of Li and Jiang (21), who reported that LP could promote hydration reaction at an early age when LP was used in a 10 wt% admixture with 55 wt% slag to replace OPC in the concrete. The result also informed that the early-age compressive strength (3 and 7 days) of concrete with a slag content of 55 and 75 wt% blended with 10 wt% LP to replace OPC was increased compared to slag concrete without LP in the mixture. In addition, Kathirvel et al. (22) investigated the concrete made from a mixture of FA, RHA and LP as a replacement for OPC. Their results stated that the appropriate ratio

of FA:RHA:LP for the mechanical properties of the concrete was 20:10:10 wt%, i.e. this ratio improved the compressive strength, flexural strength and splitting tensile strength. From the above literature, the addition of LP of about 5-10 wt% in the mixture can develop the strengths of concrete especially at an early age due to filling and nucleation effects.

The use of POFA as a pozzolanic material in cementitious materials has been investigated in the literature. However, most studies have focused on POFA replacement of no more than 50% in the concrete mixtures. In contrast, this study investigates the properties of the concrete containing high volume POFA (HVPA concrete) using POFA at 60, 70 and 80 wt% in the mixture to develop a high strength concrete containing as much POFA as possible. Furthermore, the study of the effect of LP as an admixture in concrete with high volume pozzolan is limited, especially when POFA is the primary pozzolan. Therefore, the effects of LP on the properties of HVPA concrete such as compressive strength, elastic modulus, shrinkage strain and microstructure are also investigated in this study. The findings of this study would not only add to the knowledge on the utilization of high volume POFA to produce high-strength concrete, but also contribute to the knowledge base on concrete with high volume POFA in combination with LP. It was assumed that the concrete produced from POFA with and without LP as a supplementary binder is suitable for use in sustainable construction and thus also reduces the environmental impact of concrete production.

# 2. MATERIALS AND METHODS

#### 2.1. Materials

OPC, POFA and LP are the main materials that have the properties listed in Table 1. In addition, the size distributions of particles of these materials are displayed in Figure 1. The particles of OPC retained on a 45 µm sieve amounted to 20.00 wt%, corresponding to a specific gravity of 3.15. The POFA originated from the disposal area of a palm oil mill in the Thai province of Chumphon, which was produced the burning of palm fiber, palm shells and empty fruit bundles at the temperature of 800-900 °C. The collected POFA from the disposal area was sun-dried to remove the moisture content and then ground so that the fine particles had a retained weight of 0.66 wt% on a 45 µm sieve, a specific gravity of 2.36 and a  $d_{_{50}}$  of 8.50  $\mu m.$  The ground POFA used in this study had a strength activity index of over 100% since the age of 7 days. The LP was sourced from a limestone powder company in Thailand. The specific gravity of the LP was 2.70, which corresponds to a  $d_{50}$  value of 2.64  $\mu$ m.

| TABLE 1. Physical and chemical properties of materials. |        |             |       |  |  |  |  |
|---|--------|-------------|-------|--|--|--|--|
| Physical property                                       | OPC    | Ground POFA | LP    |  |  |  |  |
| Specific gravity  | 3.15   | 2.36        | 2.70  |  |  |  |  |
| Particles retained on a No.45 µm sieve (%)              | 20.00  | 0.66        | 0     |  |  |  |  |
| Median particle size, $d_{_{50}}(\mu m)$                | 18.1   | 8.50        | 2.64  |  |  |  |  |
| Strength activity index (%)<br>7 days<br>28 days        | -<br>- | 101<br>111  | -     |  |  |  |  |
| Chemical component (%)                                  |        |             |       |  |  |  |  |
| SiO <sub>2</sub>  | 20.8   | 48.6        | < 0.1 |  |  |  |  |
| Al <sub>2</sub> O <sub>3</sub>                          | 4.5    | 1.3         | < 0.1 |  |  |  |  |
| Fe <sub>2</sub> O <sub>3</sub>                          | 3.3    | 1.9         | < 0.1 |  |  |  |  |
| CaO   | 65.1   | 16.8        | 53.9  |  |  |  |  |
| MgO   | 1.2    | 4.0         | 0.5   |  |  |  |  |
| Na <sub>2</sub> O                                       | 0.3    | 0.2         | _     |  |  |  |  |
| K <sub>2</sub> O  | 0.2    | 13.3        | -     |  |  |  |  |
| SO <sub>3</sub>   | 2.2    | 3.9         | _     |  |  |  |  |
| LOI   | 2.3    | 10.0        | 44.8  |  |  |  |  |
| $SiO_2 + Al_2O_3 + Fe_2O_3$                             |        | 51.8        | _     |  |  |  |  |

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FIGURE 1. Particle size distribution of materials.

The chemical constituents of three materials are also listed in Table 1. The principal chemical constituents of ground POFA were SiO<sub>2</sub>, CaO and K<sub>2</sub>O. The sum of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> accounted for 51.8%, while CaO accounted for 16.8% (more than 10%). Thus, the ground POFA used in this experiment was categorized as class 2 according to the TIS 2888-18 standard (standard for POFA for use as an admixture in concrete) (23). The principal chemical constituent of LP was CaO (53.9 %). The loss on ignition (LOI) of LP was high (44.8%) because LP was in the form of CaCO<sub>3</sub>, which decomposed to CaO and CO<sub>2</sub> at high temperatures (700–850 °C) (24), using a temperature of 750 °C in the LOI tests.

#### 2.2. Mixture proportions

The six concrete mixtures investigated are listed in Table 2. The OPC was replaced by POFA in the high volume of 60, 70 and 80 wt%, which were designated as HVPA60, HVPA70 and HVPA80 concretes, respectively. In the HVPA concretes blended with LP, the OPC content was kept constant at 30% wt%. LP was used at 5 wt% and 10 wt% to replace POFA (with POFA+LP maintained at 70 wt%). These concretes were designated HVPA65L5 and HVPA60L10, respectively. All HVPA concretes had a binder volume of 560 kg/m<sup>3</sup> and a W/B ratio of 0.25 in order to obtain the expected compressive strength of 55 MPa for high-strength concrete in accordance with ACI 363R (25). To prevent the compressive strengths of OPC concrete and HVPA concrete from differing greatly when comparing the concrete properties, the OPC concrete (100 wt.% OPC as binder) had an OPC volume of 500 kg/m3 and a W/B ratio of 0.30. All concrete mixture, crushed granite was used as the coarse aggregate, passing through standard sieve No. 3/8 and retained on No. 4, with the water absorption of 0.67% and specific gravity of 2.65. For the fine aggregate, river sand was sieved through standard sieve No. 4 with the water absorption of 0.92% and specific gravity of 2.61. The superplasticizer dosage was trialed to preserve the slump of all fresh concretes at 150-200 mm.

#### 2.3. Experimental methods

#### 2.3.1. Compressive strength and elastic modulus

After mixing, the concretes were filled into cylinder molds with a diameter of 100 mm and a height of 200 mm. After casting for approximately 24 hours, each concrete specimen was demolded and then stored in water to cure the concrete specimen until test age. The loading rate of 0.25 MPa/s was applied to evaluate the compressive strength and elastic modulus. The com-

| Concrete  | Mix proportion (kg/m <sup>3</sup> ) |      |    |                   |                     |       |     |       | Shump |
|-----------|-------------------------------------|------|----|-------------------|---------------------|-------|-----|-------|-------|
|           | OPC                                 | POFA | LP | Fine<br>aggregate | Coarse<br>aggregate | Water | SP. | — W/B | (mm)  |
| OPC       | 500                                 | 0    | 0  | 815               | 990                 | 150   | 1.5 | 0.30  | 150   |
| HVPA60    | 224                                 | 336  | 0  | 760               | 920                 | 140   | 6.2 | 0.25  | 160   |
| HVPA70    | 168                                 | 392  | 0  | 750               | 910                 | 140   | 6.7 | 0.25  | 165   |
| HVPA80    | 112                                 | 448  | 0  | 745               | 900                 | 140   | 7.3 | 0.25  | 170   |
| HVPA65L5  | 168                                 | 364  | 28 | 755               | 935                 | 140   | 7.8 | 0.25  | 150   |
| HVPA60L10 | 168                                 | 336  | 56 | 755               | 935                 | 140   | 7.8 | 0.25  | 165   |

TABLE 2. Mix proportions of concrete.

SP.=Superplasticizer

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pressive strength was appraised at 7, 14, 28 and 90 days, while the elastic modulus was evaluated at 28 and 90 days. The compressive strength (ASTM C39 (26)) and modulus (ASTM C469 (27)) of the concrete were appraised and recorded using the average values of three specimens.

#### 2.3.2. Shrinkage strain

High-strength concrete generally has a high shrinkage rate, especially at an early age. Therefore, the shrinkage strain of concrete was investigated. A concrete specimen (50 x 50 x 285 mm) was used to examine the shrinkage strain, which was applied according to ASTM C157 (28). After casting for 24 hours, each concrete specimen was demolded and the initial length in each mixture was evaluated immediately. All concrete specimens were cured under ambient conditions (relative humidity  $50\pm5\%$  and temperature  $23\pm2$  °C). The length changes of the concrete specimens were recorded until the end of the 28 days.

#### 2.3.3. Heat evolution.

OPC, HVPA70 and HVPA60L10 concretes were tested for heat evolution. The heat evolution test under semi-adiabatic conditions is illustrated in Figure 2. A 50 mm thick polystyrene insulation was installed on each side of the cube mold (dimensions 450 x 450 x 450 mm). After mixing, the fresh concrete specimens were poured into the molds with insulation for concrete specimens ( $350 \times 350 \times 350$  mm). A thermocouple was placed in the middle point of each concrete specimen to measure the temperature for 168 hours. All 3 concrete mixtures were tested simultaneously in a laboratory room at an ambient temperature of  $25\pm2$  °C.

#### 2.3.4. Microstructure analysis

The scanning electron microscopy (SEM) in secondary electron mode, energy dispersive X-ray spectroscopy (EDS) and thermogravimetric analysis (TGA) techniques were used to investigate the microstructure of the pastes. The three paste specimens of OPC, HVPA70 and HVPA60L10 were analyzed for their microstructure at 28 days of age. Before the SEM/EDS investigation, the 1 cm<sup>3</sup> specimen was vacuumed at 10 Pa. SEM was operated to observe the formation of the pastes while the elements of the specific point within SEM photographs region of the hardened pastes were identified by EDS analysis.

Thermogravimetric analysis (TGA) was used to estimate the extent of dehydration, dehydroxylation, and decarbonation of the pastes by examining the mass loss at each temperature range. The paste specimen was ground into powder form before the TGA test. TGA was operated using a thermogravimetric analyzer (Pyris 1 TGA). The paste sample was heated



FIGURE 2. Setup for heat evolution testing.

to 900 °C in a nitrogen gas atmosphere at a heating rate of 10 °C per minute.

# 2.3.5. Estimation of CO2-equivalent emission of concrete

The CO<sub>2</sub> equivalent (CO<sub>2</sub>-e) emission of each concrete mixture is calculated by the sum of the volume of the materials in Table 2 multiplied by the CO<sub>2</sub> emission factors per unit of material listed in Table 3. The CO<sub>2</sub> emission factor of POFA for the grinding process was determined from the product of the electricity requirement (approx. 42 kWh/t) (29) and the CO<sub>2</sub> emission factor for electricity in Thailand, which is given as 0.5986 kg·CO<sub>2</sub>/kWh (30). It was assumed that POFA is transported in cement trailers (V-shaped) with a maximum load of 32 t, which have a CO<sub>2</sub> emission factor of 0.043 kg·CO<sub>2</sub>/t-km (30). The concrete plant was hypothesized to be in Saraburi province, close to the cement plant and other concrete mixtures such as LP and aggregates. The POFA transportation distance of 1,000 km was used in this study as this is the farthest distance of a palm oil factory from the concrete plant.

| TABLE 3. $CO_2$ emission factor. |  |                |  |  |  |  |
|----------------------------------|--|----------------|--|--|--|--|
| Materials (unit)                 | $CO_2$ emission factor<br>(kg·CO <sub>2</sub> /unit) | Refe-<br>rence |  |  |  |  |
| OPC (t)                          | 820.0  | (32)           |  |  |  |  |
| POFA (t)                         | 25.1   | (29, 30)       |  |  |  |  |
| LP(t)                            | 137.2  | (33)           |  |  |  |  |
| Sand (t)                         | 3.7  | (30)           |  |  |  |  |
| Granite (t)                      | 24.4   | (31)           |  |  |  |  |
| Water (t)                        | 0.8  | (30)           |  |  |  |  |
| Superplasticizer (t)             | 771.0  | (31)           |  |  |  |  |
| POFA transportation<br>(t-km)    | 0.043  | (30)           |  |  |  |  |

### **3. RESULTS AND DISCUSSION**

#### 3.1. Superplasticizer requirement

The superplasticizer requirement of concrete to keep the slump at 150-200 mm is presented in Table 2. OPC concrete required the superplasticizer of 1.5 kg/m<sup>3</sup> which was less than HVPA concretes. The superplasticizer requirements of HVPA60, HVPA70 and HVPA80 concretes were 6.2, 6.7 and 7.3 kg/m<sup>3</sup>, respectively. This result indicated that the ground

POFA content had a significant influence on the superplasticizer requirement. In other words, the more ground POFA the mixture contained, the higher the need for superplasticizer. Sripan et al. (34) found that POFA that was ground had less porosity than original POFA from the disposal area, but the particles of ground POFA still had irregular shapes. The irregular shapes of ground POFA particles affected the friction between themselves, between the particles of ground POFA and OPC, and between the particles of ground POFA and aggregates. Therefore, HVPA concretes required more superplasticizer than OPC concrete.

When the effect of LP on the superplasticizer requirement of HVPA concrete was considered. The HVPA70 concrete mixture was used to compare HVPA65L5 and HVPA60L10 concretes as the OPC replacement was the same. The superplasticizer requirement of HVPA65L5 and HVPA60L10 concretes (7.8 kg/m<sup>3</sup>) was higher than HVPA70 concrete (6.7 kg/m<sup>3</sup>). This was due to the fact that the LP had ultrafine particles ( $d_{50}$ = 2.64) compared to ground POFA ( $d_{50}$ = 8.50), which led to an increase in surface area. For this reason, the concrete containing LP resulted in higher water absorption which meant that more superplasticizer was required than for concrete without LP.

#### 3.2. Compressive strength

# 3.2.1. Effect of POFA on compressive strength of concrete

At 7, 14, 28 and 90 days, the compressive strengths of the OPC concretes were 57.1, 61.7, 65.9 and 78.0 MPa, respectively, as displayed in Figure 3. The compressive strength of HVPA concrete at 7 days was 34.7, 29.4 and 26.4 MPa for HVPA60, HVPA70 and HVPA80 concretes, respectively. The HVPA-containing concretes exhibited significantly lower early-age compressive strength than the OPC concrete, even though the W/B ratio of all HVPA concretes was less than that of OPC concrete. This demonstrated that the lightening of OPC content in the concrete resulted in reduced hydration products, which affected the decrease in early-age compressive strength in the HVPA concretes. It is generally known that the OPC reaction is primarily a reaction to generate the early strength of pozzolanic concrete, which depends on the W/OPC ratio. Considering the W/OPC ratio (without POFA), HVPA60, HVPA70 and HVPA80 concretes, it may be that these HVPA concrete mixtures still had the high W/OPC ratios compared to OPC concrete, even though POFA could absorb the water in the mixture. Therefore, the compressive strength of HVPA concrete decreased continuously when the ground POFA content escalated from 60 to 80 wt%. In other words, the early-age compressive strength of HVPA concrete correlated with the proportion of OPC in the mixture. The result agrees with the study by Herath et al. (35) who reported that the high-volume fly ash concrete (with 50–80 wt% fly ash) negatively affects concrete early-age compressive strength.



FIGURE 3. Compressive strength of concretes containing high-volume palm oil fuel ash.

However, as time progressed after 14 days, there was a growth in compressive strength. The compressive strengths of the HVPA60, HVPA70 and HVPA80 concretes increased to 46.5, 39.3 and 35.6 MPa respectively at 14 days and to 59.9, 46.1 and 45.4 MPa respectively at 28 days. Although all HVPA concretes still had lower compressive strengths than the OPC concrete, the compressive strengths of all HVPA concretes grew rapidly from 7 to 28 days compared to the OPC concrete. The percentage increment in compressive strength of the HVPA60, HVPA70 and HVPA80 concretes compared to the OPC concrete was 61, 51 and 46% respectively at 7 days and enhanced to 91, 70 and 69% respectively at 28 days. This was attributed to the ground POFA reacting with Ca(OH), from the hydration products, resulting in increased compressive strength over time owing to the pozzolanic reaction. Additionally, the voids in the paste matrix and the small gaps of paste-aggregates could be filled with ground POFA. As a result, secondary C-S-H compounds that formed by the pozzolanic reaction were able to fill the voids in the concrete structure more completely (8). The concrete compressive strength with a higher POFA content still tended to decrease at 90 days. The higher POFA replacement rates possibly resulted in the presence of many non-reactive POFA particles, which led to a reduction in compressive strength owing to pozzolanic reaction. It could be seen that HVPA60, HVPA70 and HVPA80 concretes were found to have a compressive strength of 70.9, 57.2 and 55.2 MPa at 90 days, corresponding to 91, 73 and 71 % compared to OPC concretes, respectively, whose values are close to their percentages at 28 days. It was a fact that in high-volume pozzolanic concrete, the compressive strength from the pozzolanic reaction was increased to a limited extent owing to the lack of Ca(OH), from cement hydration (36).

According to ACI 363R (25), the compressive strength should be 55 MPa or more for high-strength concrete. The HVPA60 concrete exhibited a compressive strength of 59.9 MPa at 28 days, exceeding the 55 MPa. Although the compressive strength of HVPA70 and HVPA80 concrete did not reach the required 55 MPa at 28 days, the compressive strengths of HVPA70 and HVPA80 concretes were 57.2 and 55.2 MPa at 90 days, respectively. It should be noted that the HVPA80 concrete only contained 112 kg/m<sup>3</sup> of OPC in the mixture.

# *3.2.2. Effect of limestone powder on compressive strength of HVPA concrete*

The above results demonstrated that the compressive strength of HVPA60 concrete was better than that of the other HVPA concretes. In addition, the HVPA60 concrete met the requirement according to ACI 363R (25) for high-strength concrete, while the HVPA70 concrete almost reached a compressive strength of 55 MPa at 28 days. For this reason, LP was admixed to the HVPA70 concrete mixture (constant OPC of 30 wt%) in an amount of 5 and 10 wt% to expect increase the early-age compressive strength while maintaining the lowest cement content. The compressive strengths of HVPA+LP concretes are exhibited in Figure 4. The compressive strengths for HVPA65L5 were 27.4, 35.5, 44.4 and 57.4 MPa at 7, 14, 28 and 90 days, respectively, while the compressive strengths for HVPA60L10 were 30.8, 39.5, 53.4 and 58.3 MPa at 7, 14, 28 and 90 days, respectively. In general, ground POFA acts as a pozzolanic material that produces extra strength when it reacts with portlandite, whereas LP is an inert material. However, the use of LP in appropriate dosage could enhance the compressive strength of concrete owing to filling and nucleation effects (14, 16). Compared to HVPA70 concrete, the compressive strength of HVPA60L10 increased slightly (by approximately 5%) at 7 days, as the LP consisted of solid, very small particles and had a higher specific gravity than the ground POFA, thus acting as a better filler material. On the other hand, HVPA65L5 concrete exhibited a slightly reduced compressive strength (by approximately 7%) at 7 days compared to the compressive strength of the HVPA70 concrete. This information suggested that the replacement of HVPA with 10 wt% LP (HVPA60L10 concrete) may have influenced the mechanism of nucleation and filler effects better than LP 5 wt% (HVPA65L5 concrete). Therefore, the compressive strength of HVPA60L10 concrete was better than that of HVPA65L5 concrete for all test ages. Ramezanianpour et al. (18) informed that in low W/B ratio concrete, the use of LP as a substitute for OPC at 10 wt% exhibited better compressive strength than concrete with 5 wt% LP at 3 days and the higher strength was maintained until 180 days. Similar to the results of Klathae et al. (37), who concluded that LP at 10 wt% is suitable for use in bagasse-ash concrete.



FIGURE 4. Effect of limestone powder on compressive strength of HVPA concrete.

#### 3.3. Elastic modulus of concrete

The elastic modulus of all concretes is illustrated in Figure 5. The elastic modulus of the OPC concrete were 35.1 and 38.7 GPa at 28 and 90 days, respectively. For the HVPA concretes, which contained ground POFA in proportions of 60-80 wt%, the elastic modulus values were in the range of 27.5-30.7 GPa and 30.6-35.1 GPa at 28 and 90 days, respectively. This meant that the HVPA concretes had slightly fewer elastic modulus than the OPC concrete. The result was in agreement with Herath et al. (35) and Siddique (38), who summarized that the concrete with high fly ash content tended to extenuate the elastic modulus of concrete compared to conventional concrete, since the elastic modulus depended on the concrete compressive strength. Additionally, the OPC concrete mixture in the current study had a greater proportion of coarse aggregate (990 kg/m<sup>3</sup>) than the HVPA concrete mixes  $(900-935 \text{ kg/m}^3)$ , which affected the increased elastic modulus (39).

For HVPA concretes with LP, the HVPA65L5 and HVPA60L10 concretes had the elastic modulus at 28 and 90 days in the range of 30.5–33.7 GPa and 30.7–34.0 GPa, respectively. Meddah et al. (40) found that

the elastic modulus of OPC concrete was hardly affected at an LP content of 15 wt%. However, the elastic modulus of the concretes decreased by about 15 and 33% when LP was replaced by 25 and 45 wt%, respectively. From the results of the current study, the use of LP at 5 or 10 wt% in the HVPA concrete mixture had no significant effect on the elastic modulus of the concrete. This was due to the fact that the elastic modulus of HVPA concrete with or without LP was highly dependent on its compressive strength.



FIGURE 5. Elastic modulus of concretes.

## 3.4. Comparison of elastic modulus of highvolume pozzolan concretes with suggested ACI

The relation between elastic modulus and compressive strengths of the HVPA concretes in this study was investigated together with concretes containing a high proportion of other pozzolans (≥50 wt%), such as high-volume fly ash (HVFA), high-volume bagasse ash (HVBA) and high-volume coal bottom ash (HVC-BA) (38, 41-44), as shown in Figure 6. The elastic modulus from ACI 318M (43) can be determined as Equation [1] for normal weight concrete. For ACI 363R (25), the elastic modulus for normal weight concrete is represented by Equation [2], which contains boundary conditions for compressive strengths between 21 MPa and 83 MPa. Both equations are used to compare the elastic modulus of high-volume pozzolan concrete (HV-pozzolan) from the current study with other research results. Based on the data of HV-pozzolan concrete, the equation for the elastic modulus of concrete (E<sub>c</sub>) is presented based on Equation [3]. It was interesting to note that most of the data of HV-pozzolan concrete were close to the recommended line of ACI 363R (25). When calculating the compressive strength at 21 MPa using Equation [3], the elastic modulus value of HV-pozzolan concrete was 16 and 17% lower than the elastic modulus value calculated from the Equation [1] of ACI 318M (45) and Equation [2] of ACI 363R (25), respectively. However, for the compressive strength at 83 MPa, the elastic modulus of HV-pozzolan concrete was about 6% higher than the value suggested by ACI 363R (25), while the elastic modulus value of HV-pozzolan was still about 8% less than the elastic modulus value calculated by the equation of ACI 318M (45). Further information from Figure 6 for the lower compressive strength of about 50 MPa or the square root of the compressive strength of 7 MPa shows that the trend line of the elastic modulus of HV-pozzolan concrete is underestimated based on ACI 363R (25).

From ACI 318M (45): 
$$E_c = 4.73 \sqrt{f_c}$$
 [1]

From ACI 363R (25): 
$$E_c = 3.32\sqrt{f_c+6.9}$$
 [2]

From HV-pozzolan: 
$$E_c = 4.81\sqrt{f_c} - 3.5$$
,  $R^2 = 0.71$  [3]

where  $E_c$  is the elastic modulus of concrete (GPa) and  $f_c$  is the compressive strength of concrete (MPa).



FIGURE 6. Relationship between elastic modulus and square root of compressive strength.

#### 3.5. Shrinkage strain of concrete

The shrinkage strain of concrete is displayed in Figure 7. The shrinkage strain of the OPC concrete enhanced from 161 to 874 micro strains from 1 to 28 days. Although the W/B ratio of the OPC concrete at 0.3 was higher than that of the HVPA concretes (W/B ratio = 0.25), the shrinkage strains of the HVPA60, HVPA70 and HVPA80 concretes decreased by approximately 35, 45 and 58% of the values for the OPC concrete at day 1 and 14, 23 and 32% of the values for the OPC concrete at 3 days of age. It should be noted that self-drying occurred in high-strength concrete without external water supply and that autogenous shrinkage occurred in the early stages owing to the hydration reaction in the cement (46). Therefore, the use of high-volume POFA could reduce the shrinkage strain owing to the lower OPC content, which led to a reduction in the hydration rate of the cement. The shrinkage strain of all concretes increased continuously with increasing concrete age and the shrinkage strain was lower with a higher POFA content. At 28 days, the shrinkage strains of the HVPA60, HVPA70 and HVPA80 concretes were also about 10, 14 and 19 % lower than those of the OPC concrete, respectively. These results were consistent with another study (47), which found that the total shrinkage of the concrete decreased by approximately 30% and 40% after 28 days when the concrete samples contained 55% and 65 wt% of fly ash in the binder, respectively. This result was due to the fact that the concrete with a high POFA or pozzolanic content in the mixture resulted in a reducing cement paste (OPC + water), while the remaining POFA from the pozzolanic reaction acted as an aggregate (48). Consequently, the shrinkage strains of these HVPA concretes decreased as time progressed.



In HVPA concretes with the same cement content (HVPA70, HVPA65L5 and HVPA60L10 concretes), the shrinkage strain increased with increasing LP content in the mixture (replacement of POFA at 5 and 10 wt%) at an early age of 1-3 days. This can be attributed to the fact that the nucleation effect of the LP content accelerated the hydration reaction (15, 17). Consequently, the higher autogenous shrinkage (at an early age) of the concrete containing LP occurred due to water loss during the reaction and intensified the hydration degree. De weerdt et al. (49) observed that OPC mortar containing 5 wt% LP had higher early-age shrinkage than OPC mortar without LP. However, as time progressed, the shrinkage strains in the HVPA65L5 and HVPA60L10 concretes showed similar values to the HVPA70 concrete. This result also demonstrated that the HVPA70, HVPA65L5 and HVPA60L10 concretes exhibited lower shrinkage than OPC concrete, which could be owing to the reduction in autogenous shrinkage and drying shrinkage. It should be noted that the HVPA70, HVPA65L5 and HVPA60L10 concretes had the same OPC replacement of 70 wt%; therefore, the less cement paste resulted in a reduction in autogenous shrinkage. In addition, the pozzolanic product of POFA could refine the pores in the concrete and also serve as a filler together with POFA and LP. Therefore, the pores of the concrete were expected to be filled by both POFA and LP. Consequently, the slowing of water evaporation led to a lessening in the drying shrinkage of the concrete.

### 3.6. Heat evolution of concrete

OPC, HVPA70 and HVPA60L10 concretes were used to investigate the effect of POFA and PA on the heat generation of the concrete (see Figure 8). OPC concrete got a maximum temperature rise of 40.9 °C per 500 kg/m<sup>3</sup> OPC content, which corresponds to a time of 15 hours at maximum temperature. This result is close to that reported by Amnadnua et al (50), who reported a temperature rise of 42 °C per 500 kg/m3 OPC content 18 hours after mixing for OPC concretes with a W/B ratio of 0.25. For the HVPA70 and HVPA60L10 concretes, the maximum temperature rises were 19.4 and 19.7 °C at the times of 41 and 40 hours, respectively. Although the appropriate amount of LP replacement in the concrete could accelerate the hydration reaction, the heat evolution for the HVPA60L10 concrete was similar to that for the HVPA70 concrete. This finding indicated that the use of LP at 10 wt% binder had little effect on the heat evolution of HVPA concrete. In other words, reducing the OPC in the mixture was the main factor influencing the reduction of heat evolution of concrete. This reduction in OPC content resulted in a decreased tricalcium silicate (C<sub>3</sub>S) content, leading to a lower heat temperature rise during the hydration reaction. The use of supplementary material up to 70 wt% in the concrete mixture not only reduced the peak temperature rise by about 50%, but also delayed it by about 25 hours compared to the OPC concrete. In ad-



FIGURE 8. Heat evolution curves of different concrete types.

dition, both the HVPA70 and HVPA60L10 concretes required high superplasticizer dosages, which resulted in a delay in heat hydration (51). The temperature rises of HVPA70 and HVPA60L10 concretes was started 20–40 hours after mixing, the temperature change of these HVPA concretes was equal to that of OPC concrete about 60 hours.

#### 3.7. Microstructure of pastesa

#### 3.7.1. SEM/EDS

Figure 9 shows the microstructure characterization of high-volume pastes containing 70 % POFA and 60 wt% POFA blended with 10 wt% LP in comparison the microstructure of OPC paste. Figure 9a shows the microstructure of OPC paste. It was clear that the hydration products of OPC paste had a fibrous network structure that intertwines in an across-linked manner, connecting and appearing compact. The EDS spectra of OPC paste were Si and Ca, indicating the formation of C-S-H, which occurred from the hydration reaction of cement.

The microstructure image of the high volume POFA pastes matrix (Figure 9b and Figure 9c) shows the good quality of adhesion at the interfaces of the structural matrix. The Ca, Si and Al elements detected in the high volume POFA paste could induce the formation of C-S-H and C-A-H compounds such as the hydration and pozzolanic products. For example, at point 2 in Figure 9b, the compact and homogeneous paste matrix has a high content of Ca, Si and small amounts of K, which could be a pozzolanic product of POFA. However, the un-hydrated or unreacted POFA particles can be seen in the structural matrix of the HVPA70 paste. The surface of POFA was still smooth, and no hydrate had formed on their surface (point 1, Figure 9b). It is worth noting that the EDS results of HVPA70 at the unreacted position of the POFA particle at point 1 in Figure 9b indicated the elements Si and K, which are related to the chemical composition of the raw materials of POFA. For the paste with the addition of LP (Figure 9c), the SEM image of HVPA60L10 paste displays an altered structure, there is an improvement in the microstructure of the samples. The HVPA60L10 paste exhibits a more compact matrix compared to the HVPA70 paste and the particle of POFA (point 1 in Figure 9c) appeared to be coated with a thin layer of hydration products. When comparing the EDS results of HVPA70 and HVPA60L10, it was found that the Ca element increased with the replacement of LP in the mixture. The Ca content in the HVPA60L10 paste was not only due to the hydration and pozzolanic products, but also

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due to the raw LP that was replaced in the mixture. This was because the raw LP consisted of a configuration of Ca and O atoms in calcite that was similar to the CaO layers in the formation of C-S-H (19). Therefore, more elemental Ca was detected in the EDS results for the HVPA60L10 paste than for HVPA70 paste. In addition, the EDS results at point 2 in Figure 9c show high Ca, O and C elements, suggesting that these are LP particles. The LP had bonding on interface of POFA particles, resulting in the formation of more compactness and less permeable voids. This result was related to the findings of Liu and Yan (13), who found that the LP could act as a filler and improved the microstructural matrix of the cement paste by reducing porosity. These results and the SEM images confirm the effect of limestone powder on the compressive strength in section 3.2.2, according to which the compressive strength of the concrete could be increased by adding LP.





FIGURE 9. SEM morphologies and EDS spectra of HVPA and HVPA60L10 pastes. A: OPC paste; B: HVPA70 paste; C: HVPA60L10 paste.

# 3.7.2. TGA

The hydration products such as C-S-H, C-A-H and Ca(OH)<sub>2</sub>, including the CaCO<sub>3</sub> compound of the OPC, HVPA70 and HVPA60L10 pastes were analyzed by mass loss at specific temperature boundaries as shown in Figure 10. The loss of bound water, i.e., the decomposition of C-S-H and C-A-H (dehydration), occurs in the temperature range of 105-430 °C. The dehydroxylation of Ca(OH)<sub>2</sub> and the decarbonation of CaCO<sub>3</sub> occur in the temperature ranges of 430 -550 °C and 550-900 °C, respectively (52).

The mass losses during the dehydration phase of OPC, HVPA70 and HVPA60L10 pastes were 11.1 %, 9.9 % and 10.0 %, respectively, which corresponds directly to their compressive strength. In other words, a higher mass loss in the dehydration phase of the OPC paste resulted in a higher compressive strength of the OPC concrete. It is noteworthy that the mass loss of OPC paste in the temperature range of 430-550 °C was the highest at 3.0 % due to the decomposition of Ca(OH)<sub>2</sub>. In contrast, the mass losses of HVPA70 and HVPA60L10 pastes in the dehydroxylation phase were lower at 0.7% and 1.5%, respectively. The observed values of this result imply that the HVPA70 and HVPA60L10 pastes had lower Ca(OH), content, which could be due to their lower OPC content and the potential consumption of Ca(OH), by pozzolanic reactions with POFA. In the decarbonation phase, the OPC, HVPA70 and HVPA60L10 pastes exhibited mass losses of 3.5, 5.4 and 8.1%, respectively. In particular, the



HVPA60L10 paste.

HVPA60L10 paste, which contained a considerable amount of  $CaCO_3$  due to the presence of LP, showed the highest mass loss during decarbonation. Furthermore, it was observed that the HVPA70 paste had a greater mass loss compared to the OPC paste. This can be attributed to the high amount of unburned carbon in the POFA, as shown by its LOI value.

# **3.8.** Estimation of CO<sub>2</sub>-equivalent emission of concrete

Figure 11a shows the  $CO_2$ -e value of the individual concrete mixtures in this study. The  $CO_2$ -e value of OPC concrete was 438 kg· $CO_2$ /m<sup>3</sup>. This result was consistent with the data for OPC concrete from another study for the similar strength class of 50–60 MPa,



FIGURE 11. A: CO<sub>2</sub>-e emission of concrete in this study; B: Relationship between CO<sub>2</sub>-e emission and compressive strength of concrete.

for which about 400-460 kg·CO<sub>2</sub>/m<sup>3</sup> was reported (36, 31). The  $CO_2$ -e value of OPC concrete was related to the amount of OPC in the mixture (94% of the total CO<sub>2</sub>-e of the OPC concrete mixture). HVPA concretes (based on the feedstock only) had CO<sub>2</sub>-e values of 222, 178, 134, 182 and 185 kg·CO<sub>2</sub>/m<sup>3</sup> for HVPA60, HVPA70, HVPA80, HVPA65L5 and HVPA60L10, respectively. When the addition of POFA transportation at 1,000 km, the CO<sub>2</sub>-e values of HVPA60, HVPA70, HVPA80, HVPA65L5 and HVPA60L10 concretes increased to 236, 195, 153, 198 and 200 kg·CO<sub>2</sub>/m<sup>3</sup>, respectively. The result indicated that the POFA transportation distance of 1,000 km had little effect on the CO<sub>2</sub>-e intensity of the HVPA concretes. Figure 10a also demonstrates that although the HVPA concretes are able to reduce the OPC content in the mixtures to 60-80 wt%, the CO<sub>2</sub>-e value still depends on the amount of OPC in the mixtures. For the HVPA60 and HVPA80 concretes, for example, the CO<sub>2</sub>-e values owing to OPC in the mixtures (184 and 92  $kg \cdot CO_2/m^3$ respectively) accounted for around 84 and 70% of the total CO<sub>2</sub>-e values of these concretes.

Figure 11b illustrates the relation between  $CO_2$ -e and compressive strength at 28 days for the other

OPC concretes (37, 54-57) in comparison to a similar strength class of HVPA concretes. It is obvious that the CO<sub>2</sub>-e value of OPC concrete increased with increasing compressive strength. As already mentioned, the CO<sub>2</sub>-e value in concrete is mainly generated by the OPC in the mixture. The increased compressive strength required a higher OPC content to keep the W/C ratio low, resulting in high CO<sub>2</sub>-e values. Figure 10b gives more information about the trend line with the CO<sub>2</sub>-e values for OPC concretes. When the CO<sub>2</sub>-e values of HVPA concretes are compared with those of OPC concretes with the same strength (from the trend line), the CO<sub>2</sub>-e values for HVPA60, HVPA70, HVPA80, HVPA65L5 and HVPA60L10 concretes decrease by approximately 44, 49, 62, 47 and 51%, respectively. However, even when including the transportation of POFA (approx. 1,000 km), HVPA60, HVPA70, HVPA80, HVPA65L5 and HVPA60L10 concretes still had lower CO2-e values than OPC concrete (40, 44, 56, 42 and 47%, respectively). This result indicates that concrete with low CO<sub>2</sub>-e emissions can be produced by using high replacement rates of OPC by POFA and LP in the mixture.

# 4. CONCLUSIONS

In this experiment, the mechanical properties, shrinkage strain, heat evolution, microstructure, and CO<sub>2</sub> emissions of HVPA concretes with an LP content of 5 or 10 wt% were investigated. The results can be summarized as follows:

- Concrete with POFA content of 60 wt% (HVPA60 concrete) achieved the compressive strength of 59.9 and 70.9 MPa at 28 and 90 days, respectively. In addition, the compressive strength of the HVPA80 concrete at 90 days met the requirements of ACI 363R for high-strength concrete (more than 55 MPa).
- 2. The addition of 5 and 10 wt% LP to concrete with high-volume POFA at a replacement rate of 70 wt% maintained comparable mechanical properties in terms of compressive strength and elastic modulus to HVPA70 concrete. This indicates the viability of ternary mixtures (PO-FA+LP+OPC) for strength development and the potential for further reduction of OPC content in high-strength concrete for construction applications.
- 3. The elastic modulus of high volume POFA concretes without and with LP was more similar to the elastic modulus of the proposed ACI 363R than that of ACI 318M. However, for high volume pozzolan concrete, the elastic modulus could be lower than the modulus of elasticity

proposed by ACI 363R when the compressive strength was below 50 MPa.

- 4. The use of LP in the HVPA concrete mixture resulted in a slight increase in early-age shrinkage strain compared to HVPA concrete without LP at the same replacement level. However, the use of high POFA content, both with and without LP, effectively reduced the shrinkage strain of the concrete compared to OPC concrete.
- 5. The addition of 10 wt% LP in combination with 60 wt% POFA had a modest effect on the increase in heat evolution of the concrete compared to concrete containing only POFA with a replacement level of 70 wt%. However, the peak temperatures of HVPA70 and HVPA60L10 concretes were significantly lower, approximately 20°C or about 50% lower than OPC concrete, and the peak temperature time was delayed by 25 hours.
- 6. The SEM micrograph of the HVPA pastes revealed the reaction products, which were confirmed by the EDS and TGA results. However, unreacted POFA particles were observed in the HVPA70 paste, and there were gaps between the hydrated products and the unreacted POFA particles. The addition of LP to the HVPA paste effectively filled the gaps between the hydrated products and the unreacted POFA particles, resulting in a significant improvement in the microstructure of the HVPA paste.
- 7. In terms of environmental impact, the concrete with a high replacement of OPC with POFA and LP was able to significantly reduce CO<sub>2</sub>-e levels by 44–62% compared to OPC concrete of similar strength. Although the CO<sub>2</sub>-e value of the concrete depended on the transportation of raw materials, including POFA transportation over 1000 km, the CO<sub>2</sub>-e emissions of HVPA concrete were still considerably lower than those of OPC concrete.

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#### Authorship contribution statement

**Penpichcha Sanit-in**: Investigation, Writing - original draft.

Thepparit Charoensuk: Data curation, Methodology, Resources.

**Saofee Dueramae**: Formal analysis, Investigation, Writing - review & editing.

Akkadath Abdulmatin: Formal analysis, Funding acquisition, Project administration, Visualization, Writing - original draft.

**Pokpong Rattanachu**: Validation, Writing - review & editing.

**Weerachart Tangchirapat**: Conceptualization, Validation, Writing - review & editing.

Chai Jaturapitakkul: Validation, Writing - review & editing.

# **Declaration of competing interest**

The authors of this article declare that they have no financial, professional or personal conflicts of in-terest that could have inappropriately influenced this work.

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