



Properties of Cement Paste Containing Sustainable Non-Conforming Metakaolin for Minimising Environmental Impact

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ABSTRACT

In the building and construction industry, the use of ordinary Portland cement is often associated with a high carbon footprint, significantly contributing to climate change. Consequently, extensive research has explored the benefit of supplementary cementitious materials (SCMs) to reduce CO₂ emissions. One such material is metakaolin, which is widely available and has gained popularity. However, not all metakaolin properties meet the requirements set by standard specifications for SCMs. This study explores the use of non-conforming metakaolin as a partial replacement in cement paste to minimise environmental impact. Cement paste samples incorporating 0%, 5%, 10%, 15%, and 20% metakaolin were prepared using a 0.6 water-to-cement ratio and tested for properties such as compressive strength, water absorption, ultrasonic pulse velocity, and density at curing periods of 7, 14, and 28 days. Results showed that water absorption and pulse velocity increased with higher metakaolin content, while density and compressive strength improved up to a 10% replacement level before declining. The highest compressive strength of 66.34 MPa was achieved at a 10% replacement level after curing at 28 days. These findings suggest that non-conforming metakaolin has the potential to be used as a partial replacement for ordinary Portland cement, offering a viable alternative with a lower environmental impact and reduced carbon footprint.

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1. INTRODUCTION

Cement is extensively used in the construction industry as a fundamental binding material for producing concrete-based composite materials. The production and use of concrete are driven by infrastructure expansion and construction activities, which often have a significant environmental impact, attributing for approximately 8% of global carbon dioxide (CO₂) emissions [1]. Raw materials such as limestone, clay, shale, sand and iron ore used in cement production are heated to temperatures exceeding 1,450 °C, releasing CO₂ both from the thermal decomposition of minerals and from the combustion process [2]. Nearly 43% of the CO₂ emitted to the atmosphere during the cement manufacturing process is through the calcination process alone [3]. Although cement products can reabsorb almost half of the CO₂ originally released during their production, the cement industry remains one of the largest sources of CO₂ emissions globally, particularly due to the decomposition of carbonates.

To address this issue, numerous researchers have explored the partial replacement of ordinary Portland cement in concrete mixes with supplementary cementitious materials (SCMs). Incorporating SCMs as partial substitutes for ordinary Portland cement is an effective strategy to minimise CO₂ emissions associated with cement production. Common SCMs include natural pozzolans such as calcined clays, artificial pozzolans from industrial by products such as silica fume, fly ash, ground granulated blast furnace slag, as well as alternative and emerging waste such as rice husk ash, waste glass powder, sewage sludge ash and palm oil fuel ash [4, 5]. Both natural and artificial pozzolans consists of siliceous and aluminous materials that react with calcium hydroxide when there is the presence of water to form cementitious compounds such as calcium silicate hydrate (C-S-H) gel during hydration at ambient temperature [6].

Among these materials, metakaolin is considered one of the most effective. Produced by calcining the mineral kaolin at elevated temperatures between intervals of 600 and 800°C, metakaolin serves as a high-performance cement replacement,

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enhancing both the durability and strength of concrete [7]. Beyond improving strength and durability properties, the incorporation of metakaolin also helps address sustainability challenges, particularly by promoting the development of low-carbon cements designed to minimise environmental impact. This is achieved through the utilisation of metakaolin as part of sustainable mineral resource conservation practices, which emphasise responsible extraction and the efficient, maximal use of materials to ensure mineral resources remain available while minimising their environmental and societal impacts [8,9].

ASTM C618-25 [10] provides technical guidelines for evaluating fly ash and natural pozzolans in both raw and calcined forms that are utilized as supplementary cementitious materials (SCMs) in concrete. The standard specifies the chemical, physical, and uniformity requirements that pozzolans must meet to ensure their suitability for use. The chemical requirements specify limits for sulphur trioxide (SO_3), moisture content, and loss on ignition (LOI), as well as a minimum total amount of combination between silicon dioxide (SiO_2), aluminium oxide (Al_2O_3), and iron oxide (Fe_2O_3) compounds. The physical and uniformity requirements include assessments of fineness, strength activity index, water demand, volumetric stability (measured by autoclave expansion), and density. However, some alternative SCMs exhibit pozzolanic or hydraulic reactivity and can beneficially enhance the strength, durability, workability, or other properties of concrete, even though they do not fully comply with the specifications of ASTM C618.

Therefore, this study investigates the performance of non-conforming metakaolin at various replacement levels in cement paste mixtures. The experimental program evaluates the properties such as compressive strength, water absorption, ultrasonic pulse velocity (UPV), and density of cement pastes incorporating non-conforming metakaolin. The aim is to minimise environmental impact through the adoption of low-carbon cementitious materials such as metakaolin, and to assess whether non-conforming metakaolin can also boost the strength and durability properties of cement paste.

2. MATERIALS AND METHODS

2.1 Materials

The materials used to produce the cement paste in this study were ordinary Portland cement (OPC), metakaolin (MK), and water. The OPC was provided by Cement Industries of Malaysia Berhad (CIMA) under the brand name Blue Lion OPC, meeting the requirements of MS EN 197-1 [11] standard. The MK was supplied by Delta Corp Sdn. Bhd. and was obtained by converting kaolin into metakaolin at a temperature of 800°C for two hours. Water was sourced from Syarikat Air Perlis (SAP) in accordance with MS EN 1008:2010 [12].

Table 1 shows the characteristics of OPC and MK with respect to the chemical requirements specified in ASTM C618-25 [10] for classes N, F and C of raw and calcined natural pozzolan. The MK does not comply with standard specifications due to its high moisture content and loss on ignition, which exceed the maximum limits of 3% and 10%, respectively. However, the chemical composition of MK, in terms of the combined $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ content, as well as the CaO and SO_3 contents, meets the 70% minimum requirements, a maximum of 18%, and a maximum of 5%,

respectively. Therefore, this MK is classified as a non-conforming pozzolan material, and its performance was further assessed in the cement paste mixtures.

The high moisture content of 3.2% and LOI value of 12.75% usually indicates the presence of chemically bound water and unburnt carbon in the MK, potentially due to the incomplete dehydroxylation during calcination procedure. This might cause the MK to act as inert filler material when substituting in the cement paste. Although this low grade or non-conforming MK often associated with lower reactivity and poorer long-term durability, the level of performance is relatively unknown, and the further analysis can help determine the suitability of this material as supplementary cementitious materials.

Regarding to calcination procedure, the temperature of 800°C is at the acceptable limit for transformation of kaolin into metakaolin for supplementary cementitious material production. While this temperature should ideally reduce LOI and moisture content to achieve conforming range, the high residue LOI and moisture content could be due to high initial carbon content and bound water in the raw kaolin. However, kaolin particles are natural occurring materials which may behave varying depending on the sources. Therefore, by applying different duration and temperature could have brought the LOI and moisture content into the conforming range as long as full dehydroxylation is achieved.

Table 1. Characteristics of OPC and MK Subjected to Chemical Requirements by ASTM C618

Chemical Requirements	Class			OPC	MK	Description
	N	F	C			
$\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ (min, %)	70	50	50	28.93	94.45	Conforming
CaO (max, %)	18	18	18	62.70	0.27	Conforming
SO_3 (max, %)	4	5	5	2.63	0.01	Conforming
Moisture content (max, %)	3	3	3	0.64	3.20	Non-conforming
Loss on ignition (max, %)	10	6	6	2.70	12.75	Non-conforming

*Note: The conforming status is only applicable to MK under this standard for raw or calcined pozzolans, and is not applicable to OPC. The OPC data are provided for comparison to highlight the differences.

2.2 Methods

The mixture design of the cement paste incorporated MK as a partial replacement for ordinary Portland cement at proportions of 0%, 5%, 10%, 15%, and 20%. Each mixture had a water-to-cement ratio of 0.6. After mixing, the fresh cement paste was placed and compacted into cube moulds of $50\text{ mm} \times 50\text{ mm} \times 50\text{ mm}$ dimensions. Once demoulded, all samples were water-cured in a curing tank for 7, 14, and 28 days before testing.

The density and water absorption tests for the specimens were conducted using air-dried and oven-dried methods, respectively, in accordance with ASTM C642-21 [13]. Compressive strength was determined using a Computerized Compression Machine following ASTM C109/C109M-24 [14], while the Ultrasonic Pulse Velocity (UPV) test was executed as a non-destructive test fulfilling the ASTM C597-22 [15], as

shown in Figure 1. Correlation analysis was performed using Microsoft Excel v2502.

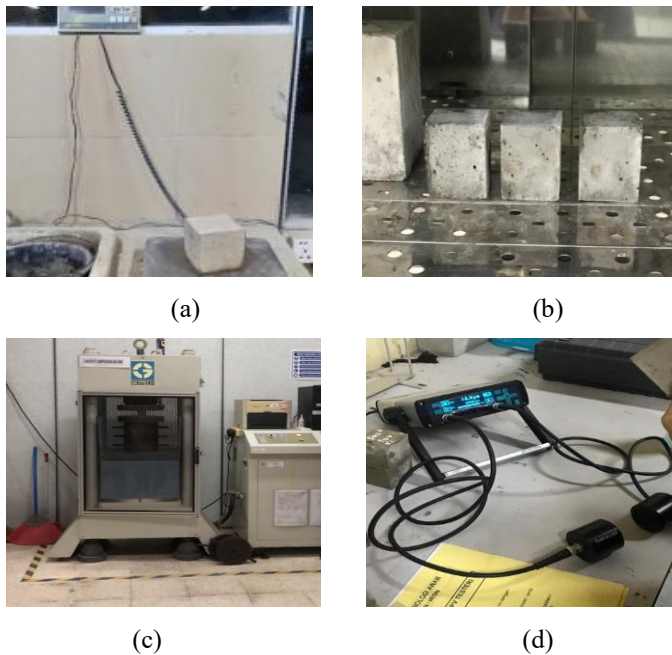


Fig. 1. Testing of Cement Paste Containing Non-Conforming Metakaolin at Various Levels and Curing Periods: (a) Air-Dried for Density and Water Absorption Test, (b) Oven-Dried for Density and Water Absorption Test, (c) Compressive Strength Machine, and (d) Ultrasonic Pulse Velocity

3. RESULTS AND DISCUSSION

3.1 Density

Figure 2 shows the density of cement paste incorporating 0, 5, 10, 15, and 20% of non-conforming metakaolin at curing periods of 7, 14 and 28 days. In all samples, the density of the cement paste increased with the curing periods, at 7, 14, and 28 days. This is because, as hydration progresses with an increasing curing period, the volume of hydration products fills an empty portion of the pore space, increasing the density of the cement paste. However, as the metakaolin content increased, the density of the cement paste containing non-conforming metakaolin rose only up to a 10% replacement level, after which it began to decrease. At 10% metakaolin content, the maximum density recorded was 1976 kg/m³ at 28 days. However, this value remained lower than that of the control sample, which measured at 2050 kg/m³.

The lower density of cement paste containing metakaolin is attributed to the lower specific gravity of metakaolin compared to OPC. Metakaolin has a specific gravity of approximately 2.65 g/cm³, whereas OPC has a specific gravity of about 3.15 g/cm³. As a result, incorporating metakaolin into the mixture reduces the overall density of the cement paste due to the increased water demand. Additionally, the increase in density at 10% metakaolin content can be attributed to the formation of calcium silicate hydrate (CSH) gel [16]. However, beyond this threshold, the additional CSH gel formation is insufficient to offset the reduction in density caused by the increased metakaolin content.

Apart from lower specific gravity of MK as compared to OPC, the lower density performance of cement paste containing MK particle across all levels of replacement in relation to control sample can be attributed to low reactivity of the MK due to high LOI and non-conforming MK, as well as dilution effect where the cementitious content was reduced as increased in MK replacement levels. This makes the benefits of the pozzolanic reaction by the MK pozzolans was insufficient to promote the hydration in the mixture of displaced cement for better densities.

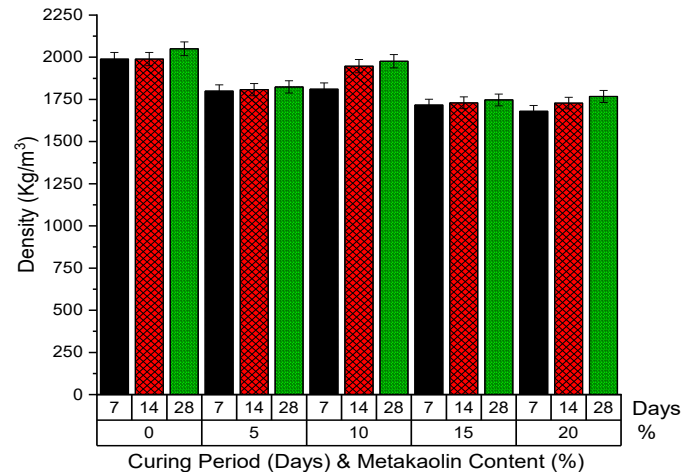


Fig. 2. Density of Cement Paste Containing Non-Conforming Metakaolin at Various Levels and Curing Periods

3.2 Water Absorption

Figure 3 shows the water absorption of cement paste containing 0, 5, 10, 15, and 20% non-conforming metakaolin at curing periods of 7, 14, and 28 days. In all samples, the water absorption of the cement paste increased with the curing period. At the early stage of hydration on 7 days of curing, the cement paste approaches saturation, resulting in a significant difference in water absorption uptake per unit area. However, a notable increase in water absorption is observed during the later stages of hydration, particularly at 28 days of curing. This process contributes to the overall increase in water absorption in cement paste containing non-conforming metakaolin.

As metakaolin content increases, the water absorption of the cement paste also increases. This is due to the higher capillary absorption caused by the poor bonding between metakaolin and the hydrated cement paste, leading to increased water retention [17]. Additionally, metakaolin has a larger specific surface area than OPC, giving it a higher water absorption capacity, especially at higher substitution levels. However, cement paste containing 5% metakaolin exhibited lower water absorption compared to the control sample. The absorption values ranged from 24.1% to 28.7% for the metakaolin mixture, while the control sample displayed values between 31.5% to 37.1%. This lower absorption is attributed to the fact that 5% metakaolin is insufficient to enhance hydration and produce enough CSH gel, leading to more voids and a more porous structure in the cement paste.

Typically, pozzolans help to reduce water absorption through pore refinement which subsequently may reduce porosity. However, in this case the high surface area and high moisture content of this specific MK increased the water demand in the mixture leading to increased water absorption.

At a fixed water-to-cement ratio of 0.6, the higher MK content reduced workability which leading to formation of interconnected capillary pores or entrapped air in the mixture which facilitate higher water uptake in the mixture casing high water absorption.

In the case of lower water absorption in the 5% MK replacement level than control sample, the MK particles at low levels seem to achieve optimal dispersion, effectively filling the interstitial spaces between cement grain during hydration without significantly increasing the water demand through filling effects. Beyond 5% MK replacement, the agglomeration of MK particles at higher levels causing starvation or high-water demand in the mixture which likely to create more porous network causing the rise of water absorption in the mixture.

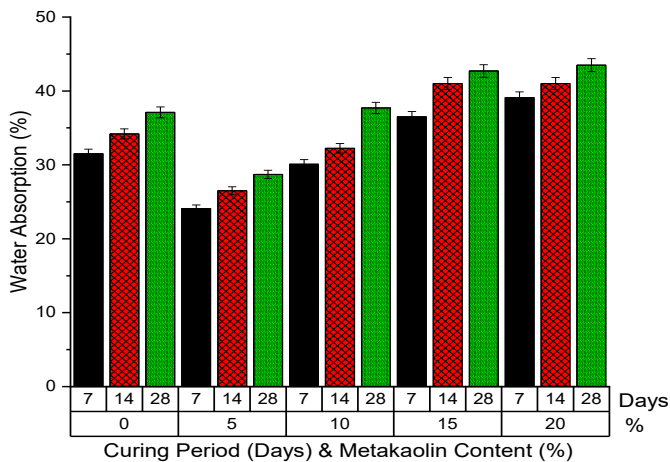


Fig. 3. Water Absorption of Cement Paste Containing Non-Conforming Metakaolin at Various Levels and Curing Periods

3.3 Compressive Strength

Figure 4 shows the water absorption of cement paste containing 0, 5, 10, 15, and 20% non-conforming metakaolin at curing periods of 7, 14, and 28 days. In all the cement paste samples, compressive strength increased with the curing period. This is due to the pozzolanic reaction, in which metakaolin reacts with calcium hydroxide to produce CSH gel, enhancing hydration over time [18]. The 28-day curing period is considered the mature stage for hydration. With increasing metakaolin content, the compressive strength values of the cement paste increased up to a 10% replacement level before decreasing. This indicates that 10% is the optimal metakaolin substitution level, allowing for complete hydration and the most reactive pozzolanic reaction. However, beyond this point, additional metakaolin reduces the compressive strength.

Despite the optimal metakaolin content at 10%, the compressive strength remained lower than that of the control sample, which ranged between 98.33 MPa and 122.84 MPa, compared to 53.338 MPa to 66.34 MPa for the cement paste containing metakaolin samples. Additionally, all samples exhibited a similar failure pattern under compression. Cracking or failure primarily occurred on the four exposed sides, while the top and bottom surfaces showed little to no visible damage, as shown in Figure 5 and Figure 6. This indicates a satisfactory mode of failure when the compressive load was applied.

The failure pattern where damage occurred on the four exposed sides or known as semi-explosive is one of the typical satisfactory failure modes of cube specimens where other being non-explosive and explosive failure patterns. For a small cube size of 50mm, the friction between the loading platens and the cube faces provides lateral restraint of confinement zone at the top and bottom. This caused the side effect damage for 50 mm cubes more profound compared to 100 mm or 150 mm cubes. The bigger cubes might be subjected to the non-explosive failure mode where shear cracking is often observed.

Also to note that, due to the non-conforming nature of MK particles used in the cement paste mixture, the high moisture content and LOI contains impurities such as unburnt carbon and bound water can acts as inert filler to the mixture. This means the MK would be less reactive in the pozzolanic reaction and not contributing strength in the cement matrix. This aligns with the observed lower compressive strength in all MK replacement mixtures as compared to the control sample. For long term period, the presence of residual carbonates due to high LOI can increased the carbonation in the material and reduce the durability performance of concrete as observed by Do et al. [19] and Geu et al. [20].

Since the water-to-cement ratio was fixed at 0.6 in this study, the effects of additional of MK particles to the mixture can be isolate or control without any interference of chemical admixtures. However, if the water-to-cement ratio is adjusted to improve the water demand of MK such as incorporating of superplasticizer for better workability, it would likely to enhance the pozzolanic reaction and improve the compressive strength in the mixtures, as well as other physical and mechanical properties of cement paste.

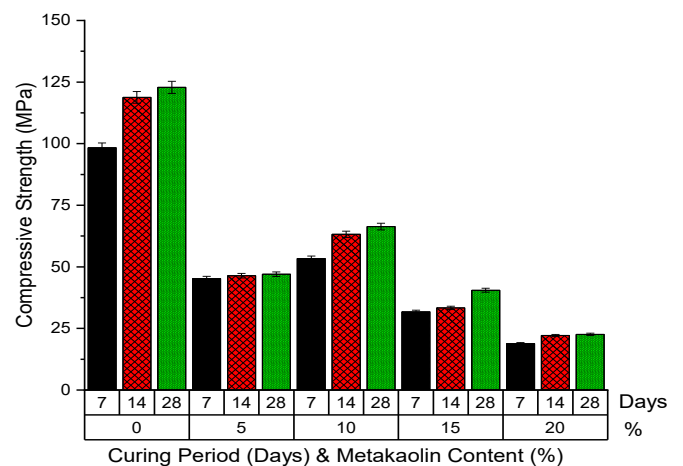


Fig. 4. Compressive Strength of Cement Paste Containing Non-Conforming Metakaolin at Various Levels and Curing Periods

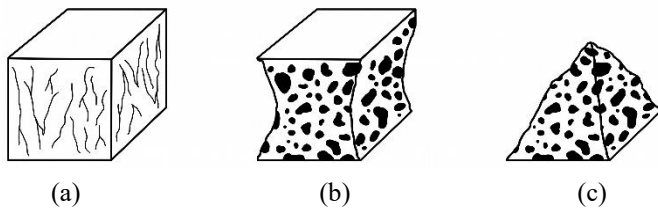


Fig. 5. Satisfactory Mode of Failure of Cubes: (a) Non-Explosive, (b) Semi-Explosive, and (c) Explosive [21]



Fig. 6. Failure Mode of Cement Paste Containing Non-Conforming Metakaolin at Various Levels and Curing Periods

3.4 Ultrasonic Pulse Velocity

Figure 7 shows the ultrasonic pulse velocity (UPV) of cement paste containing 0%, 5%, 10%, 15%, and 20% non-conforming metakaolin at curing periods of 7, 14, and 28 days. In all the cement paste samples, UPV increased with curing time. The 28-day curing period is considered the mature stage of hydration. As the samples reached maturity, the pozzolanic reaction, which involves metakaolin reacting with calcium hydroxide to produce C-S-H gel, enhanced hydration over time [22]. This reaction led to the formation of a dense layer, increasing the compactness in the concrete mixtures and, consequently, the UPV values.

As the metakaolin content increased, the UPV of the cement paste rose to a 15% replacement level before declining. The addition of metakaolin to the mixture reduced the number of voids by filling the pore space, a phenomenon known as the filler effect. The presence of more fine particles in the cement matrix occupied the pore space more effectively, leading to higher UPV values. Despite the optimal metakaolin content being 15%, the UPV of cement paste containing metakaolin remained lower than that of the control sample, ranging from 3.215 to 3.671 m/s, compared to 3.713 to 3.906 m/s, respectively.

Also worth mentioning, while the maximum compressive strength was found at 10% replacement, the UPV continued to rise at 15% replacement due to the filler effect of the metakaolin particles, which continues to pack the pores causing the UPV

value to increase as UPV measures the overall density and homogeneity of the cement paste matrix. On contrary, the compressive strength was subjected to the dilution effect after achieving the optimum replacement level of 10% during pozzolanic reaction, supported by high water demand of metakaolin particles, which leading to weaker structural bonds and reduce in strength despite denser matrix. This is because the compressive strength is sensitive to microcracking during pozzolanic reaction and effective binder content can affect the strength. Similar trend was observed by Chandak and Pawade [23] which found that compressive strength only increases up to optimum level while UPV value continues to increase due to the prolonged filler effect causing the formation of the homogeneous structure due to the addition of metakaolin.

Besides, utilizing of MK in this study contributes to improvement in the UPV values through two most important mechanisms in supplementary cementitious materials which are filler effect and pozzolanic reaction. The filler effect is a physical mechanism that reaches its peak efficiency when the particle size distribution and all empty spaces between voids are optimize through filling. This can be observed at 15% MK replacement levels whereby the amount of MK is fully utilized. In contrast, the chemical pozzolanic reaction is limited to the availability of the calcium hydroxide required for hydration and the reactivity of MK. In this case, the pozzolanic reaction can be observed to reach its peak at 10% MK replacement levels before dilution of cement became the dominant factor. This also can be verified by the observation of highest compressive strength at 10% MK replacement levels.

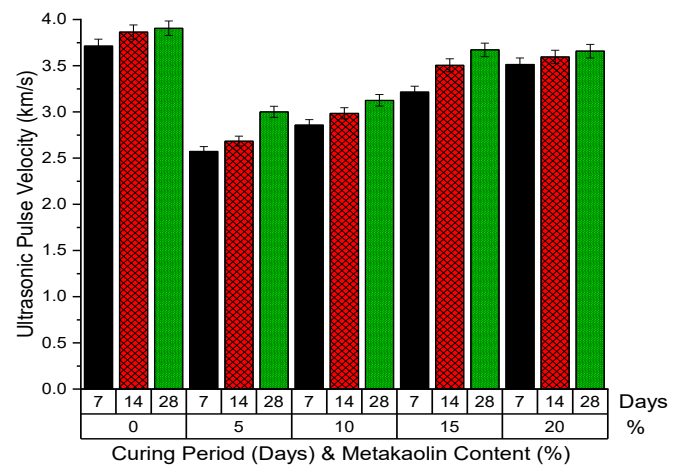


Fig. 7. Ultrasonic Pulse Velocity of Cement Paste Containing Non-Conforming Metakaolin at Various Levels and Curing Periods

3.5 Correlation Analysis

Table 2 shows the correlation coefficients between the variables of density, water absorption, compressive strength, and ultrasonic pulse velocity with non-conforming metakaolin at various replacement levels, which are determined using linear and non-linear regression models. The water absorption and ultrasonic pulse velocity of cement paste incorporating non-conforming metakaolin exhibited linear relationships with increasing replacement levels. In contrast, the density and compressive strength parameters were better described by non-

linear regression models under similar conditions. Among the evaluated properties, only water absorption has demonstrated a statistically significant correlation, with an R^2 value exceeding 0.75. This finding indicates that water absorption is significantly influenced by the incorporation of non-conforming metakaolin at varying replacement levels.

Table 2. Correlation Coefficient Between the Variables of Density, Water Absorption, Compressive Strength and Ultrasonic Pulse Velocity with Non-Conforming Metakaolin at Various Replacement Levels

Variables	Model	Equation	R^2
Density	Linear	$y = -12.947x + 1967.1$	0.6245
Water Absorption	Non-Linear	$y = -0.0136x^3 + 0.457x^2 - 3.3782x + 34.13$	0.8211*
Compressive Strength	Linear	$y = -3.9057x + 94.429$	0.7316
Ultrasonic Pulse Velocity	Non-Linear	$y = 0.0075x^2 - 0.1461x + 3.6549$	0.5761

*Note: This marked symbol indicates that the correlation is significant ($R^2 > 0.75$)

Meanwhile, Table 3 presents the Pearson correlation coefficients among the variables of density, water absorption, compressive strength, and ultrasonic pulse velocity, incorporating non-conforming metakaolin at various replacement levels. Water absorption of the cement paste showed a low to moderate correlation with density, compressive strength, and ultrasonic pulse velocity when incorporating non-conforming metakaolin at various replacement levels. Similarly, ultrasonic pulse velocity exhibited a moderate correlation with density, water absorption, and compressive strength under the same conditions. The compressive strength of the cement paste indicated a low to moderate correlation with both water absorption and ultrasonic pulse velocity when incorporating non-conforming metakaolin at different replacement levels. Among all variables, compressive strength was the only one to exhibit a strong correlation ($R^2 > 0.75$) with density, suggesting that compressive strength directly influences the density of cement paste containing non-conforming metakaolin.

Table 3. Pearson Correlation Coefficient Among the Variables of Density, Water Absorption, Compressive Strength, and Ultrasonic Pulse Velocity Incorporating Non-Conforming Metakaolin at Various Replacement Levels

Variables	Density	Water Absorption	Compressive Strength	Ultrasonic Pulse Velocity
Density		0.3886	0.8549*	0.5060
Water Absorption	0.3886		0.3530	0.5999
Compressive Strength	0.8549*	0.3530		0.6190
Ultrasonic Pulse Velocity	0.5060	0.5999	0.6190	

*Note: This marked symbol indicates that the correlation is significant ($R^2 > 0.75$)

Practically, this strong correlation between compressive strength and density shown the linear relationship between parameters which can be useful in identifying the compressive strength of concrete in the field work. Since the compressive strength cannot be directly measured without breaking up the

material, the non-destructive testing (NDT) method can first measure the density and compactness of material and later serve as accurate proxy for estimating the compressive strength through this NDT approach.

4. CONCLUSION

In conclusion, this study successfully evaluated the performance of non-conforming metakaolin as a SCM in cement paste. The incorporation of this low-carbon SCM can help reduce carbon dioxide emissions, thereby contributing to a lower-impact and more sustainable environment.

Based on the findings of this study, the following conclusions can be outlined:

(1) Although this SCM does not comply the requirements specified in standards such as ASTM C618, the non-conforming metakaolin still demonstrates satisfactory performance in cement paste, exhibiting strength and durability properties comparable to compliant SCMs.

(2) The density and compressive strength of cement paste incorporating non-conforming metakaolin were found to be optimal at a 10% replacement level, which is comparable to compliant metakaolin conforming to ASTM C618.

(3) The water absorption and UPV results indicate that non-conforming metakaolin achieved optimal performance at a 20% replacement level, suggesting that the durability properties of the cement paste improve with increased metakaolin substitution.

(4) In terms of the relationship, only the water absorption properties were significantly influenced by the increasing replacement level of non-conforming metakaolin in the cement paste. In addition, a strong relationship was observed between variables compressive strength and density as the proportion of non-conforming metakaolin increased. This strengthens the finding that incorporating non-conforming metakaolin can enhance certain strength and durability properties of cement paste.

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