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Research Article

Influence of Raw Kaolin Clay and Its Dehydroxylated Form on The Properties and Performance of Portland Cement Mortar

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Keywords

Kaolin clay,
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Strength development,
Porosity,
Water absorption,
Pore size distribution,
Sustainable binder.

Abstract

Kaolin, also known as China clay, is one of the materials that can be used as partial replacement for Portland cement but most of the research has been focused on its dehydroxylated form (metakaolin). The lack of interest in raw kaolin clay as a cement replacement material is partly due to its negative impact on strength and the traditional perception that raw clay is detrimental to concrete. However, the use of raw kaolin clay as cement replacement may offer other benefits, such as energy saving and the potential to produce durable cement-based material at low cost. Therefore, this paper presents findings on the influence of raw kaolin clay on the properties and durability performance of Portland cement mortar in comparison with metakaolin, when used as partial substitute. The results show that the use of raw kaolin clay as a partial substitute for Portland cement improved all aspects of the durability properties investigated, which became more apparent with age. Despite having the lowest compressive strength, the raw kaolin clay mix displayed a lower porosity, better resistance to water absorption and finer pores than the control. In contrast to the raw kaolin clay, the metakaolin significantly enhanced both strength and durability. The results also reveal that at a given superplasticizer dosage and replacement level, the kaolin and metakaolin mixes exhibited the same consistency in the fresh state and a similar range of pore size distribution and total intrusion volume at 28 days. The findings further demonstrate that raw kaolin clay can be used as Portland cement replacement material to produce durable mortar and concrete, particularly for applications that do not require high strength.

1. Introduction

The use of supplementary cementitious material (SCMs) as Portland cement replacement is a popular concept nowadays [1] and is one of the proposed strategies for mitigating the Portland cement industry's CO_2 emissions [2, 3]. Industrial wastes or by-products such as fly ash, blast furnace slag, and silica fume are among the most commonly used SCMs [4, 5]. However, these industrial wastes are not available in less industrialised countries [6, 7]. Thus, prompting the need for non-traditional SCMs in these regions, such as calcined clays, an attractive alternative for developing countries [8]. Among the calcined clays, metakaolin, which is the calcined or dehydroxylated form of kaolin clay has been widely studied [9–12]. Nonetheless, the cost associated with pure metakaolin as well as the competition from other industries often limit its applications in the concrete industry [13, 14].

Kaolin, also known as China clay, is one of the most abundant clay minerals [15] and has a wide range of industrial applications [16]. Unlike the calcined form, raw kaolin clay is not extensively utilized in Portland cement mortar and concrete. Research on the use of raw kaolin clay as cement replacement has attracted relatively little attention, probably due to the raw clay's strength-reduction effect reported by most studies [17]. However, most of the available data not only show a decrease in compressive strength but also an improvement in durability. Moukwa et al. [18] investigated the effects of illite and kaolinite clays on Portland cement paste and mortar, with each clay replacing 20% of the cement by mass. The authors reported that the introduction of clay reduces compressive strength while at the same time enhancing durability. It was observed that the clays increased the volume fraction of finer pores in the paste sample and reduced the total volume of all pore sizes in the mortal samples. Examining the pozzolanic activity of six types of clay minerals including kaolinite, He et al. [19] reported that the raw clay's structure greatly affected the compressive strength, and calcination is effective in enhancing the pozzolanic activity of clays. Studying the influence of various metakaolin replacement levels on the transport properties of Portland cement mortar, Courard et al. [20] included a single replacement level of raw kaolin clay to highlight the importance of calcination. The authors reported that the raw kaolin clay caused a decrease in the flexural and compressive strengths and had little to no influence on the resistance to water absorption and chloride diffusion. However, it should be noted that the metakaolin mixes also displayed higher water absorption than the control, which the authors attributed to improper mixing caused by the high-water demand of the mixes.

Despite the presence of raw clay in concrete even as microfine has been deemed detrimental to strength and durability, there is also well-documented evidence of positive impacts if the issues of high-water demand can be alleviated [21]. Raw clay as cement replacement does offer the potential of producing durable and eco-friendly cement-based materials at low cost while at the same time saving energy. Other than space-filling and serving as nucleation sites for the growth of hydration products, raw clays are generally considered nonreactive in Portland cement system. However, the possibility of pozzolanic behaviour has been suggested [22].

Since designers of concrete structures should now be more concerned with durability and sustainability than just strength [23], it is imperative to expand the concept of cement replacement to other materials such as raw clay and assess their viability in terms of cost, energy saving and durability enhancement. In this regard, this research seeks to investigate the influence of raw kaolin clay on the properties and durability performance of Portland cement mortar in comparison to its dehydroxylated form, metakaolin. Of interest to this investigation, are the workability of the fresh mix, the compressive strength, water absorption, porosity, and pore size distribution of the hardened mortar.

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2. Materials and Experimental Details

2.1. Materials

Ordinary Portland Cement 32.5N, commercial grade kaolin (China clay) and metakaolin were the binding materials employed in this investigation. The Metakaolin was obtained by calcining the kaolin clay in a laboratory furnace at 800˚ C. The chemical composition and physical properties of the Portland cement, kaolin clay and metakaolin are presented in Table 1. The fine aggregates were of natural quarry sand and had a maximum particle size of 5 mm.

Table 1. Chemical composition and physical properties of the binders

Parameters	OPC	Kaolin	Metakaolin
SiO_2 (%)	20.40	46.90	51.52
Al_2O_3 (%)	4.90	38.50	40.13
Fe_2O_3 (%)	3.10	0.76	1.23
CaO (%)	63.10	0.95	2.00
MgO (%)	3.10	0.09	0.12
K_2O (%)	1.05	0.26	0.53
SO_3 (%)	2.30	0.00	0.00
TiO_2 (%)	0.00	1.32	2.27
Na_2O (%)	0.25	0.04	0.09
LOI (%)	1.35	12.5	2.10
Specific gravity	3.12	2.6	2.6
Surface Area (m^2/g)	1.12	29	12

2.2. Experimental Details

2.2.1. Mix Design

Five mixes were considered in this investigation and details of the mix designs are presented in Table 2. The control mix consisted of 100% Ordinary Portland cement, whereas the other four mixes were binary, with either the raw kaolin clay or metakaolin as the replacing material. As shown in the mix design table, the total binder content, water-binder ratio, and aggregate content were held constant. In this study, only one replacement level of the kaolin clay was investigated while for the metakaolin, three replacement levels were considered to examine the effects of varying the metakaolin content. A naphthalene-based superplasticizer was added to the binary mixes to improve consistency. The workability of the fresh mixes was assessed by the flow table test according to BS 4551, Part 1 [24].

Table 2. Mix Designs

	Mix Designs				
Materials (kg/m^3)	OPC	K15	MK5	MK15	MK30
Portland Cement	360	306	342	306	252
Kaolin	-	54	-	-	-
Metakaolin	-	-	18	54	108
Fine Aggregate	740	740	740	740	740
Water Content	180	180	180	180	180
Superplasticiser*	-	1.2	0.8	1.2	2.2
Spread Flow (mm)	280	300	310	305	307

* percentage by mass of the total binder

2.2.2. Specimen Description and Preparation

The mortar specimens were cylindrical in shape, with a height of 25 mm and a diameter of 100 mm. The mixing was done in a rotary mixer. The aggregates and binder(s) were first mixed for 2 minutes before adding water and the mixing then continued for another 3 minutes. The specimens were cast in PVC moulds and properly vibrated. All specimens were demoulded 24 hours after casting and cured in water at a temperature of 20 ± 3 ˚C. After specific periods of curing, the mortar specimens were oven-dried for 48 hours at 105 ± 5 ˚C and then allowed to cool within a desiccator to a temperature of 20 ± 3 ˚C before being tested for porosity, water absorption and pore size distribution.

2.2.3. Compressive strength

The testing of the compressive strength was done on concrete specimens of 100 mm cubes according to the procedure indicated in BS 1881, Part 116 [25]. The mix design parameters were the same as the mortar, with the exception that the coarse aggregates were added at 1.5 times the fine aggregate content and the superplasticizer dosages were also modified to suit the consistency of the mortar.

2.2.4. Porosity

The porosity was investigated by the vacuum saturation method, which has been proven to be one of the most efficient methods of evaluating porosity in concrete [26]. Figure 1 shows the setup of the vacuum saturation apparatus. The setup consisted of an evacuation chamber, a vacuum pump, and a mass balance. The specimens were placed in the evacuation chamber, which was then sealed and evacuated to a pressure of 20 millibars and maintained for at least 3 hours. After the 3-hour period, distilled water was slowly introduced into the chamber to slightly cover the specimens and the pressure was re-established for another three hours. The specimens were then removed from the evacuation chamber and weighed in air using a conventional balance and under water using an Archimedes balance. After that, the specimens were dried in an oven at 105 ± 5 ˚C for about 48 hours to a constant weight determined in air with a conventional balance. The total porosity of the samples was then calculated using Equation 1.

$$\text{Porosity} = \frac{M_3-M_1}{M_3-M_2} \times 100 \quad (1)$$

Where, M_1 – mass of the dried sample weighed in air, M_2 – mass of saturated sample weighed in water and M_3 – mass of saturated sample weighed in air.

Figure 1. Vacuum Saturation Apparatus [27]

2.2.5. Water Absorption

Water absorption of the specimens was examined by the initial surface absorption test (ISAT) and was carried out as prescribed by BS 1881, Part 5 [28]. The setup consisted of a clamp for holding the specimen, a reservoir, a cap, capillary tubes, and a capillary scale. The specimen was placed in the clamp set along with the cap in position and tightened. The capillary tubes were then connected from the reservoir to the cap's inlet and from the cap's outlet to the capillary scale. The reservoir was set up to have a water head of 200 ± 20 mm above the specimen's surface. The reservoir was then filled with water, and the inlet valve of the cap opened to allow water to flow into the cap. To maintain the water head of 200 ± 20 mm, the reservoir was regularly refilled. Trapped air was removed by allowing the water to overflow through the capillary scale. After that, the valve was then closed, and the stopwatch started when the flow on the capillary scale reached zero, and the reading was recorded after 10 minutes.

2.2.6. Pore Size Distribution

The pore size distribution of the specimens was determined using the Mercury Intrusion Porosimetry (MIP) technique. Mercury Porosimetry is based on capillary law governing liquid penetration into small pores. In the case of a non-wetting liquid such as mercury and based on the assumption of cylindrical pores, this law is presented in Equation 2.

$$D = \left(\frac{1}{P}\right) 4\gamma\cos\theta \quad (2)$$

Where D is pore diameter, P the applied pressure, γ the surface tension, and θ the contact angle. AutoPore II 9220, a mercury porosimeter capable of measuring pore diameters ranging from 360 to 0.003 µm at pressure up to 414 MPa (60 000 psi), was used in this investigation. This unit has four built-in low-pressure ports and two high-pressure chambers. All aspects of low-pressure and high-pressure analyses, as well as data collection, reduction, and display, were processed by the control module. In the absence of specific test data, the surface tension and contact angle were considered 485 dynes/cm and 130 degrees, respectively, as recommended by the AutoPore operator manual.

3. Results and Discussion

3.1. Consistency

The consistency of the mortar mixes was assessed by the spread flow diameter and the results are presented in Table 1. The results show that the use of either kaolin clay or metakaolin as cement replacement material decreased the workability of the mortar, thus, requiring a superplasticizer to match the consistency of the control mix. At a given superplasticizer dosage and replacement level, the kaolin clay mix seems to have a similar consistency as the metakaolin. Mix K15 and the MK15 mix displayed spread flow diameters of 300 and 305 mm, respectively, which were 7.14 and 8.93% greater than the control. It was also observed that as the metakaolin content increased, a higher dosage of superplasticizer was required to maintain consistency as the control.

3.2. Compressive Strength

Results of the compressive strength are presented in Figure 2. The results show that the addition of raw kaolin clay does cause a decrease in the compressive strength at the age tested, which is consistent with previous studies [17–19, 29]. The strength reduction effect has been attributed to the raw clay's tendency to coat the cement particles as well as absorb most of the mixing water [30, 31]. Thereby, causing an increase in water demand and hampering the hydration process. Despite having the lowest strength, the 28th-day compressive strength of the raw kaolin clay mixture was above the minimum strength specified by most standards for structural concrete. For the mixes containing metakaolin, an increase in compressive strength is observed at all ages tested, with the compressive strength increasing with higher replacement levels. At 28 days, samples containing 5, 15 and 30% metakaolin displayed 17, 39, and 63% increase in strength, respectively, than the OPC mix. This improvement in strength is due to the metakaolin's filler and acceleration effects, as well as its pozzolanic reactivity [32]. During the hydration of Portland cement and in the presence of a pozzolan such as metakaolin, the silica and alumina from the metakaolin react with calcium hydroxide to produce additional spacing-filling hydrates, which contribute to strength development and microstructural enhancement [33, 34].

3.3. Porosity

Figure 3 shows the results of the vacuum saturation porosity test. All binary mixes including the mix containing kaolin clay displayed lower porosity than the control. However, there is a noticeable difference between the porosities of the mixes containing metakaolin compared to the mix OPC and K15. Despite mix K15 displayed lower porosity than the control, which can be accredited to the filler effect of the raw clay, the impact of kaolin clay on porosity reduction appears to be less effective than the metakaolin when compared to mix MK15 or even the mix containing 5% metakaolin after 28 days of curing. The rate of porosity reduction in the metakaolin mixes is faster than in the kaolin clay mix, and this is due to the metakaolin's pozzolanic reactivity and the acceleration effect. The metakaolin mixes showed the lowest porosity, reducing with increasing replacement level, and showing a similar trend as the compressive strength. Samples containing 30% metakaolin obtained the lowest porosity at all ages tested. As a highly reactive pozzolan, metakaolin is very effective at reducing porosity in Portland cement mortar and concrete, especially when used with a superplasticizer. It is also important to note that for all mixes, including mix K15, the porosity reduces with time. Therefore, the raw kaolin clay's contribution to porosity reduction cannot be ignored.

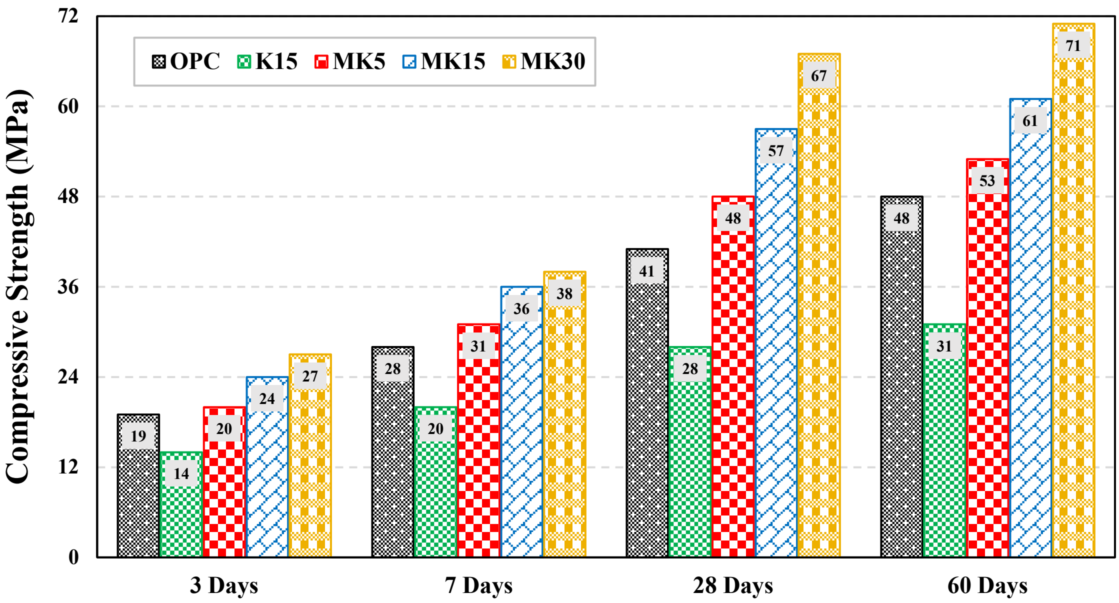

Figure 2. Compressive Strength of the Mixes

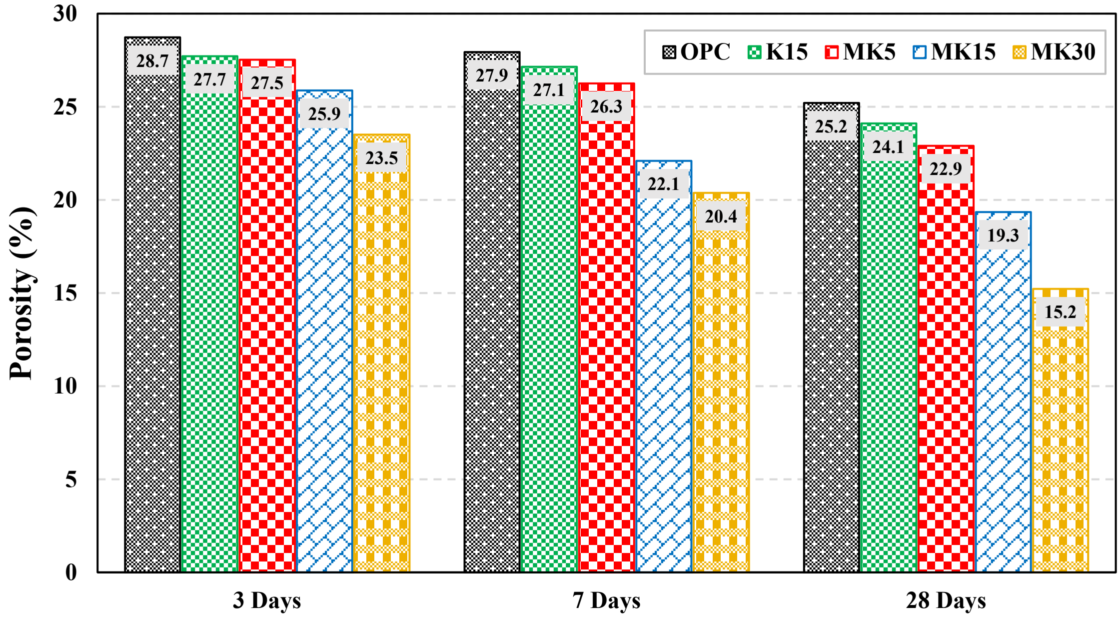

Figure 3. Porosity of the Mixes

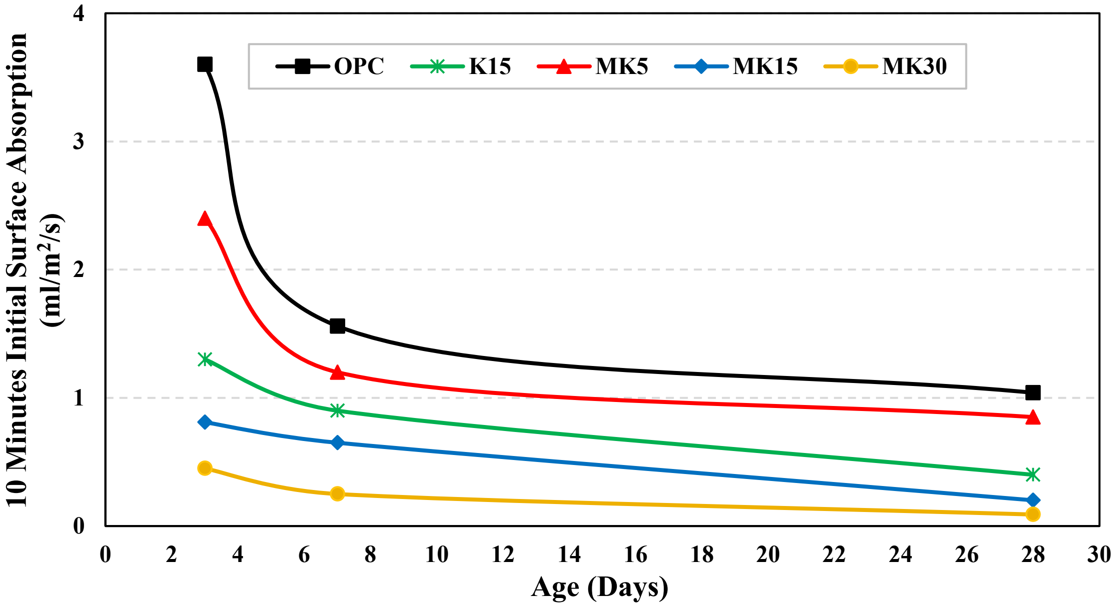

Figure 4. Initial Surface Absorption of the Mixes

3.4. Initial Surface Absorption Test

Results of the 10 minutes initial surface absorption test are presented in Figure 4. The results show a decrease in water absorption for all mixes with time. Interestingly, the mix containing raw kaolin clay absorbed water at a lower rate than the control and mix MK5. As early as 3 days after casting, the 10 minutes initial surface absorption value of mix K15 was 63.9 and 45.8% lower than that of the control and mix MK5, respectively. Since verifying the possibility of pozzolanic reactivity of the raw kaolin clay was not part of this study, the performance of mix K15 can be ascribed to the relatively high surface area of kaolin clay, which enhanced the filler effect contribution. The results also indicate that samples of MK15 and MK30 absorbed water at significantly lower rates compared to the other mixes, with the 30% replacement level obtaining the lowest water absorption. Therefore, an increase in the replacement level of metakaolin resulted in better resistance to water absorption. Again, the performance of the metakaolin mixes against water absorption demonstrates the pozzolanic reactivity and the filler effect of the metakaolin. These contributions improve the penetration resistance of Portland cement mortar and concrete by the generation of additional hydrates and the pore-blocking influence, thereby, reducing the total porosity and causing pore structure refinement.

3.5. Mercury Intrusion Porosimetry (MIP)

The pore size distribution results are presented in Figure 5, 6, and 7 for 3, 7, and 28 days, respectively. For all mixes, the results show that the cumulative pore volume (total intruded volume) and the threshold pore entry diameter decrease with curing time, which can be attributed to the continuation of hydration and pozzolanic reaction. All binary mixes exhibited a larger volume of finer pores compared to the control mix. The mix containing raw kaolin clay displayed the highest total intruded volume at an early age (3 and 7 days) but also, the fewest pores larger than 1 μm. At 28 days, the raw kaolin clay mix (K15) attained a lower total intruded volume and fewer pores larger than 1 μm, compared to the OPC, MK5 and MK15 mixes. However, the kaolin clay mix also displayed a larger volume of pores within the range of 0.01 – 1 μm than MK15 at 28 days. Mix MK30 also shows a higher total intruded volume at 3 days than the OPC and the other mixes containing lower replacement levels of metakaolin. Notwithstanding, MK30 obtained the lowest cumulative intruded pore volume from the 7th day onward and the smallest threshold pore entry diameter on the 28th day. The findings also show that as the metakaolin content increases, the total intruded volume and threshold pore entry diameter decrease. The MIP results reveal that the use of raw kaolin clay as cement replacement material also reduces the among of pores that are detrimental to concrete's durability.

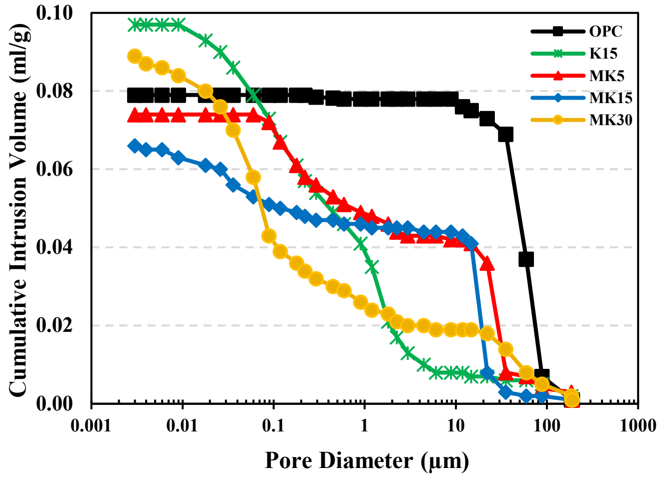

Figure 5. Pore Size Distribution of the Mixes at 3 Days

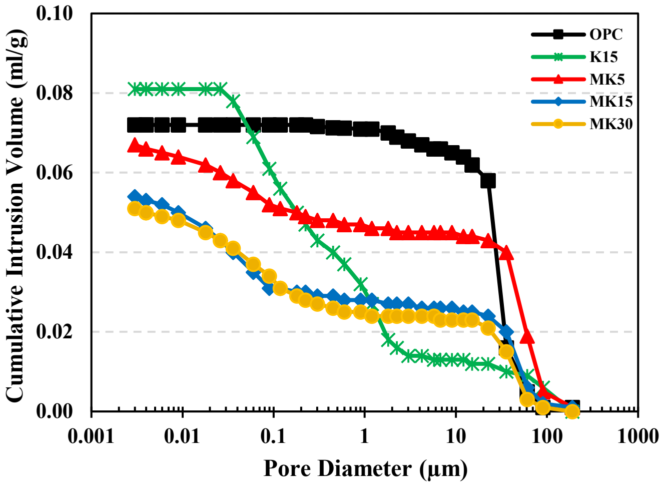

Figure 6. Pore Size Distribution of the Mixes at 7 Days

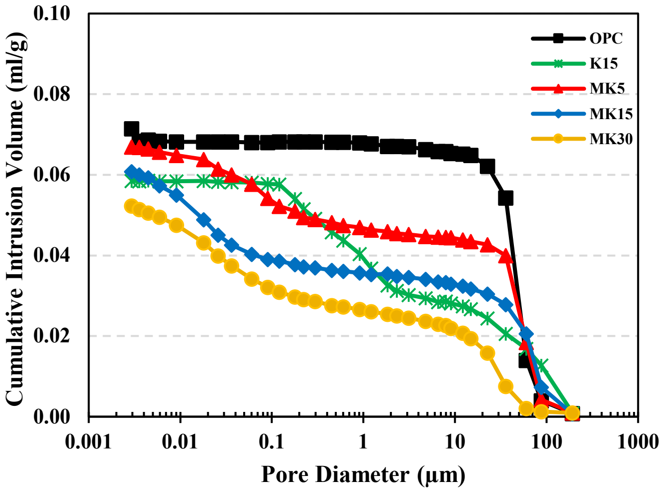

Figure 7. Pore Size Distribution of the Mixes at 28 Days

4. Conclusions

This study presents findings on the influence of raw kaolin clay and its dehydroxylated form, metakaolin, on the properties and durability performance of Portland cement mortar, when used as partial substitute. Within the scope of this experiment, the following conclusions can be drawn:

- The use of raw kaolin clay as cement replacement material does cause a decrease in compressive strength. However, the 28th-day compressive strength of the raw kaolin clay mix was above the minimum value specified by most standards for structural concrete.
- All binary mixes including the mix containing raw kaolin clay exhibited better durability performance than the control mix.
- In comparison to the control, the kaolin clay mixture displayed a lower porosity, better resistance to water absorption and fewer pores larger than 1 µm from an early age, as well as a lower total intruded pore volume at 28 days.
- The impact of the raw kaolin clay on porosity reduction appears to be less effective than the metakaolin.
- The metakaolin remarkably contributed to both strength development and durability enhancement, which improved with increasing replacement level and the highest replacement level considered in this study achieved the best performance.
- Although the influence of the raw kaolin clay on the porosity was not as profound as the metakaolin at the same replacement level, there are also noticeable contributions on other durability parameters such as water absorption and pore size distribution. Hence, can be used to produce durable cement-based material, particularly for structures that do not require high strength.

Declaration of Conflict of Interests

The authors declare that there is no conflict of interest. They have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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