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Effect of pore modifiers on physical and mechanical properties of high-performance cement mortar

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Abstract

Two different pore modifiers (PM), artificial pozzolan (AP) and modified aluminum salt (MA), were introduced into the composition of a high-performance cement mortar (HPCM). The chemical compositions of raw materials, ordinary Portland cement (OPC), sea sand, AP, and MA were identified by X-Ray Fluorescence spectrometry (XRF). The hydration kinetics resulting from each PM added to the HPCM was investigated by the relevance of hydration temperature. The mechanical properties such as compressive strength, dynamic modulus of rupture, and dynamic modulus of elasticity were measured as well as water absorption and density. Additionally, Scanning Electron Microscope (SEM) and Brunauer-Emmett-Teller (BET) were used to reveal the beneficial effects of appropriate PM through the microstructure, pore size distribution and specific surface area. Experimental results showed that the PM increased the hydration temperature, resulting in the generation of stress at early stages throughout the HPCM structure. This stress caused the formation of micropores, which increased water absorption, decreased density, and enhanced the structural integrity

1. Introduction

Global construction development will accelerate by 35% over the next decades, fueled by historic amounts of stimulus expenditure on infrastructure and the release of excess family savings, according to a new analysis [1]. This is a positive indicator for the construction industry. Globally, building and construction operations utilize 3 billion tons of raw materials each year, accounting for 40% of total global consumption. Moreover, 6 million tons of energy are consumed and 23 million tons of carbon dioxide (CO_2) are released during the manufacturing and delivery of these construction materials. Concrete is the primary material used in buildings, which have a high embodied energy [2-4].

According to the Paris Agreement and the 26th United Nation Climate Change Conference (COP26), the aim is to achieve net zero emissions and climate resilience by 2050 [5]. The building industry has historically been unable to achieve this goal. Therefore, a major aim should be to reduce waste and CO_2 emissions in order to support the goal in the building industry. This may be achieved by utilizing high-performance products. For example, concrete is one of the most frequently utilized building materials, so high-performance concrete (HPC) is critical to contributing to this goal; it provides higher durability, enhanced mechanical properties, and a longer lifespan than regular concrete [6]. In every form of concrete, the first critical component prior to production is the cement-sand mixture or mortar, also used to adhere construction materials together such as brick or stone [7], enhancements to the mortar properties can be termed high-performance cement mortar (HPCM), a component to HPC.

Typically, in addition to aggregate, HPC has the following components: cement, water, fine sand, superplasticizer, fly ash, and silica fume [8]. Furthermore, ultra-high performance concrete (UHPC) is achieved by a densified structure, lower porosity by lowering the water to cement ratio, using a superplasticizer, and a reduction in aggregate size [9]. Silica fume is introduced in many studies because of the results of the nanometer particle size, high reactivity with cement and ability to improve the interfacial transition zone between aggregates and paste [8,9]. However, silica fume affects concrete in the terms of higher viscosity, water content, and cost [10]. Additionally, it increases the autogenous shrinkage of the cement slurry and the percentage of inclusion exceeds 5%, therefore increasing the danger of cracking. Since these materials bear loads, in this case, it is quite easy to develop cracks in mortar and concrete, necessitating concrete repair [10].

Importantly, concrete and mortar properties are also enhanced by silica fume because of its ability to modify porosity within concrete structures since it has small sizes, high reactivity and shows a reduction in the porosity [9,11]. This is critical development in the term of the concrete ability to produce high and ultra-high performance concrete based on a reduction in porosity [9]. Furthermore, nano size

of a high silica (SiO_2) content more than 90% of silica fume [11-13] promotes the calcium silicate hydrate (C-S-H) during the hydration process of cement [14] which is considered a pozzolan material, a critical component for enhancement of the mechanical properties of HPCM.

The Circular Economy (CE) concepts, evolved from industrial ecology, aims to bring together pre-existing concepts from various scientific fields that share common qualities and characteristics, such as industrial ecosystems and industrial symbioses, the 3Rs principle (reduce, reuse, recycle), cleaner production, circular material flows in manufacturing systems, product-service systems, eco-efficiency, cradle-to-cradle design and green growth [15]. The Ellen MacArthur Foundation (EMF) defines CE as "Restorative by design and aims to keep products, components and materials at their highest utility and value at all times, distinguishing between technical and biological cycles" [16].

According to CE and the silica fume concept, the agro-waste ash has a significant amount of silica (SiO_2) and alumina (Al_2O_3) content and has been investigated as alternative cementitious materials in concrete [17]. In addition, the dangerous by-product from aluminum industry as aluminum dross also has high percentage of alumina (Al_2O_3) [18]. Moreover, their mentioned chemical composition was considered as pozzolan materials in concrete [19].

Stefanoviæ *et al* [20] reported that calcium silicate hydrate (C-S-H), calcium hydroxide ($Ca(OH)_2$), and calcium aluminate hydrate (C-A-H) are the results of the hydration process of cement, as stated in equations (1-3).

$$2(3CaO{\cdot}SiO_2) + 6H_2O \rightarrow 3CaO{\cdot}2SiO_2{\cdot}3H_2O\ (C\text{-}S\text{-}H) + 3Ca(OH)_2 \quad (1)$$

$$2(3CaO{\cdot}SiO_2) + 4H_2O \rightarrow 3CaO{\cdot}2SiO_2{\cdot}3H_2O\ (C\text{-}S\text{-}H) + Ca(OH)_2 \quad (2)$$

$$Ca(OH)_2 + SiO_2 + H_2O \rightarrow CaO{\cdot}SiO_2{\cdot}2H_2O\ (C\text{-}S\text{-}H) \quad (3)$$

Being a major chemical reaction of pozzolan materials, a hydration reaction also occurs between calcium hydroxide ($Ca(OH)_2$), a by-product from former reaction, and silica and alumina as shown in equations (4-5) [14].

$$xCa(OH)_2 + ySiO_2 + zH_2O \rightarrow xCaO{\cdot}ySiO_2{\cdot}zH_2O \quad (4)$$

$$xCa(OH)_2 + yAl_2O_3 + zH_2O \rightarrow xCaO{\cdot}yAl_2O_3{\cdot}zH_2O \quad (5)$$

To promote the global trends, state of art materials development for building products or most notably cement-related products is inspired. The following critical point is to produce pozzolan materials for concrete from agro-waste ash and aluminum dross due to their high silica (SiO_2), in the lower amount than silica fume, and alumina (Al_2O_3) content, which will be advanced through calcination and chemical treatment.

Consequently, the objectives of this work are to promote climate goals by considering alternative materials that mitigate the influence of silica fume while retaining the features of a HPCM. New pore modifiers, artificial pozzolan (AP) and modified aluminum salt (MA), are introduced to investigate their effects on mechanical and physical properties in HPCM. The characterization of raw materials, investigation of the influence of pore modifiers on hydration temperature, as well as mechanical properties were observed. Furthermore, Scanning Electron Microscope (SEM) and Brunauer-Emmett-Teller (BET) were obtained to demonstrate the beneficial effects of PM on the microstructure, pore size distribution, and specific surface area of HPCM in order to explain its physical and mechanical properties.

2. Experimental

2.1 Materials

The raw materials used in this study were ordinary Portland cement (OPC) according to ASTM Type I as cementitious materials, sea sand as fine aggregates, and pore modifiers (PM) introduced with artificial pozzolan (AP) from the calcination of agro-waste ash and modified aluminum salt (MA) from the chemical treatment of aluminum dross provided by Shera public company limited.

2.2 Mix design

The mix design was based on ASTM C595/C595M at the water-to-cement ratio (W/C) of 0.45 and was divided into two parts: for hydration temperature testing with various proportions of each PM ranging from 1 wt% to 4 wt% of OPC content as shown in Table 1 and for HPCM proportion with the optimal percentage of PM.

2.3 Casting

Samples for the main testing, i.e., compressive strength, modulus of rupture, and modulus of elasticity, are cast and compacted by tamping of two layers in cube molds (5 cm × 5 cm × 5 cm) and prism molds (4 cm × 16 cm × 16 cm), held for 24 h, then demolding with testing after 1, 7, 14 and 28 curing days.

Table 1. Hydration temperature tests proportion at different PM addition.

Formular	OPC (wt%)	Sea Sand (wt%)	AP (wt%) of OPC content	MA (wt%) of OPC content	W/C
REF	75	25	-	-	0.45
RAP-1	75	25	1	-	0.45
RAP-2	75	25	2	-	0.45
RAP-3	75	25	3	-	0.45
RAP-4	75	25	4	-	0.45
RMA-1	75	25	-	1	0.45
RMA-2	75	25	-	2	0.45
RMA-3	75	25	-	3	0.45
RMA-4	75	25	-	4	0.45

2.4 Curing

The curing days were at 1, 7, 14 and 28 days using standard room temperature curing, which is the most prevalent, cost-effective, and ecologically friendly method used in practice [9]. The specimens must be stored in the box and left at room temperature nominally between 10°C to 32°C [21].

2.5 Testing and analysis

The chemical compositions of the raw materials (OPC, sea sand, AP and MA) were determined using X-Ray Fluorescence spectroscopy (XRF) (Panalytical, Netherlands). The proportion of each PM on the HPCM in Table 1 was prepared as a slurry in the non-air chamber and examined the heat of hydration kinetics using the heat of hydration recorder by a thermal recorder (Compact thermal logger, AM-8000K, type K: -200°C ~ 1370°C) in every 30 sec until the hydration temperature of slurry reached a constant value. After that, the specimens with AP and MA from the optimal hydration temperature results were investigated for preparing HPCM specimens for mechanical property tests. The compressive strength test was evaluated in accordance with ASTM C91/C91M using a compression machine (CST, Thailand). The modulus of rupture and elasticity were determined in accordance with ASTM C109 using a universal testing machine (Toni Technikwas, Germany). After mechanical testing, specimens were used for water absorption and density tests in accordance with ASTM C90-05.

Scanning Electron Microscopy (SEM) was preformed to study the effects of the addition of AP and MA on the microstructure, while Brunauer–Emmett–Teller (BET) nitrogen adsorption–desorption experiments at 70K were used to determine the pore size distribution and specific surface area of the HPCM.

3. Results and discussion

3.1 Chemical composition

The chemical composition of OPC, sea sand, AP, and MA, is shown in Table 2. It was discovered that the chemical composition of the major raw materials adheres to the criteria for cement and sea sand. As a consequence, AP provided the percentage of SiO_2 and Al_2O_3 due to SiO_2 and Al_2O_3 content in chemical compositions of agro-waste ash [22]. The processing of calcination could remove the organic compositions from AP. Also, MA provided a high percentage of Al_2O_3 due to Al_2O_3 content in chemical compositions of aluminum dross [18]. Moreover, MA presented chemical composition of Na_2O in considerable amount. Furthermore, Table 2 also indicated that AP and MA could be classified as pozzolan materials [19] due to the composition of the significant amount of SiO_2 and Al_2O_3. In addition, the comparison of the percentage of SiO_2 in the composition of AP and MA to silica fume, which is more than 90% [23,24], demonstrated that the SiO_2 content in pozzolan materials has decreased.

3.2 Hydration temperature

The influence of AP (RAP 1-4) concentrations ranging from 1 wt% to 4 wt% on the hydration temperature of cement are illustrated in Figure 1(a). When compared to a control sample (REF), AP enhanced the hydration temperature of cement. The highest rate of heat of hydration temperature of REF was 59°C in 6 h 28 min, whereas 1 wt% to 4 wt% of AP addition resulted in increasing the temperature to 59.2°C in 6 h 15 min, 62.7°C in 6 h 15 min, 61.1°C in 5 h 58 min, and 60.1°C in 6 h 32 min, respectively.

The influence of MA (RMA 1-4) concentrations ranging from 1 wt% to 4 wt% on the hydration temperature of cement is demonstrated in Figure 1(b). The MA also contributed to the acceleration of the heat of hydration temperature of OPC. REF reached its maximum heat of hydration temperature of 59°C in 6 h 28 min, while the MA from 1 wt% to 4 wt% increased the temperature to 60°C in 6 h 32 min, 62.1°C in 6 h 15 min, 60.9°C in 6 h 19 min, and 60.5°C in 6 h 54 min, respectively.

As a consequence of the hydration temperature results, both the AP and MA had a notable influence on an increase in the hydration temperature at a 2% addition, increasing hydration temperature rate up to 6.27% and 5.25%, respectively. As a result, they represent the optimal hydration temperature rate used in the manufacturing of HPCM in this study. Therefore, Table 3 represents the HPCM prototyped proportion at the addition AP and MA at 2 wt% of OPC content relative to the control sample.

As the results of chemical compositions of the PM, introduced with AP and MA, in the Table 2, pozzolan materials could react with the $Ca(OH)_2$ which is a by-product during the cement hydration to generate the hydration products as C-S-H and C-A-H [14] as equation (4) and (5). The reactions induced the graph to elevate and shift to the left, indicating an increase in cement setting and hardening [25].

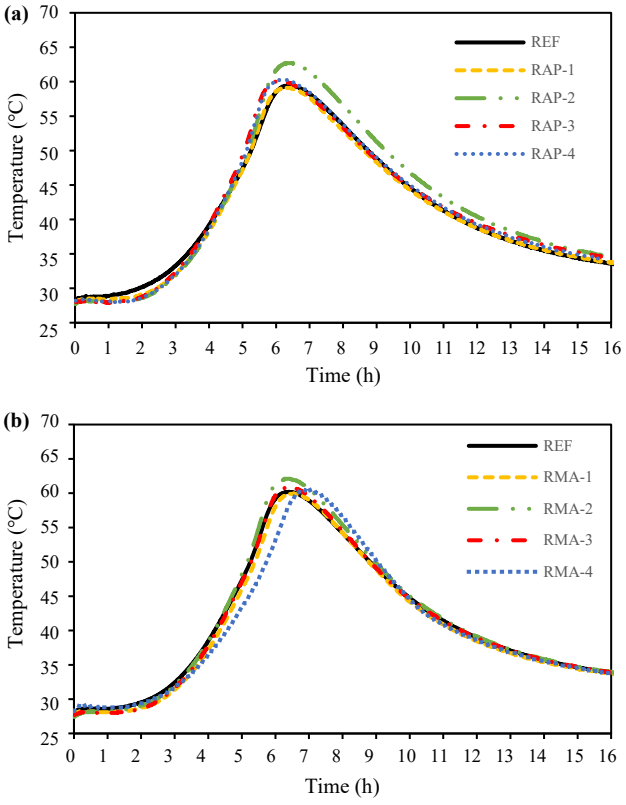

Figure 1. Hydration temperature after adding each PM separately; (a) Adding AP from 1wt% to 4 wt% of OPC and (b) Adding MA from 1 wt% to 4 wt% of OPC.

Table 2. Chemical and mineralogical composition of cement, sand, AP, and MA by XRF.

Compound	Sample				
	OPC	**Sea Sand**	**AP**	**MA**	**Unit (wt%)**
SiO_2	19.80	98.06	51.45	6.77	%
Al_2O_3	5.98	1.10	46.61	88.35	%
Na_2O	0.00	0.01	0.09	3.13	%
K_2O	0.34	0.27	0.07	0.00	%
CaO	64.45	0.24	0.17	0.66	%
Fe_2O_3	3.18	0.26	0.42	0.03	%
MgO	1.21	0.01	0.00	0.00	%
SO_3	2.73	0.00	0.03	1.03	%
Others	2.31	0.05	1.17	0.04	%

Table 3. HPCM proportion for prototyping of control sample (REF) and, at the addition of 2% PM determined for the optimal hydration temperature.

Formular	OPC (wt%)	Sea Sand (wt%)	AP (wt%) of OPC content	MA (wt%) of OPC content	W/C
REF	75	25	-	-	0.45
RAP-2	75	25	2	-	0.45
RMA-2	75	25	-	2	0.45

3.3 Mechanical and physical properties

The compressive strength, MOR, and MOE of HPCM at 1, 7, 14 and 28 days with 2 wt% of both PM and MA addition at the optimal hydration temperature specified, are given in Figure 2(a-c), respectively. When compared to the control sample (REF), the addition of AP and MA had a significant effect on the early mechanical properties of HPCM. For the 1-day test, the compressive strength, MOR, and MOE increased by 46.28%, 18.68%, and 12.71%, for the AP (RAP-2) and also for the MA (RMA-2) 14.24%, 5.74%, and 7.18%, respectively.

The effects of AP and MA at 2 wt% of OPC addition, RAP-2 and RMA-2, on HPCM's water absorption and density at 1, 7, 14 and 28 days, is shown in Figure 3(a-b), respectively. The specimens introduced with the AP and MA had a considerable change on modifying the typical properties of HPCM by increasing water absorption and decreasing density, compared to the control sample (REF) at 1-day test, when other days did not. At the 1-day test, RAP-2 increased water absorption by 4.55% and decreased density by 3.15%, whereas RMA-2 raised water absorption by 2.33% and decreased density by 2.38%.

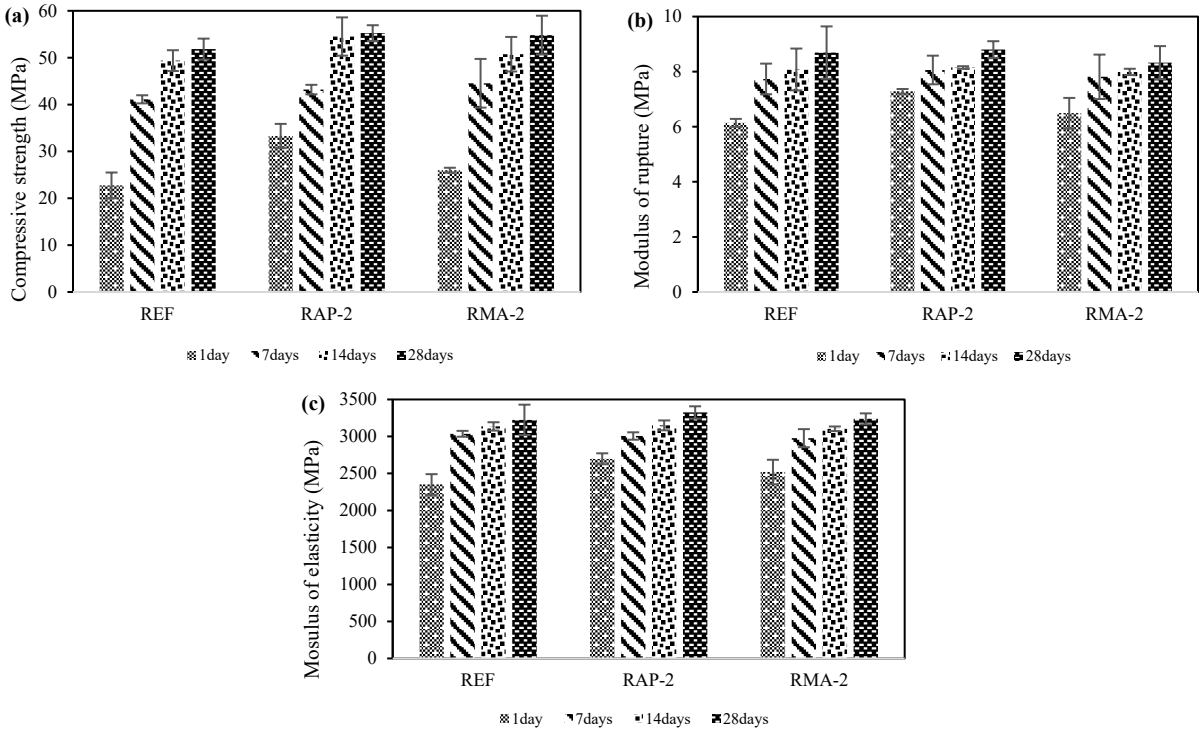

Figure 2. Mechanical properties of HPCM at 2% addition of each PM for 1, 7, 14 and 28 days; (a) Compressive strength, (b) Modulus of rupture (MOR), and (c) Modulus of elasticity (MOE).

Figures of the interfacial transition zone (ITZ) of HPCM at 1 day at room temperature, where the boundary between aggregate and cement develops following the addition of PM with MA (RMA-2), and AP (RAP-2), are provided in Figure 4(a-c), respectively. The control sample (REF) exhibited larger porosity between ITZ, then ITZ was developed by the MA and AP additions, respectively. The HPCM cement paste for 1 day at room temperature are shown in Figure 4(d-f). The hydration product in the control sample (REF) showed non-crystalline with a gel-like structure, but when MA (RMA-2) and AP (RAP-2) were introduced, the hydration product developed to semi-crystalline and crystalline, respectively, and also showed on the shape of needles.

The values of specific surface area obtained by BET using nitrogen adsorption of the HPCM at 1 day test are given in Table 4. It was found that, RAP-2 had a greater specific surface area than those of the RMA-2 and REF as a result of the increased micropore volume in the HPCM structure.

The pore size distribution is shown in Figure 5 obtained by BET using nitrogen adsorption of HPCM at 1 day test. The results revealed that after adding AP (RAP-2) and MA (RMA-2) they increased the volume of the micropores (diameter < 64 nm). When compared to that of the control sample (REF), RAP-2 generated a greater volume of micropores, followed by, RMA-2, and REF, respectively. This also revealed all had a similar volume of pores for a diameter larger than 64 nm.

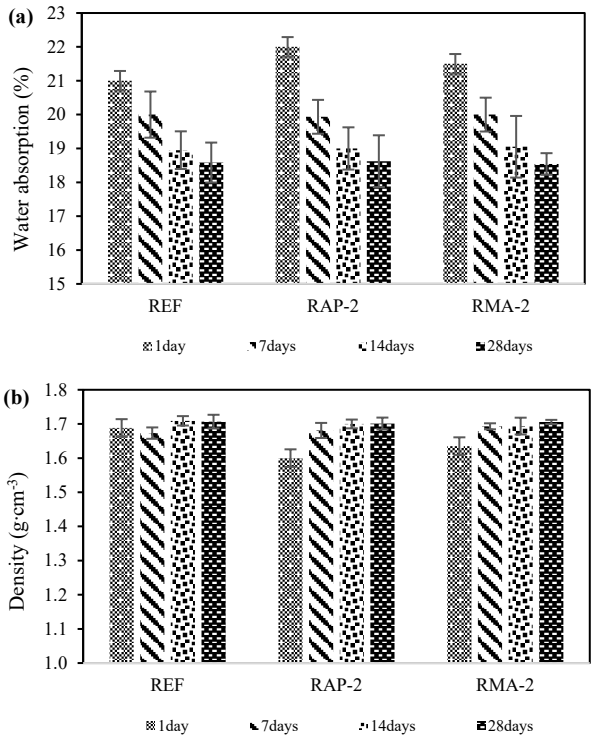

Figure 3. Typical properties of HPCM at 2% addition of each PM for 1, 7, 14 and 28 days; (a) water absorption rate and (b) density.

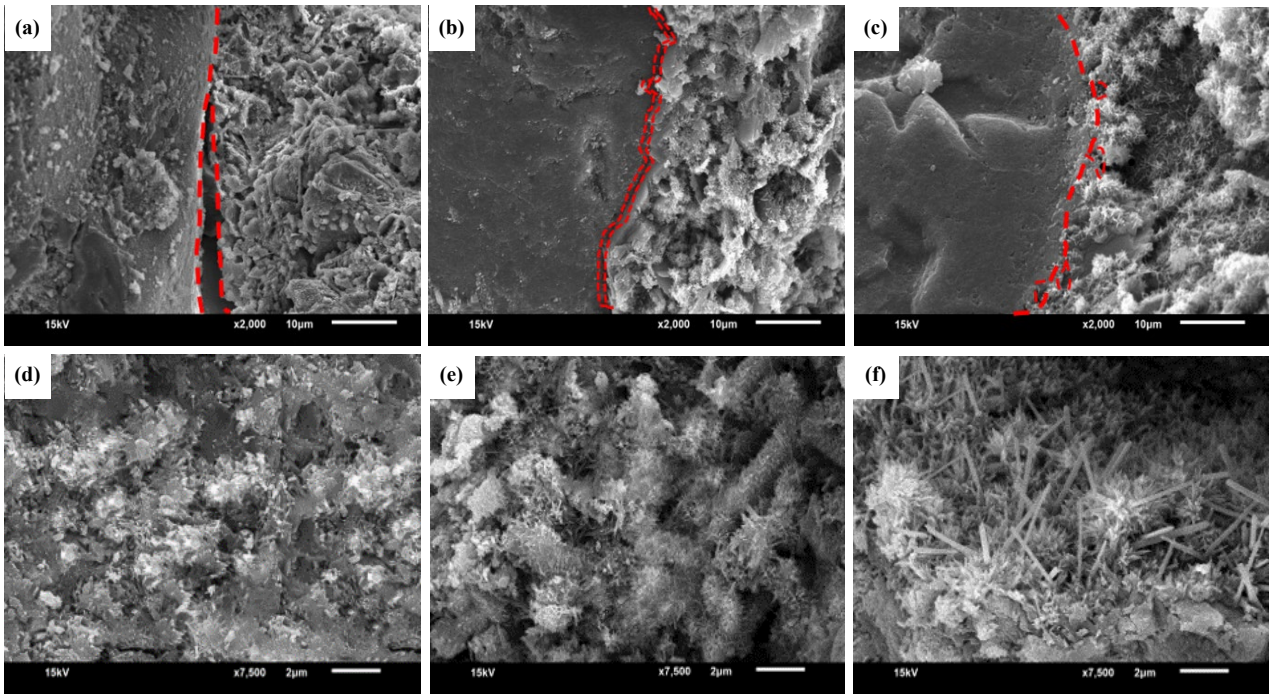

Figure 4. SEM images of HPCM at 1 day with the addition of 2 wt% AP (RAP-2) and 2 wt% MA (RMA-2); (a), (b), and (c) Represent the interfacial transition zones (ITZ) of REF, RMA-2, and RAP-2, respectively. (d), (e), and (f) Represent the cement pastes of REF, RMA-2, and RAP-2, respectively.

Table 4. Specific surface area obtained by nitrogen absorption.

Specific surface area	REF	RAP-2	RMA-2
BET_{N2} (m^2/g)	11.6453 ± 0.1438	15.8279 ± 0.1423	13.2677 ± 0.1637

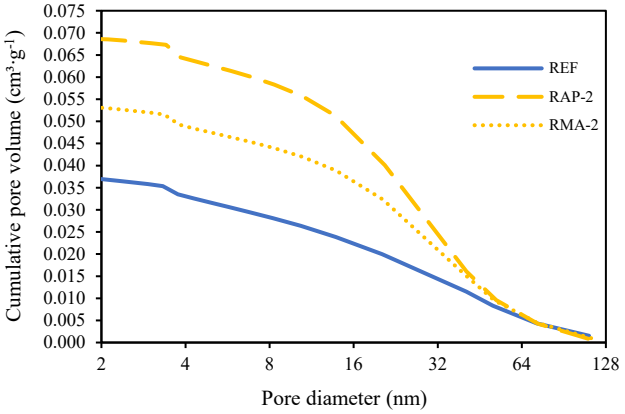

Figure 5. Pore size distribution obtained by nitrogen adsorption.

As previously stated, the proportions of the prototype HPCM were selected based on the optimal hydration temperature results, which were at the highest temperature given for each PM. The mechanical properties, compressive strength, MOR, and MOE, of HPCM were enhanced at the early curing day due to increase hydration temperature [25] and hydration products as C-S-H and C-A-H by adding AP and MA as pozzolan materials resulted from the reaction of SiO_2 and Al_2O_3 in AP and MA with $Ca(OH)_2$ [26]. In addition, the enhancement of mechanical properties, compressive strength, MOR, and MOE, could also be explained by the results of microstructure modification after adding AP and MA. According to the explanation of Figure 4, control sample (REF) presented the gel-like structure of C-S-H gel, which was existed as stand-alone cluster with the presence of $Ca(OH)_2$ crystal [27]. For the sample after adding AP (RAP-2), the denser microstructure was shown in an oriented needle-like shape of ettringite and more C-S-H crystals were found as crystalline structures with the absence of $Ca(OH)_2$ [27] when compared to REF. Meanwhile, the sample after adding MA (RMA-2) showed the semi-crystalline structure of C-A-H with more oriented structure and a lower amount of $Ca(OH)_2$ [28] when compared to REF.

Moreover, in accordance to microstructure, pore size distribution and surface area results, the cement hydration rate increased due to the incorporation of PM introduced with AP and MA, alternating the form of the hydration product. The modified needle-shape altered the pore size at the ITZ and cement paste from macro size to micro size, resulting in an increase in the specific surface area of the HPCM structure and improved bonding at the ITZ. As a consequence, the mechanical properties and water absorption were enhanced while the density was decreased.

Comparatively, AP and MA revealed distinct mechanisms of hydration according to their main chemical compositions, as validated by hydration temperature; the high concentration of SiO_2 in AP could result in a faster rate of hydration product generation than the high concentration of Al_2O_3 in MA. In addition, other chemical compositions of MA, such as Na_2O, can perform the effect of alkalinity in HPCM, which stimulates microcracking in OPC during the early curing day. Although, these microcracks can likely self-heal as a consequence of continued hydration and/or stress relaxation induced by microstructural rearrangement [29]. Furthermore, the structure of the sample after the addition of AP was more orientated and compacted than that of the sample after the addition of MA. However, the improvement in needle-shaped structures in AP resulted in a higher increased surface area than in MA. Consequently, the presence of AP could have a greater effect on the early mechanical properties than the presence of MA, provided that MA reduces water absorption, surface area, and pore size distribution more than AP in HPCM.

4. Conclusions

The optimal pore modifiers enhanced the hydration kinetics and resulted in accelerating the hydration temperature from pozzolanic reaction in HPCM. The transformation of the hydration product from a gel-like structure to a semi-crystalline structure and crystalline structure as needle-shaped is developed by introducing MA and AP to HPCM, respectively. Furthermore, the presence of pore modifiers resulted in the reduction of macropores to micropores, resulting in increased mechanical properties, especially in early cure time compressive strength. Subsequently, water absorption is increased, while the density decreased. Additionally, using AP and MA as pore modifiers has the additional benefit of reducing silica content in HPCM.

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