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Development of Fly Ash-Based Geopolymer Lightweight Aggregates

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Abstract

This research delves into the utilization of microwave hybrid heating as a curing technique for producing fine aggregates from fly ash. Various concentrations of alkali activator solutions were employed as binders to pelletize the fly ash (ranging from 0% to 10%). The resultant green fly ash-based geopolymer (FAG) aggregates were subjected to a 15-minute microwave kiln treatment. The microwave-sintered FAG fine aggregates, varying in sizes from 0.60 to 2.36 mm, were then utilized to fabricate 50 mm cubic cement mortar samples. Findings indicated that the density of FAG aggregates was approximately 36% lower than that of natural sand, while the water absorption capacity of FAG aggregates exhibited a fivefold increase compared to natural sand. Notably, cement mortar samples made with FAG aggregate of 4% alkali activator exhibited maximum compressive strengths of 31, 37, and 42 MPa at 7, 14, and 28 days of curing, respectively. These compressive strengths were only 12% lower than those of cement mortars made with natural sand across all curing periods. The use of microwave hybrid heating to transform fly ash into aggregates with sufficient strength appears viable, suggesting that these manufactured FAG aggregates could serve as substitutes for natural aggregates in concrete production.

Keywords: Alkali-activated fly ash, Fly ash-based geopolymer, Geopolymer aggregates, Lightweight aggregates, Microwave hybrid heating, Sintered fly ash aggregates

1. Introduction

All around the world, more than 500 million tons of fly ash are generated annually from coal-fired power plants, with less than 30% of it being utilized in construction and other industries [1]. There is mounting pressure on coal and utility industries, along with associated waste management, to address the environmental challenges linked with coal ash storage and management. One promising avenue for high-value-added utilization of fly ash is its conversion into lightweight aggregates, serving as a natural aggregate replacement for lightweight concrete production and other applications [2]. The rationale behind this approach lies in two primary factors which are the escalating demand for aggregates, and the depletion of natural aggregate resources coupled with a decline in the establishment of new quarries [3]. The manufacturing of cost-effective, high-quality fly ash aggregate would mitigate the environmental impact of fly ash while yielding significant economic benefits [4], [5].

The fundamental process of producing aggregates from fly ash entails transforming fine fly ash particles into larger particles through various agglomeration techniques, followed by employing different curing methods to bond or solidify the agglomerated fly ash particles, thus achieving the desired aggregate properties. The manufacturing process of fly ash aggregates typically comprises the following steps: mixing fly ash and additives, agglomeration, hardening or binding of the fly ash particles, and additional processing if necessary, such as crushing and screening [6].

The primary methods employed to agglomerate fly ash particles can be categorized into non-pressure and pressure agglomeration techniques. When selecting the appropriate agglomeration equipment for a material, various factors beyond cost must be taken into consideration to ensure an informed decision. Some of these crucial factors include agglomeration equipment capabilities, properties and quantity of the feed material, characteristics of the binder, specifications for final product quality including moisture content and shape, energy

consumption requirements, production environment considerations, and production rate requirements. Careful assessment of these factors will facilitate the selection of the most suitable agglomeration approach for the material at hand, ensuring optimal process efficiency and product quality [7].

The primary advantage of pressure agglomeration is typically lower processing costs, often due to minimal or no binder requirement and the absence of drying or heating procedures. However, this advantage doesn't directly apply to fly ash aggregate manufacturing due to the necessity of adding a binder in the process. Additionally, drying or heating steps are essential in certain pressure agglomeration techniques like sintering, hydrothermal, and some cold bonding processes. Moreover, after pressure agglomeration, the pellets must undergo crushing to achieve the desired sizes of fly ash aggregate, thereby adding extra costs to the manufacturing process [7].

Conversely, non-pressure agglomeration typically demands lower capital investment. Additionally, the incorporation of binders' aids in enhancing pelletization efficiency and achieving optimal aggregate properties. Furthermore, employing pelletization as an agglomeration technique can circumvent the need for supplementary processing such as crushing and screening. This streamlining of steps not only reduces energy consumption but also simplifies the design of the production line and diminishes the footprint of the manufacturing plant [6]. For these compelling reasons, the non-pressure agglomeration process is adopted in this research.

Bonding methods in aggregate manufacturing can be categorized based on the curing temperature utilized, with three main approaches: Sintering (above 900°C), hydrothermal processes (100-250°C), and cold bonding (below 100°C) [4], [6]. Sintering is often regarded as the optimal method for producing high-quality lightweight aggregates such as Lytag. However, its high cost and the increased amount of cement required to attain satisfactory concrete strength render it more suitable for specialized applications [4], [8]. Conversely, aggregates produced through hydrothermal and cold bonding processes tend to be limited to the production of low-strength concrete or concrete blocks due to their restricted compressive strength [4].

There are limited studies in literature focusing on the use of fly ash as an aggregate. However, these studies often incorporate additional components such as cement, ground granulated blast furnace slag (GGBS), and rice husk ash (RHA) to enhance strength [9]-[11]. Moreover, curing fly ash pellets at temperatures of 100°C or lower with extended curing periods has been explored, but this approach is deemed uneconomical and impractical [12], [13]. While there are some studies in the literature discussing microwave sintering or microwave hybrid heating as curing methods, these primarily focus on ceramics [14]-[17], composites [18]-[22], metallic materials [23], and glass [24]. Only a few works have been cited in the literature regarding microwave sintering of fly ashes, albeit without alkali activation [25]-[27]. To date, the utilization of microwave hybrid heating for curing fly ash-based geopolymer (FAG) has not been documented in the literature.

In this study, a disc pelletizer was employed to produce lightweight fly ash aggregate through the pelletization technique. Subsequently, conventional oven curing and microwave hybrid heating were utilized to cure the FAG aggregates. The investigation encompassed three primary stages:

- Examination of the influence of alkali activator content on the properties of FAG aggregates cured using conventional oven curing, followed by an analysis of the strength development of cement mortars incorporating these oven-cured FAG aggregates.
- Introduction of microwave hybrid heating as an alternative curing method for FAG aggregates, followed by an assessment of the properties of microwave-sintered FAG aggregates and the subsequent strength development of cement mortars made with these aggregates.
- Utilization of various alkali activator contents and adjustment of the batch weight of the FAG aggregate during microwave hybrid heating to further explore the impact of alkali activator content and batch weight on the properties of microwave-sintered FAG aggregates, as well as on the properties of cement mortars incorporating these aggregates.

2. Experimental Work

2.1. Materials

Unclassified fly ash served as the primary raw material for producing the FAG aggregate. This fly ash can be classified as low calcium (class F) according to the ASTM C618 standard. The chemical composition of the fly ash, along with its loss on ignition (LOI), is presented in Table 1. The alkali activator for the fly ash was sodium metasilicate anhydrous. The fly ash alkali activator solution was prepared by dissolving the necessary quantity of sodium metasilicate anhydrous beads in tap water. Table 2 provides an overview of the chemical and physical characteristics of sodium metasilicate anhydrous. General-purpose ordinary Portland cement was utilized in all mortar mixes, and Table 1 outlines the chemical composition of the cement. Additionally, washed natural sand was used to create the mortar control mix.

Table 1. Chemical composition of fly ash and cement (wt. %).

Oxides	Al_2O_3	SiO_2	CaO	Fe_2O_3	K_2O	MgO	Na_2O	P_2O_5	TiO_2	MnO	SO_3	LOI
Fly ash	25.2	63.2	0.07	3.36	1.81	0.57	0.72	0.25	0.99	0.07	0.18	1.31
Cement	5.50	19.30	62.0	2.90	0.40	1.40	0.06	0.11	0.30	0.20	2.46	2.30

Table 2: Physical and chemical characteristics of sodium metasilicate anhydrous.

SiO_2 %	Na_2O %	Particle size (0.3 – 1.0 mm) %	Bulk density (g/cm^3)	Whiteness %
45.0 - 47.0	50.0 – 52.0	93 (minimum)	0.98 – 1.35	8

2.2. Manufacturing of FAG Lightweight Fine Aggregate

A laboratory pelletizer was utilized to manufacture the FAG lightweight fine aggregate. The pelletizing disc had dimensions of 60 cm in diameter and 12.5 cm in depth. This pelletizer disc could be adjusted to an angle ranging from 40° to 60° and operated at speeds between 12 to 34 RPM (refer to Fig. 1). Upon feeding approximately 2 kg of fly ash into the disc, the required quantity of alkali activator solution was sprayed onto the fly ash to form spherical green pellets, known as green fly ash aggregates. Various alkali activator contents ranging from 0% to 10% by the weight of fly ash were employed during the production of the FAG aggregate. Pelletization typically took around 20 minutes to complete. The amount of sprayed activator solution utilized during pelletization was calculated to serve as the binding agent for forming spherical pellets under the motion of the rotating disc.

Figure 1. Image of the laboratory pelletization disc utilized in the production of green FAG lightweight aggregates.

Achieving maximum pellet strength entails ensuring that all capillaries are filled with alkali activator solution throughout the pelletization process. Insufficient activator solution may lead to the trapping of air voids within the pellet's pore structure, limiting capillary action and weakening the final pellet's strength. Conversely, an excessive amount of activator solution can result in the formation of an activator solution film on the pellet surface, disrupting capillary forces [28], [29].

Following pelletization, the green fly ash aggregates underwent curing using two distinct methods: conventional oven curing and microwave hybrid heating. For oven curing, a dry oven was employed to cure the green fly ash aggregates at a temperature of 90°C for 24 hours. Microwave hybrid heating, on the other hand, was conducted in a microwave oven utilizing a microwave kiln. The green fly ash aggregates were cured at a power of 1200 W for 15 minutes. The microwave kiln features an aluminosilicate refractory with a specialized internal lining composed of silicon carbide, known as a microwave susceptor. This susceptor absorbs microwave energy, converting it into heat, which is then transferred to the specimen. Fig. 2 displays an image of the microwave kiln utilized in this study for curing the FAG aggregate, while Fig. 3 depicts the microwave-sintered aggregates immediately following microwave hybrid heating, which were subsequently allowed to cool rapidly to ambient temperature. Following curing, the hardened FAG aggregates were sieved into fractions ranging from 0.6 to 2.36 mm in size for use in mortar mixes.

Figure 2. Image of the microwave kiln utilized in microwave hybrid heating.

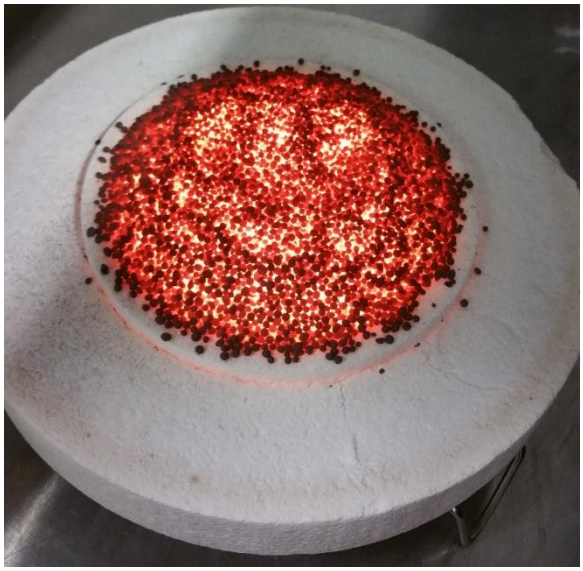

Figure 3. Image showing microwave-sintered aggregates immediately after undergoing microwave hybrid heating. In the image, the lid of the microwave kiln has been removed, revealing the sintered aggregates within the kiln.

2.3. Mortar Specimen Preparation and Curing

Before preparing the mortar mixes, both natural sand and FAG fine aggregates underwent testing according to ASTM C128 standards to determine their density, specific gravity, and water absorption. Due to the high water absorption capacity of the FAG fine aggregates and to ensure uniformity across all mortar mixes, both the natural sand and the cured FAG aggregates were utilized in their SSD (saturated surface dry) condition to prevent absorption of mixing water. It's worth noting that both the natural sand and the cured FAG aggregates exhibited the same grading which conforms to ASTM C33 standard specifications, as indicated in Table 3.

Table 3. Grading of natural sand and FAG aggregates

Sieve size mm)	9.5	4.75	2.36	1.18	0.60
% Passing	100	100	100	60	0

For all mortar mixes, a cement-to-fine aggregate volume ratio of 1:3 and a water-to-cement ratio of 0.5 were employed. The fine aggregates and cement were initially dry-mixed in a mechanical mixer for 1 minute. Subsequently, water was added, and mixing was continued for an additional 4 minutes. The resulting mortar mixture was then molded into cubic molds with 50 mm edges. The mortar in the molds was compacted for 1 minute using a vibrating table to ensure proper consolidation. After 24 hours, the mortar specimens were demolded and subsequently cured in a water tank at a temperature of 23°C for varying curing durations, namely 7, 14, and 28 days.

2.4. Compressive Strength Test

The compressive strength of the mortar specimens was evaluated using a Technotest 300 kN testing machine with a load rate of 0.5 N/mm²/second. Testing was done according to ASTM C109 standards. Three samples were tested for each mixture, and the average value of the compressive strength was recorded.

3. Results and Discussion

3.1. Oven Heating Method

3.1.1. Properties of oven-cured FAG aggregates

The oven-cured FAG aggregate produced with fly ash exhibits a gray coloration, featuring predominantly rounded to sub-rounded particles. An image depicting the appearance of the oven-cured FAG aggregate is provided in Fig. 4.

In Table 4, the physical properties of FAG aggregates after oven curing at 90°C for 24 hours were compared with those of natural sand. The density of the manufactured FAG aggregates was 35% lower than that of natural sand. For oven-cured FAG aggregates, the saturated surface dry densities were 1,677 kg/m³ and 1,681 kg/m³ for alkali activator contents of 5% and 10%, respectively. In comparison, the saturated surface dry density of natural sand was 2,585 kg/m³. On the other hand, the water absorption capacity of FAG aggregates was significantly higher, measured at 31% and 30% for alkali activator contents of 5% and 10%, respectively, compared to 1.3% for natural sand.

Figure 4. Image of the oven-cured FAG aggregate.

Table 4. Properties of natural sand and oven-cured FAG aggregates

Material	SSD Density (kg/m³)	Water absorption (%)
Natural sand	2585	1.3
Oven-cured FAG aggregate (5% alkali activator)	1677	31
Oven-cured FAG aggregate (10% alkali activator)	1681	30

These differences in properties are likely related to the green pellet-forming mechanism during the pelletization process, which traps air and water between fly ash particles. During oven curing, the trapped water evaporates, leaving behind porous pellets. It's worth noting that the alkali activator content has a minor effect on the water absorption capacity of oven-cured FAG aggregates.

3.1.2. Properties of cement mortars containing oven-cured FAG aggregate

The cement mortars prepared with oven-cured FAG aggregates exhibited a weight reduction of 20 to 21.4% compared to the control cement mortar utilizing natural sand. Following a 28-day water curing period, the saturated surface dry densities of these cement mortars ranged from 1,742 to 1,776 kg/m³ for alkali activator contents of 5% (GM1) and 10% (GM2), respectively. In contrast, the saturated surface dry density of the control cement mortar (CM) was measured at 2,216 kg/m³. This variation in density can be attributed to the inherent lower densities of FAG aggregates discussed previously.

Fig. 5 shows the strength development of cement mortars manufactured with natural sand and oven-cured FAG aggregates. For cement mortars incorporating oven-cured FAG aggregates with a 5% alkali activator (GM1), the compressive strengths were 17, 20, and 23 MPa after 7, 14, and 28 days of curing, respectively. These values represent 49%, 48%, and 49% of the compressive strengths of mortars made with natural sand, which were 35, 42, and 47 MPa, respectively. Similarly, for cement mortars containing oven-cured FAG aggregates with a 10% alkali activator (GM2), the compressive strengths were

20, 22, and 24 MPa after 7, 14, and 28 days of curing, respectively. These values correspond to 57%, 52%, and 51% of the compressive strengths of mortars made with natural sand, respectively.

Traditional cement mortar and concrete can be conceptualized as composite systems comprising three key components: cement paste, aggregate, and the interfacial transition zone between the cement paste and aggregate. The strength of concrete and mortar is heavily influenced by the properties of these components. In traditional cement mortar, where normal-weight aggregates like natural sand are used, the aggregate component is typically the strongest. Consequently, stresses tend to concentrate in the aggregates and are transferred to the interfacial transition zone between the cement paste and aggregate. As a result, failure often occurs due to the breaking of the cement paste-aggregate interfaces. However, when cement mortars are produced with weak aggregates, such as lightweight aggregates, cracks may develop through the aggregates, leading to a reduction in the overall strength of the composite system (i.e., the mortar) [30]-[40].

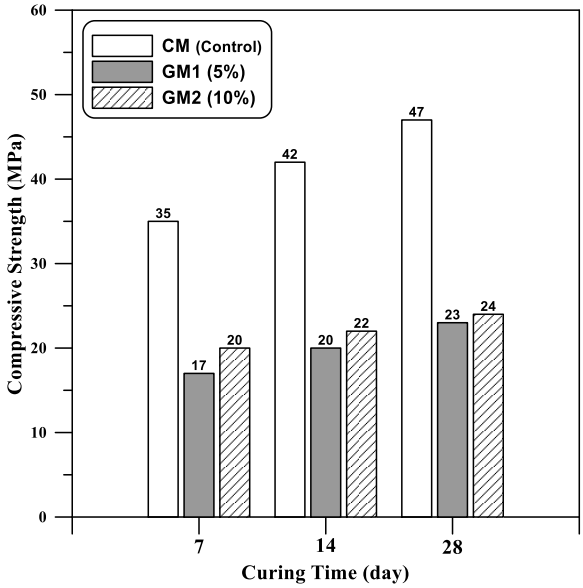

Figure 5. Strength development of cement mortars containing natural sand and oven-cured FAG aggregates.

Upon visual inspection of fracture failure surfaces of mortar specimens composed of oven-cured FAG aggregates after compression testing, it becomes apparent that the manufactured aggregates constituted the weakest component within the mortar composite structure. The fracture path extended through the aggregates, with the majority of oven-cured FAG aggregates exhibiting cracking and splitting into halves, as depicted in Fig. 6.

The findings also reveal that the strength development rate of mortars crafted with oven-cured FAG aggregates, utilizing a 5% alkali activator, closely mirrors that of mortars made with natural sand. Specifically, for 7-day and 14-day curing periods, the compressive strength reached 74% and 87%, respectively, of the 28-day compressive strength of the mortars composed of oven-cured FAG aggregates with a 5% alkali activator. This pattern aligns with the strength development rate observed in mortars made with natural sand, where the compressive strength at 7 days and 14 days accounted for 74% and 89%, respectively, of the 28-day compressive strength.

When the alkali activator content was increased from 5% to 10%, a noticeable trend of enhanced strength development rate emerged in mortars comprising oven-cured FAG aggregates compared to mortars containing natural sand. Specifically, for 7-day and 14-day curing periods, the compressive strength reached 83% and 92%, respectively, of the 28-day compressive strength of the mortars crafted with oven-cured FAG aggregates utilizing a 10% alkali activator. These values represent a 9% and 3% increase, respectively, in the strength development rate compared to mortars made with natural sand for the same curing durations. Further investigation is needed to comprehensively understand this behavior, although it falls beyond the scope of the current study.

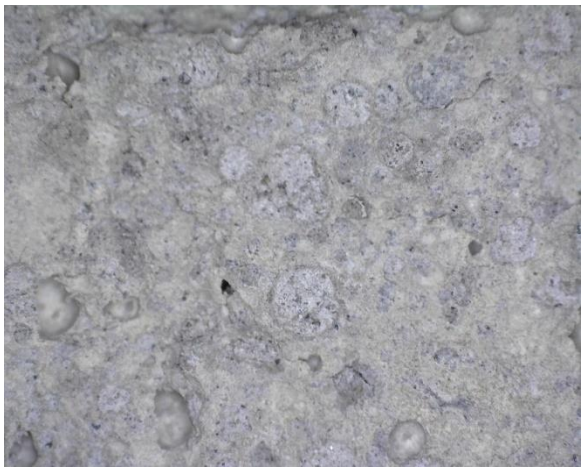

Figure 6. Image of fracture failure surfaces of the mortar specimen containing oven-cured FAG aggregates after compression test.

The above results suggest that oven-cured FAG aggregates, derived from unclassified (run-of-station) fly ashes, hold promise for use in the production of grade M20 (20 MPa) structural reinforced concrete, commonly employed in residential constructions. Subsequent investigations aimed to explore the potential of producing stronger FAG aggregates, and thereby, more robust cement mortars or concretes incorporating these geopolymer aggregates. This was achieved through the utilization of microwave hybrid heating as a novel curing method, detailed further below.

3.2. Microwave Hybrid Heating Method

3.2.1. Properties of FAG aggregates cured using microwave hybrid heating

Three alkali activator solutions with varying alkali activator contents - 2%, 5%, and 10% (by the weight of the fly ash) - were utilized to pelletize the fly ash. Subsequently, the green FAG aggregates were subjected to curing using a microwave kiln at 1200 W power for 15 minutes. Additionally, non-activated pelletized fly ash aggregates (i.e., 0% alkali activator content) underwent the same curing procedure for comparative

analysis. Each batch consisted of 350 g of green fly ash aggregate, which is the maximum capacity of the microwave kiln. After curing, microwave-sintered FAG aggregates near or in contact with the kiln lining (i.e., the susceptor) exhibited a light brown color, whereas aggregates at the center of the kiln appeared grey. This disparity in coloration may be attributed to the differential curing temperatures experienced by the fly ash aggregates. Aggregates in proximity to the microwave susceptor were subjected to higher temperatures compared to those positioned at the center of the kiln. To validate this hypothesis, an infrared thermometer was employed to measure temperatures at both the center and the lining of the microwave kiln (i.e., the interior wall). Results indicated that the temperature of the microwave kiln lining (i.e., the susceptor) could reach levels between 200 to 300°C higher than those at the center of the microwave kiln during the heating process. A maximum temperature of 810°C was recorded at the center of the microwave kiln after 15 minutes of microwave heating. An image depicting the microwave-sintered FAG aggregates is presented in Fig. 7.

Figure 7. Image of microwave-sintered FAG aggregates.

The microwave-sintered non-activated fly ash aggregates (i.e., with 0% alkali activator content) predominantly disintegrated into fine particles post-curing, displaying no indications of hardening or sintering. Furthermore, microwave-sintered FAG aggregates produced with a 2% alkali activator exhibited weakness and were easily broken by hand. Consequently, it was determined that only microwave-sintered FAG aggregates prepared with 5% and 10% alkali activator content would be utilized for the production of cement mortars.

Table 5 presents the physical properties of FAG aggregates after microwave hybrid heating at 1200 W for 15 minutes, alongside those of natural sand. The saturated surface dry density of the microwave-sintered FAG aggregates is observed to be 36% lower than that of natural sand. Specifically, the saturated surface dry densities of microwave-sintered FAG aggregates were measured at 1,652 and 1,649 kg/m^3 for alkali activator contents of 5% and 10%, respectively, while the corresponding density of natural sand stood at 2,585 kg/m^3. In contrast, the water absorption capacity of FAG aggregates (8.4% and 8.6%) was found to be 6 times higher than that of natural sand (1.3%).

Table 5. Properties of microwave-sintered FAG aggregates cured via microwave hybrid heating using 350 g/batch.

Alkali activator (%)	SSD Density (kg/m^3)	Water absorption (%)
5	1652	8.6
10	1658	8.4

Notably, the water absorption capacity of microwave-sintered FAG aggregates exhibited a notable reduction compared to that of oven-cured FAG aggregates. This reduction amounted to more than threefold, with the water absorption capacity decreasing from an average of 30.5% for oven-cured aggregates to an average of 8.5% for microwave-sintered aggregates.

Additionally, the saturated surface dry density of microwave-sintered FAG aggregates was observed to be lower than that of oven-cured counterparts, with a density decrease of 5% and 7% for FAG aggregates made with alkali activator contents of 5% and 10%, respectively. Furthermore, the results indicate that alkali activator content has a minor effect on the properties of microwave-sintered FAG aggregates.

The decrease in water absorption capacity observed in microwave-sintered FAG aggregates is primarily attributed to the reduction in both total and open porosity, which results from the sintering process and the densification of the samples.

3.2.2. Properties of cement mortars containing microwave-sintered FAG aggregates

The saturated surface dry densities of cement mortars composed of microwave-sintered FAG aggregates and water-cured for 28 days were measured at 1,655 and 1,728 kg/m^3 for alkali activator contents of 5% (GM3) and 10% (GM4), respectively. These densities were found to be 25% and 22% lighter than the saturated surface dry density of the control cement mortar (2,216 kg/m^3), respectively. Moreover, the saturated surface dry densities of cement mortars for GM3 and GM4 mixes, made with microwave-sintered FAG aggregates, were 5% and 3% lighter than the saturated surface dry densities of the cement mortars for GM1 and GM2 mixes, respectively, which were made with oven-cured FAG aggregates.

Fig. 8 illustrates the strength development of cement mortars incorporating microwave-sintered FAG aggregates. For mortars containing 5% alkali activator (GM3), the compressive strength was recorded as 22, 24, and 26 MPa after curing for 7, 14, and 28 days, respectively. These values represent 63%, 57%, and 55% of the compressive strength observed in mortars made with natural sand (35, 42, and 47 MPa), respectively. On the other hand, for cement mortars composed of microwave-sintered FAG aggregates containing 10% alkali activator (GM4), the compressive strength of mortars was measured at 26, 30, and 33 MPa for curing periods of 7, 14, and 28 days, respectively. These values represent 74%, 71%, and 70% of the compressive strength of mortars made with natural sand, respectively.

The utilization of microwave hybrid heating enhanced the characteristics of the FAG aggregates, resulting in an improvement in the strength of the cement mortars fabricated with microwave-sintered fly ash geopolymer aggregates.

Furthermore, there is a discernible pattern indicating that increasing the alkali activator content contributes to the enhancement of the properties of microwave-sintered FAG aggregates.

The temperature variations during microwave hybrid curing ranged between 200 to 300°C, contingent upon the measurement location (either at the center or the inner sides of the microwave kiln) as discussed previously. This discrepancy leads to differences in the properties of the microwave-sintered FAG aggregates, evident from the color variation observed (grey at the center and light brown at the inner sides of the kiln). To mitigate the variation in properties of the microwave-sintered FAG aggregates, it could be beneficial to position the green aggregates (pellets) uniformly, such as at the center of the kiln, by employing smaller batch weights.

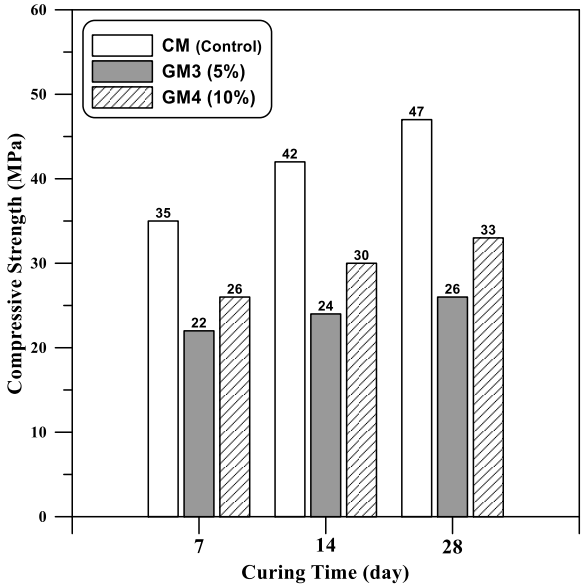

Figure 8. Strength development of mortars containing microwave-sintered FAG aggregates cured via microwave hybrid heating using 350 g/batch.

Additional investigations were conducted to explore the impacts of alkali activator content, sintering temperature, and batch weight on the characteristics of microwave-sintered FAG aggregates, as well as their effects on the properties of cement mortars fabricated using these aggregates.

3.2.3. The Effect of batch weight on the properties of microwave-sintered FAG aggregates and cement mortars containing these aggregates

The fly ash underwent pelletization using varying alkali activator contents, specifically 4%, 6%, 8%, and 10%. Subsequently, the green FAG aggregates were subjected to curing in a microwave kiln at 1200 W power for 15 minutes. Each batch consisted of 100 g of green fly ash aggregates, positioned at the center of the microwave kiln, in contrast to the previous 350 g batches. Following the curing process, the microwave-sintered FAG aggregates produced with 4% and 6% alkali activator contents displayed ease of separation from each other. However, those produced with an 8% alkali activator exhibited fusion at contact points yet could be separated without causing damage. Conversely, aggregates produced with 10% alkali activator were fully fused at contact points, rendering separation impossible without disintegration, as depicted in Fig. 9. Consequently, only the microwave-sintered FAG aggregates produced with 4%, 6%, and 8% alkali activators were subjected to testing and subsequently utilized in cement mortar preparation. These aggregates exhibited a color range from light brown to light grey, comprising mostly rounded to sub-rounded particles, as illustrated in Fig. 10.

Figure 9. Image of microwave-sintered FAG aggregates containing alkali activator content of 10%.

Figure 10. Image of microwave-sintered FAG aggregates containing alkali activator content of 4%.

It should be noted that the issue of fusion could potentially be addressed by employing a rotating kiln. This method mitigates the aggregation of aggregates by facilitating continuous motion during the heating process, thus preventing fusion between individual particles.

The characteristics of microwave-sintered FAG aggregates are outlined in Table 6. Generally, there is a decrease in the saturated surface dry density of microwave-sintered FAG aggregates with an increase in the alkali activator content. Conversely, an increase in the water absorption capacity is observed as the alkali activator content rises. The saturated

surface dry density of these aggregates is approximately 37% to 38% lower than that of natural sand (2,585 kg/m^3). Specifically, the saturated surface dry densities of microwave-sintered FAG aggregates were recorded as 1,640 kg/m^3, 1,627 kg/m^3, and 1,590 kg/m^3 for alkali activator contents of 4%, 6%, and 8%, respectively. Regarding water absorption, FAG aggregates exhibit a significantly higher capacity compared to natural sand, ranging from 6 to 8 times higher. For alkali activator contents of 4%, 6%, and 8%, the water absorption capacities of these aggregates were measured at 8%, 8.9%, and 10.5%, respectively. It is evident that the water absorption capacity increases as the saturated surface dry density of microwave-sintered FAG aggregates decreases.

Table 6. Properties of microwave-sintered FAG aggregates cured via microwave hybrid heating using 100 g/batch.

Alkali activator (%)	SSD Density (kg/m^3)	Water absorption (%)
4	1640	8.0
6	1627	8.9
8	1590	10.5

The optical microscopic images provide insights into the microstructure of the microwave-sintered FAG aggregates. In the case of aggregates with 4% alkali activator content (Fig. 11), small-sized voids are scattered throughout the cross-section cut. Conversely, with an increase in alkali activator content to 8% (Fig. 12), larger-sized voids become dominant across the cross-section cut. Furthermore, aggregates produced with an 8% alkali activator exhibit a significantly higher number of voids compared to those with a 4% alkali activator. This microscopic observation suggests that, under similar microwave heating conditions, higher alkali activator content results in the formation of larger voids with a greater overall volume. Consequently, this leads to a reduction in density and an increase in water absorption capacity in the microwave-sintered aggregates.

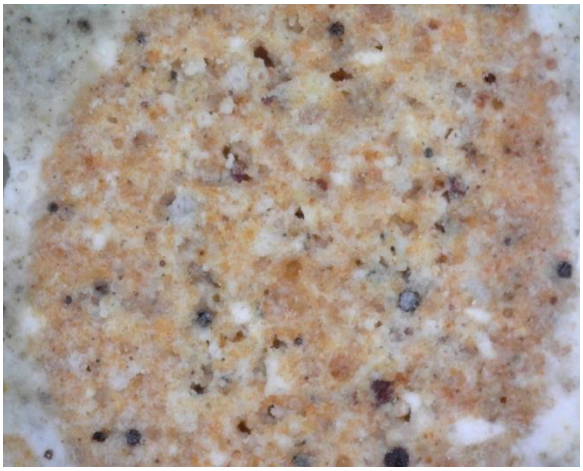

Figure 11. Optical microscopic image of microwave-sintered FAG aggregate cross-section cut (containing 4% alkali activator).

The above investigation revealed that adjusting the batch weight of the uncured aggregates had a significant impact on the properties of the microwave-sintered FAG aggregates in relation to the alkaline activator content. To gain deeper insights into the curing conditions during microwave hybrid heating, the internal temperatures of the microwave kiln were closely monitored using a thermocouple. Table 7 outlines the temperature data recorded at the center of the kiln during the curing process of green FAG aggregates at a power setting of 1200 W for various durations (ranging from 1 to 15 minutes) with differing batch weights (100 g and 350 g). In general, there was a decline in the heating rate as the curing time advanced. With a batch weight of 350 g, the heating rate started at 105-132°C/min over the initial three minutes, followed by a reduction to 67-89°C/min in the subsequent two minutes. This was then followed by a further decrease to a range of 10-44°C/min. Conversely, when using a 100 g batch weight, the heating rate was notably higher. For the 350 g batch, the kiln's internal temperature peaked at 810°C after 15 minutes of curing, with this temperature being reached after 5 minutes when the batch weight was reduced to 100 g. The heating rate for the 100 g batch was considerably higher, peaking at 180-200°C/min in the first 3 minutes before gradually declining to 80-110°C/min over the next two minutes. Subsequently, the rate continued to decrease, reaching a range of 30-60°C/min. The maximum recorded temperature at the kiln's interior for the 100 g batch after 15 minutes of curing was 1197°C, marking a 48% increase compared to the maximum temperature recorded for the 350 g batch over the same curing duration.

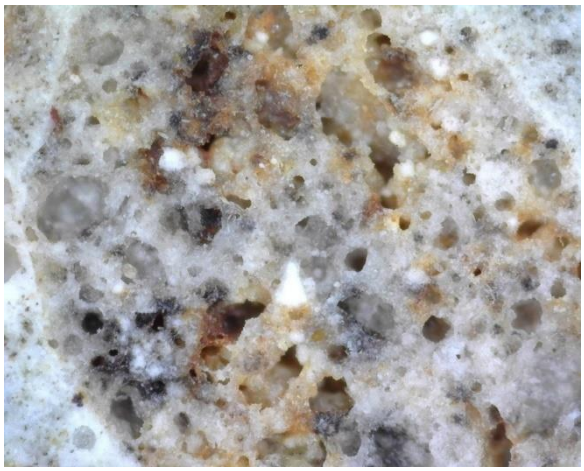

Figure 12. Optical microscopic image of microwave-sintered FAG aggregate cross-section cut (containing 8% alkali activator).

The observed differences in heating rates and temperatures can be attributed to the dynamics of microwave energy absorption during the curing process. When curing the maximum batch weight of green FAG aggregates (350 g), a significant portion of the microwave energy is absorbed by the aggregates themselves. This results in a slower heating rate of the microwave kiln susceptor, which in turn limits the maximum curing temperature to approximately 800°C. Conversely, reducing the batch weight of green FAG aggregates to 100 g increases the amount of microwave energy absorbed by the

microwave kiln susceptor. This leads to a faster heating rate and allows for an increase in curing temperatures beyond 800°C, reaching a maximum of approximately 1200°C.

It appears that at lower heating rates and curing temperatures below 800°C, the alkali activator content does not notably impact the properties of the microwave-sintered fly ash-based aggregates, as indicated in Table 6. However, at higher heating rates and curing temperatures ranging from 800°C to 1200°C, increasing the alkali activator content tends to reduce the density and increase the water absorption capacity of the microwave-sintered fly ash-based aggregates. Furthermore, employing an alkali activator content exceeding 6% at elevated curing temperatures can lead to surface melting of the FAG aggregates. This phenomenon results in the fusion of aggregates at points of contact. The complete fusion observed in the microwave-sintered FAG aggregates produced with a 10% alkali activator, leading to aggregate disintegration during separation, aligns with this explanation.

Table 7. Microwave kiln interior temperatures during operation at 1200 W microwave power using different batch weights of green FAG aggregates.

Curing time (min)	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Microwave kiln interior temp. (°C) for 100 g batch weight	23	195	390	580	710	800	865	911	942	1004	1060	1097	1124	1152	1176	1197
Microwave kiln interior temp. (°C) for 350 g batch weight	23	128	260	377	466	533	552	596	623	664	708	730	741	760	786	810

The saturated surface dry densities of the cement mortars, incorporating microwave-sintered FAG aggregates and subjected to water curing for 28 days, were recorded as 1,765, 1,685, and 1,654 kg/m^3 for alkali activator contents of 4% (GM5), 6% (GM6), and 8% (GM7), respectively. These values represent reductions of 20%, 24%, and 25% when compared to the saturated surface dry density of the control cement mortar (2,216 kg/m^3). It is evident that as the alkali activator content increases, the saturated surface dry densities of the cement mortars incorporating microwave-sintered FAG aggregates decrease.

Fig. 13 presents the strength development of cement mortars incorporating microwave-sintered FAG aggregates with varying alkali activator contents. For cement mortars containing 4% alkali activator (GM5), compressive strengths of 31, 37, and 42 MPa were recorded after 7, 14, and 28 days of curing, respectively. These values correspond to 89%, 88%, and 89% of the compressive strengths of mortars made with natural sand (35, 42, and 47 MPa), respectively. In contrast, cement mortars made with microwave-sintered FAG aggregates containing 6% and 8% alkali activators (GM6 and GM7) exhibited lower compressive strengths compared to those made with 4% alkali activators for all curing durations.

Increasing the alkali activator content beyond 4% did not lead to improvements in the strengths of the microwave-sintered FAG aggregates, thereby failing to enhance the maximum compressive strength of the resulting cement mortars. Additionally, there were no significant differences in compressive strength values observed for cement mortars containing microwave-sintered FAG aggregates with 6% and 8% alkali activators, particularly for 14- and 28-day curing periods.

Reducing the batch weight of the uncured aggregates has significantly enhanced the compressive strengths of cement mortars incorporating microwave-sintered FAG aggregates, while also reducing the required amount of alkali activator content. Specifically, the 28-day compressive strength of GM5 (4% activator content and 100 g batch weight) cement mortars is 62% higher than that of GM3 (5% activator content and 350 g batch weight) and 27% higher than that of GM4 (10% activator content and 350 g batch weight).

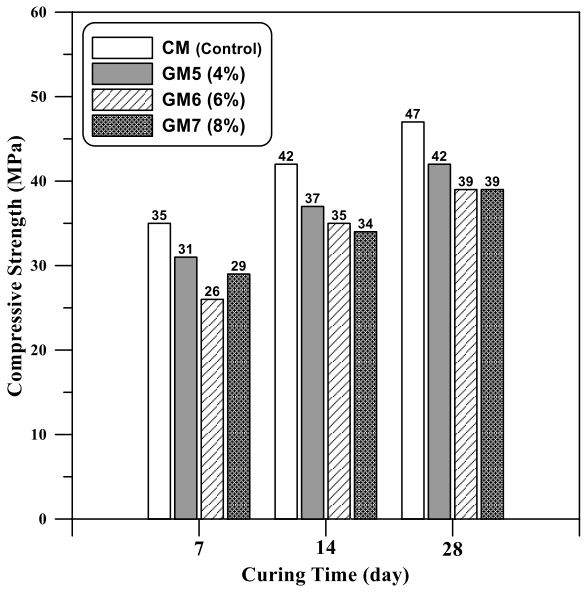

Figure 13. Strength development of mortars containing microwave-sintered FAG aggregates cured via microwave hybrid heating using 100 g/batch.

Upon visual examination of fracture failure surfaces of mortar specimens incorporating microwave-sintered FAG aggregates following compression testing, it becomes apparent that the manufactured aggregates contributed significantly to the strength of the mortar composite structure. The failure observed is primarily attributed to the breaking of cement paste-aggregate interfaces. Notably, only a small percentage of the microwave-sintered FAG aggregates exhibited cracking, as illustrated in Fig. 14.

By controlling both the alkali activator content and batch weight, microwave-sintered FAG aggregates with varying

properties can be effectively produced. Furthermore, it has been demonstrated that high-quality microwave-sintered FAG lightweight aggregates can be manufactured with an alkali activator content as low as 4% by weight of fly ash. Such lightweight aggregates exhibit favorable characteristics and can be utilized in the production of grade M40 (40 MPa) high-strength lightweight structural reinforced concrete.

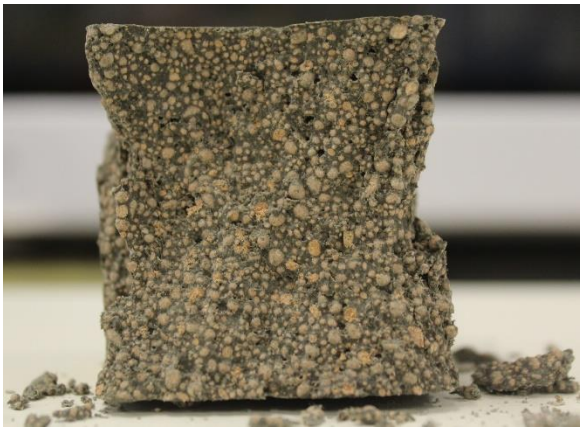

Figure 14. Image of the mortar specimen containing microwave-sintered FAG aggregates after compression test.

4. Conclusions

The primary objective of this study was to evaluate the viability of rapidly producing cost-effective and high-quality FAG aggregates using microwave hybrid heating as a novel curing technique. Results revealed that the density of microwave-sintered FAG aggregates was approximately 36% lighter compared to natural aggregates, while the water absorption capacity of these aggregates increased fivefold in comparison to natural sand. Moreover, by adjusting the alkali activator content and batch weight, microwave-sintered FAG aggregates with varied properties could be tailored. The compressive strengths of cement mortar specimens incorporating microwave-sintered FAG aggregates with 4% alkali activator content were only around 12% lower than those made with natural sand across all curing durations. In summary, microwave hybrid heating presents a promising alternative to traditional oven curing for the production of FAG aggregates, offering economic benefits and efficient curing.

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Conflict of interest

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

Author Contribution Statement

Author 1 proposed the research problem and design, collected the data, and analyzed and interpreted the results.

Both authors contributed to the final manuscript.

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