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MECHANICAL AND DURABILITY PROPERTIES OF MORTARS CONTAINING OLIVE WASTE ASH

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Abstract: The potential usage of olive waste ash (OWA) as a partial replacement for cement in mortar production is investigated in this study. The experimental program was conducted in two phases. In the first phase, OWA was calcined at 400 °C and 600 °C, then prepared in three particle size ranges (<90 µm, 90–180 µm, and 180–360 µm). The most suitable calcination conditions and particle size were identified to achieve high pozzolanic activity. Using these optimum conditions, the second phase involved producing mortars with different replacement levels (5–20% by weight of cement), and evaluating mechanical strength, alkali–silica reaction (ASR), capillary water absorption, and microstructural properties. Results from the first phase showed that finer particles and higher calcination temperatures enhanced pozzolanic activity as expected, and the processing conditions of OWA for the second phase are determined. In the second phase, strength decreased gradually with increasing replacement levels, although acceptable results were generally obtained at 10–15% replacement. Accelerated mortar bar tests (AMBT) indicated decreases in ASR expansion in mortars containing OWA, while capillary water absorption tests revealed higher water uptake due to increased porosity. XRD and SEM analyses confirmed the participation of OWA in pozzolanic reactions and the formation of calcium–silicate–hydrate (C–S–H). Overall, the findings demonstrate that OWA, particularly when calcined at 600 °C and ground to fine particle sizes, can be used as a sustainable supplementary cementitious material at 10–15% replacement levels.

Keywords: Olive waste ash, Supplementary cementitious material, Mortar, Pozzolanic activity index, Sustainability

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1. Introduction

Cement is the most widely used construction material globally, and its production is associated with significant environmental problems. Manufacturing Ordinary Portland cement (OPC), the most widely used type of cement, requires high energy consumption and is responsible for approximately 5–7% of total anthropogenic carbon dioxide (CO_2) emissions, making the industry one of the largest contributors to global warming (Chen et al., 2010; Hasanbeigi et al., 2010; Ekincioğlu et al., 2013; Tiseo, 2025). On average, the production of one ton of cement releases nearly 900 kg of CO_2, showing the urgent need to mitigate its environmental impact (Hasanbeigi et al., 2010). In addition to greenhouse gas emissions, cement production depletes natural resources and causes air and water pollution (Iqbal and Shafiq, 1995; Oluseyi et al., 2011; Cheriyan and Choi, 2020; Zhu et al, 2022), highlighting the critical importance of developing sustainable alternatives in civil engineering.

One promising approach to mitigate these impacts is the incorporation of supplementary cementitious materials (SCMs) (Malhotra, 2010; Owaid et al, 2012; Fantilli and Jóźwiak-Niedźwiedzka, 2021; Lourdu and Ali, 2025) into cement-based composites. SCMs such as fly ash (Ahmaruzzaman, 2010; Zeggar et al., 2019), silica fume (Shelote et al., 2023), and blast furnace slag (Kou et al., 2021) can partially replace Portland cement in concrete mixtures, thereby reducing the carbon footprint of cement-based materials. In addition to lowering CO_2 emissions, the use of SCMs contributes to reduced energy consumption and waste generation, offering multiple environmental and economic benefits (Ndahirwa et al., 2022). These materials often exhibit pozzolanic activity, reacting with calcium hydroxide to form additional calcium–silicate–hydrate (C–S–H), which enhances strength and durability. Due to the sharp rise in cement production and associated emissions over recent decades, the adoption of SCMs and other sustainable practices is considered essential to lower future emissions. Without intervention, global CO_2 emissions from the cement industry are projected to reach alarming levels by 2050 (International Energy Agency, 2009; Wei and Cen, 2019).

In recent years, agricultural waste ashes have attracted attention as potential SCMs due to their abundance, low cost, and environmental benefits. Rice husk ash (Elakkiah, 2019; Koya and Nair, 2021), palm oil fuel ash (Thomas et al., 2017; Norhasri et al., 2021), sugarcane bagasse ash (Sohal and Singh, 2021; Jha et al., 2021), wheat straw ash (Doğruyol and Çetin, 2025), corn cob ash (Ahmad et al., 2023; Kesarwani et al., 2026.), and olive waste ash (OWA) (Aburawi and Al-Madani, 2018; Tayeh et al., 2021; Ghanem et al., 2025) have been studied for their ability to partially replace Portland cement, offering both economic and technical advantages while reducing environmental impacts. Among these, OWA, derived from the calcination of olive mill solid residues, is particularly promising in regions where olive oil production is prevalent, although research on its utilization remains limited. Olive oil industries generate large quantities of solid waste annually, especially in Mediterranean countries, where over 750 million olive trees are cultivated, and 95% of the world's olive oil is produced (Hytiris et al., 2004; Bani Odi, 2007). Improper disposal of this waste, often through landfilling or uncontrolled dumping, poses serious risks to soil, water, and ecosystems due to contamination and the lack of effective recycling or reuse strategies (Bani Odi, 2007). Incorporating OWA into cementitious systems not only helps solve waste management challenges but also contributes to sustainable construction practices. Despite its potential, research on OWA is very limited compared to other SCMs, emphasizing the need for further studies to evaluate its properties, performance, and long-term implications for sustainable construction.

Previous studies have demonstrated that OWA can partially replace cement in mortar and concrete mixtures, improving mechanical performance, durability, and even fire resistance under certain conditions. For instance, Al-Akhras et al. (2009) reported that OWA addition reduced cracking and deterioration in concrete exposed to high temperatures, while Hakeem et al. (2002) showed that a combination of 5% OWA and 20% rice husk ash (RHA) significantly enhanced compressive, tensile, flexural, and bond strengths compared to control mixes. Similarly, Tayeh et al. (2021) noted that higher replacement rates decreased workability and compressive strength, whereas lower levels improved resistance to chemical attacks. Another study confirmed OWA's potential to mitigate ASR expansion, and 22% OWA was recommended as the ideal level for balancing strength and durability (Al-Akhras, 2012).

Despite these promising findings, the effectiveness of OWA depends strongly on calcination temperature, particle size distribution, and replacement ratio. Improper processing may reduce mechanical performance or increase porosity, whereas optimized conditions can enhance pozzolanic reactivity and improve long-term durability. Previous studies have employed a wide range of calcination conditions for olive waste ash, often with inconsistent or incomplete reporting. For example, Al-Akhras (2012) and Lila et al. (2020) used calcination at 900 °C, ensuring complete combustion but potentially reducing amorphous silica content due to crystallization. Dahim et al. (2022) reported a 2 h burning process without specifying the temperature, while Ghanem et al. (2025) combusted olive pomace at 500 °C for 8 h, resulting in partial retention of carbonaceous matter (LOI ≈ 14.7%) and moderate reactivity. In contrast, Aburawi and Al-Madani (2018) systematically compared 600–900 °C and found that 600 °C produced ash with superior strength and lower porosity, whereas higher temperatures led to reduced reactivity and increased water absorption. These findings highlight the importance of selecting appropriate calcination conditions. Based on this evidence, 400 °C was selected in the present study as a lower threshold to evaluate partial decomposition, while 600 °C was chosen as the upper threshold to maximize reactivity without excessive crystallization.

In addition to calcination, grinding and particle size also play a critical role in determining the reactivity of OWA. Finer fractions generally exhibit higher surface area and pozzolanic activity, whereas coarser fractions may limit performance. Al-Akhras (2012) prepared OWA by burning and subsequently grinding the ash for 2 h in a Los Angeles machine, producing particles finer than type I cement. Aburawi and Al-Madani (2018) burned olive fruit wastes, then ground the ash for 10 min before sieving through No.200 mesh (0.075 mm), highlighting the importance of grinding conditions alongside calcination. These findings emphasize the need for selecting appropriate calcination and grinding parameters. In the present study, a grinding duration of 2 h was adopted based on both literature and preliminary trials, which showed that a significant portion of material was lost within the initial 30 min. Achieving the target particle size was considered more critical than the exact grinding duration. However, systematic investigations into the combined effects of calcination parameters and particle size on OWA's performance as an SCM remain limited, underscoring the need for further research to establish standardized processing and application guidelines.

The present study aims to evaluate the mechanical and durability performance of mortars incorporating OWA as a partial cement substitute, thereby contributing to sustainable alternatives in construction. As explained in the previous paragraph, selecting the optimal calcination temperature and particle size was the first step before using them in production at different replacement ratios. Therefore, olive waste obtained from olive oil residues was subjected to calcination at 400 °C and 600 °C for two hours and prepared in three particle size ranges (<90 µm, 90–180 µm, and 180–360 µm) after 2 h of grinding and sieving. The influence of these processing parameters on the pozzolanic activity index (PAI) (referred to as the strength activity index (SAI) in ASTM standards) was assessed in Phase I. In Phase II, the flexural and compressive strength, ASR, and capillary water absorption were investigated, while microstructural analyses using X-ray diffraction (XRD) and scanning electron microscopy (SEM) were conducted to explain the mechanisms underlying OWA's pozzolanic activity and its interaction with cement hydration products. By identifying optimum calcination conditions, particle size distribution, and replacement levels, a balance between mechanical performance and durability is sought in this study, ensuring that the incorporation of OWA does not compromise mortar quality. Ultimately, the findings are expected to provide valuable insights into the effective utilization of OWA as an SCM, reducing cement consumption and mitigating environmental impacts.

2. Materials and Methods

2.1. Materials

2.1.1. Cement

Ordinary Portland cement (OPC), classified as CEM I 42.5 N according to British Standards Institution (2019), was used as the primary binder in all mortar mixtures. The specific gravity of the cement was measured as 3.09 g/cm^3. Its chemical composition was determined by X-ray fluorescence (XRF) and is shown in Table 1.

Table 1. Chemical composition of Portland cement and OWA

Chemical composition (%)	Portland Cement	OWA
SiO_2	16.83	20.19
Al_2O_3	3.84	2.28
Fe_2O_3	3.7	10.96
MgO	1.06	8.06
CaO	64.51	21.90
Na_2O	0.32	6.66
K_2O	1.1	29.13
TiO_2	0.25	-
P_2O_5	0.2	-
SO_3	4.73	0.26
Cr_2O_3	0.12	-
LOI	3.07	0.40

2.1.2. Fine aggregate

Natural river sand obtained from the Sakarya region was used as the fine aggregate. The sand was sieved through a 2 mm sieve before being used in mortar production. Sieve analysis confirmed that the gradation curve fell within the permissible limits of reference sand. The sand had a specific gravity of 2.68 g/cm^3 and a fineness modulus of 2.65, ensuring adequate particle size distribution for mortar production. This sand was selected due to its relatively high expansion values in AMBT tests, which allowed better observation of the effect of OWA on ASR expansion.

2.1.3. Superplasticizer

A polycarboxylate-based superplasticizer (Master Glenium 51) was incorporated to maintain workability at constant water-to-cement ratios. This admixture is a brown liquid with a specific gravity of 1.10 ± 0.03 g/cm^3 at 20 °C and a pH of 6.0 ± 1. Its alkali content is ≤ 5.00% by mass, and chloride content is ≤ 0.10% by mass, complying with British Standards Institution (2008).

For the specimens produced for mechanical properties, a superplasticizer-to-binder ratio of 1.2% was applied, while for the specimens produced for AMBT tests, the ratio was 0.5%. These dosage levels were determined based on preliminary trial mixtures to ensure uniform workability across specimens.

2.1.4. Olive waste ash (OWA)

Olive mill solid waste was obtained from olive oil producers in the Gemlik region, dried, and subjected to calcination at two temperatures (400 °C and 600 °C) for two hours. Following the calcination process, a rapid-cooling method was applied, involving the extraction of the material from the furnace and immediately immersing it in room temperature water (15°C to 20°C). This quenching process was intended to increase the amorphous content of the ash, thereby enhancing its potential pozzolanic reactivity. The quenched ashes were subsequently transferred to a drying oven maintained at 105 °C for two days to remove residual moisture and ensure complete desiccation. Samples of OWA after calcination and the raw olive waste before calcination are shown in Figure 1.

Figure 1. Samples of OWA after calcination and the raw olive waste before calcination.

The calcined ash was then ground and sieved into three particle size ranges: <90 µm, 90–180 µm, and 180–360 µm. The specific gravity of OWA was measured as 2.18. The chemical composition of <90 µm fraction, which was identified in Phase I as the most effective particle size and subsequently used in Phase II, was determined by X-ray fluorescence (XRF) and is presented in Table 1. The reported chemical composition should be interpreted as representative of the chosen <90 µm fraction used in Phase II rather than all particle size ranges. The high K_2O content (29.13%) may influence ASR risk, while the low SO_3 level (0.26%) minimizes concerns about sulfate-related expansion.

2.2. Experimental Plan

The experimental plan was designed in two phases to systematically investigate the potential of OWA as an SCM in mortar production. The framework extended from raw material characterization to the final performance validation of mortars. In Phase 1, the effects of calcination temperatures and particle size distribution of OWA were investigated to determine its pozzolanic potential. Phase 2 focused on mortar production and testing, where mechanical properties, durability indicators, and microstructural features were evaluated to validate the performance of OWA as a partial cement substitute. Mix design of mortars prepared for mechanical tests followed British Standards Institution (2016), while the mix design of mortars produced for AMBT tests was carried out in accordance with ASTM International (2023a).

2.2.1. Phase I: Effect of calcination temperature and particle size of OWA

Mortar mixtures incorporating 20% OWA replacement were prepared to evaluate the effect of calcination temperature and particle size on the strength activity index (SAI). OWA was calcined at 400 °C and 600 °C for two hours, then sieved into three particle size ranges (<90 µm, 90–180 µm, and 180–360 µm). The mortars were prepared according to British Standards Institution (2016). Mixture codes were defined to indicate both calcination temperature and particle size (e.g., 400-90 for OWA calcined at 400 °C and sieved under 90 µm). The mix proportions of mortars are shown in Table 2.

Table 2. Mix design for Phase 1 productions

	Materials (g)				
Mix codes	Water	Sand	OPC	OWA	Superplasticizer
Control	225	1350	450	0	5.5
400-90	225	1350	360	90	5.5
400-180	225	1350	360	90	5.5
400-360	225	1350	360	90	5.5
600-90	225	1350	360	90	5.5
600-180	225	1350	360	90	5.5
600-360	225	1350	360	90	5.5

2.2.2. Phase II: Effect of varying amounts of OWA

Based on Phase I results, the optimum OWA condition was selected for further investigation. Mortars with varying replacement levels (5%, 10%, 15%, and 20% along with a control (C) specimen) were produced to assess mechanical performance, durability, and microstructural properties. Mechanical strength was evaluated through flexural and compressive tests, while durability was assessed by capillary water absorption and the AMBT for alkali–silica reactivity (ASR). Microstructural analyses using scanning electron microscopy (SEM) and X-ray diffraction (XRD) were conducted to clarify the mechanisms underlying OWA's pozzolanic activity. Mix proportions for mechanical and durability tests are presented in Table 3, while AMBT mixtures are shown in Table 4. Comparative EDS results of mortars are shown in Table 5.

Table 3. Mix design of mortars produced for mechanical and durability tests

	Materials (g)				
Mix codes	Water	Sand	OPC	OWA	Superplasticizer
C	225	1350	450	0	5.5
O-5	225	1350	427.5	22.5	5.5
O-10	225	1350	405	45	5.5
O-15	225	1350	382.5	67.5	5.5
O-20	225	1350	360	90	5.5

Table 4. Mix design of mortars produced for AMBT

	Materials (g)				
Mix codes	Water	Sand	OPC	OWA	Superplasticizer
C	206.8	990	440	0	2.5
O-5	206.8	990	418	22	2.5
O-10	206.8	990	396	44	2.5
O-15	206.8	990	374	66	2.5
O-20	206.8	990	352	88	2.5

Table 5. Comparative EDS results of mortars (wt%)

Element	C	O-5	O-10	O-15	O-20
O	54.3	48.1	62.7	59.7	52.0
Ca	32.7	38.3	33.8	27.7	6.5
Si	7.2	6.7	2.4	5.7	22.9
Mg	0.7	0.4	-	0.7	0.9
Na	-	-	-	0.6	-
Al	2.0	3.0	1.1	2.5	6.4
S	0.7	-	-	-	-
K	0.7	0.9	-	1.5	2.8
Fe	1.7	2.5	-	1.7	8.5

Figure 2. Mortar flow table test setup.

2.3. Test Methods

2.3.1. Fresh mortar tests

The workability of fresh mortar was evaluated using the flow table test in accordance with British Standards Institution (1999), and air content was determined according to ASTM International (2020). Representative images of the workability test setup are shown in Figure 2.

2.3.2. Mechanical properties tests

Mortar prisms measuring 40 × 40 × 160 mm were molded, cured, and tested at 28 days, which is the standard timeframe for assessing mechanical performance. Specimens were removed from the storage water, wiped to a surface-dry condition, and centered on the supports of the testing device (Figure 3). The load was applied at a specified rate, and the maximum load indicated by the testing machine was recorded. Flexural strength was determined according to British Standards Institution (2016). After flexural strength testing, half of the broken specimens were used for compressive strength tests, while the remaining halves were kept for capillary water absorption measurements. In addition, the strength activity index (SAI) was calculated following ASTM International (2022a). This index quantifies the pozzolanic activity of OWA by comparing the compressive strength of OWA-blended mortars to that of control mortars at specified curing ages.

Figure 3. Flexural testing of a mortar specimen.

2.3.3. Durability tests

ASR was evaluated using the AMBT in accordance with ASTM International (2023a). Mortar mixtures were prepared with a cement-to-aggregate ratio of 1:2.25 by mass and a water-to-cement ratio of 0.47. Rectangular mortar bars with dimensions of 25 × 25 × 285 mm were molded (Figure 4). After demolding, specimens were submerged in water at 23 ± 2 °C for at least 16 hours before initial length readings were taken. Subsequently, the bars were transferred to sealed containers filled with NaOH solution and stored in an oven at 80 ± 1 °C for 14 days. Length changes were measured at 0, 3, 7, and 14 days, and results were reported as percent expansion. According to ASTM International (2023b), aggregates were classified as non-reactive when expansion was <0.10%, moderately reactive at 0.10-0.30%, highly reactive at 0.30-0.45%, and very highly reactive when expansion was ≥0.45%.

Capillary water absorption of specimens produced in Phase II was measured according to British Standards Institution (2002) using the broken halves of specimens previously tested for flexural strength. The specimens were 35 days old at the time of testing. This test evaluates the ability of porous materials to absorb water through capillary action, which directly affects durability by influencing freeze–thaw resistance, susceptibility to chemical attack, and long-term strength. Mortar specimens were cut from smooth sides and sealed with impermeable material to ensure water ingress occurred only from the bottom surface (Figure 5). Measurements were taken at 10 and 90 minutes following the procedure outlined by British Standards Institution (2002).

Figure 4. AMBT specimens for measuring ASR expansion.

Figure 5. Mortar specimens during the capillary water absorption test.

2.3.4. Microstructural analyses

Microstructural analyses were performed on mortars produced in Phase II to investigate the influence of OWA incorporation on hydration products and pore structure. X-ray diffraction (XRD) was performed at the Prof. Dr. Adnan Tekin Material Sciences and Production Technologies Application Research Center (ATUM), Istanbul Technical University, to examine the mineralogical composition of powdered samples. The specimens were approximately three months old at the time of analysis. Analyses were performed using a Bruker D8 ADVANCE diffractometer. Five powdered specimens were prepared, and diffraction patterns were quantitatively evaluated using Malvern Panalytical software (ver. 5.2).

Scanning electron microscopy (SEM) was conducted at the Membrane Technologies Application-Research Center (MEM-TEK), Istanbul Technical University, to investigate the microstructural properties of hardened mortar mixtures at an age of approximately three months. SEM images were obtained from specimens previously subjected to flexural and compressive testing, as well as from additional samples reserved for microstructural examination. Mortar specimens were crushed into approximately 1 cm * 1 cm pieces, and at least four pieces from each mixture were prepared. Analyses were performed using a Zeiss EVO MA10 microscope. In the provided SEM figures, images were acquired at a magnification of 5,000×, corresponding to a horizontal field width of approximately 59.7 µm.

3. Results

3.1. Phase I Test Results

Fresh mortar tests indicated that both calcination temperature and particle size of OWA influenced workability and air content. Mixes incorporating OWA calcined at 600 °C generally exhibited better workability and lower air content compared to those calcined at 400 °C. Finer particles (<90 µm) tended to reduce workability and increase air content relative to coarser fractions as expected. Adjustments in superplasticizer dosage for subsequent phase were made based on the information obtained from these preliminary productions.

3.1.1. Strength activity index

The primary focus of Phase I was the Strength Activity Index (SAI), which measures the pozzolanic reactivity of OWA. Figure 6 presents the SAI values for mortar mixes with OWA replacements at different calcination temperatures and particle size distributions. Results demonstrate that both parameters significantly affected SAI values. Mortars with OWA calcined at 600 °C consistently achieved higher SAI values compared to those calcined at 400 °C, confirming that higher calcination temperatures enhance reactivity by decomposing organic matter and forming reactive silica phases.

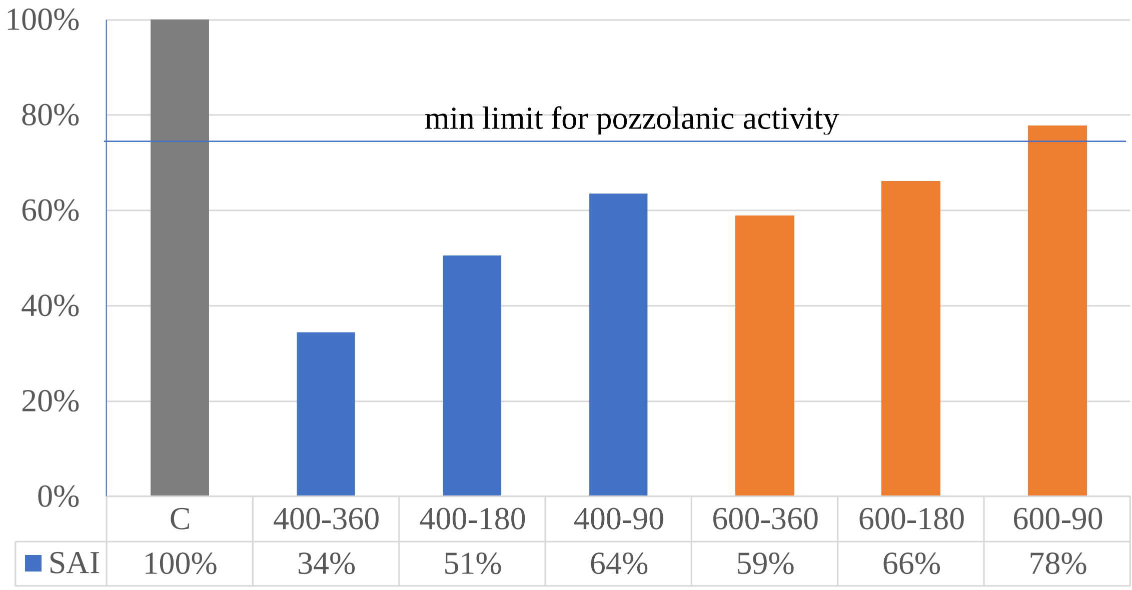

Figure 6. SAI values of mortars produced with OWA.

Particle size also played a critical role. Finer OWA fractions (<90 µm) increased SAI values, reaching 64% at 400 °C and 78% at 600 °C relative to the control mix. Coarser fractions (180–360 µm) exhibited lower SAI values due to reduced surface area and increased porosity. These findings are consistent with the commonly observed behavior of waste materials, where finer particles typically enhance pozzolanic activity and mechanical performance.

Importantly, among all mixtures, only the mix with OWA calcined at 600 °C and sieved below 90 µm surpassed the ASTM International (2022b) threshold of 75% of the control compressive strength. Therefore, the optimum calcination temperature and particle size of OWA were identified as these conditions, and used for subsequent mortar productions in Phase II.

3.2. Phase II Test Results

Based on the findings of Phase I, OWA calcined at 600 °C and ground to a particle size below 90 µm was identified as the optimum condition. In Phase II, mortars incorporating varying replacement levels of OWA (5%, 10%, 15%, and 20%) were produced. These mixes were systematically evaluated in terms of their fresh properties, mechanical performance, durability characteristics, and microstructural features, providing a comprehensive assessment of OWA's suitability as an SCM.

3.2.1. Fresh mortar properties

Fresh mortar tests conducted with varying replacement levels of OWA provided valuable insights into the material's influence on workability and air content. A clear trend was observed in both parameters as the OWA content increased, and the results are given in Figure 7.

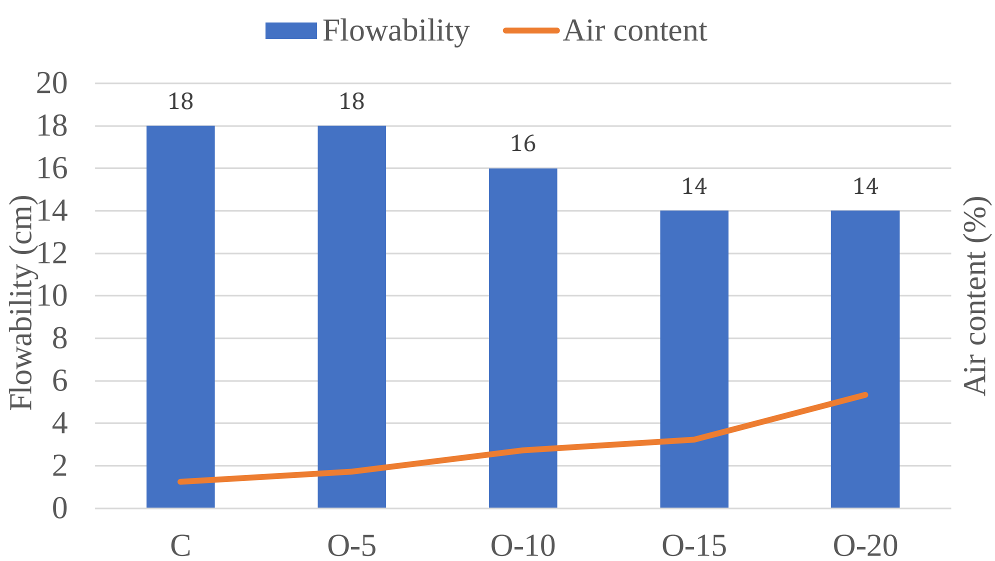

Figure 7. Fresh mortar tests (flowability and air content) for mortars produced for mechanical tests.

Regarding workability, the control mix and the O-5 mix exhibited comparable values, both maintaining a flow of 18 cm. However, with higher OWA replacement levels (O-10, O-15, and O-20), a gradual reduction was recorded, with values decreasing to 16, 14, and 14 cm, respectively. This decline can be attributed to the introduction of OWA particles, which alter the consistency of the mortar and reduce its ease of handling. Nevertheless, acceptable workability was still achieved even at 20% replacement through the use of a superplasticizer.

Air content displayed a consistent increase with higher OWA replacement percentages, as expected. The control mix demonstrated the lowest air content at 1.51%, while the O-20 mix reached the highest value at 5.34%. This upward trend suggests that OWA incorporation introduces additional void spaces within the mortar matrix, leading to greater entrapped air volumes. These voids may arise from the physical characteristics of OWA particles or their interaction with other constituents in the mix. These findings align with the observations of Al-Akhras and Abdulwahid (2010), who reported that increasing OWA content in mortars reduces workability while simultaneously increasing air content, thereby confirming the consistency of observed trends.

3.2.2. Mechanical properties

The mechanical performance of mortars incorporating OWA was evaluated with flexural and compressive strength tests. Mortar mixes were prepared with varying OWA replacement levels (O-5, O-10, O-15, and O-20) alongside the control mix (C=0% OWA), and tested at 28 days under identical curing conditions. Flexural strength test results, together with normalized values relative to the control mix (C), are given in Figure 8, while compressive strength results are shown in Figure 9.

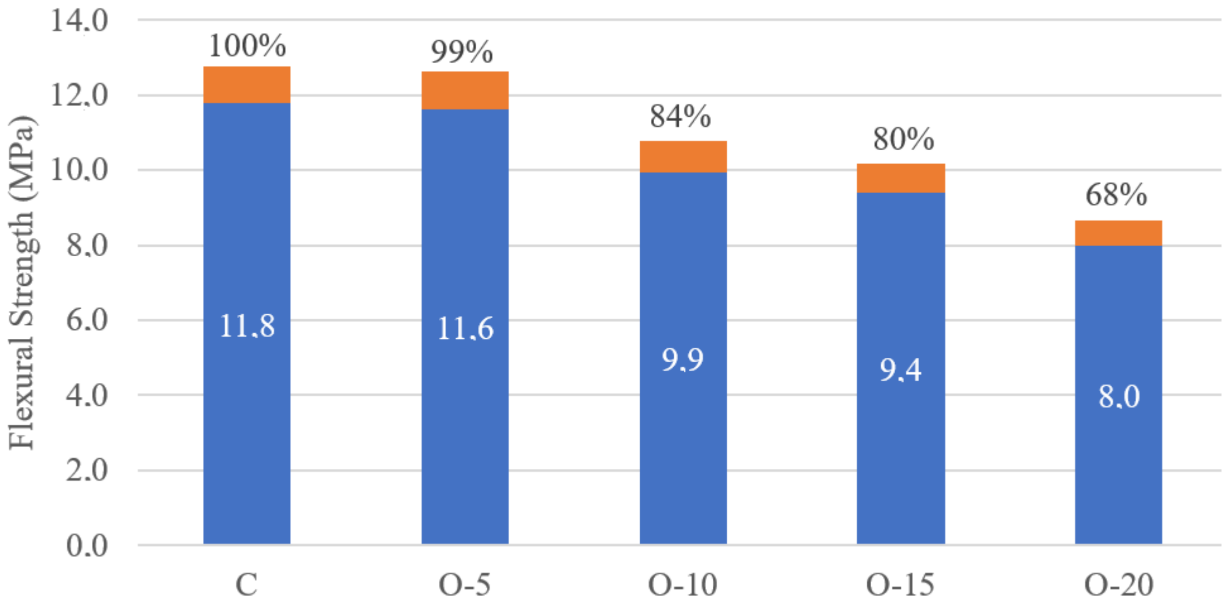

Figure 8. Flexural strength test results.

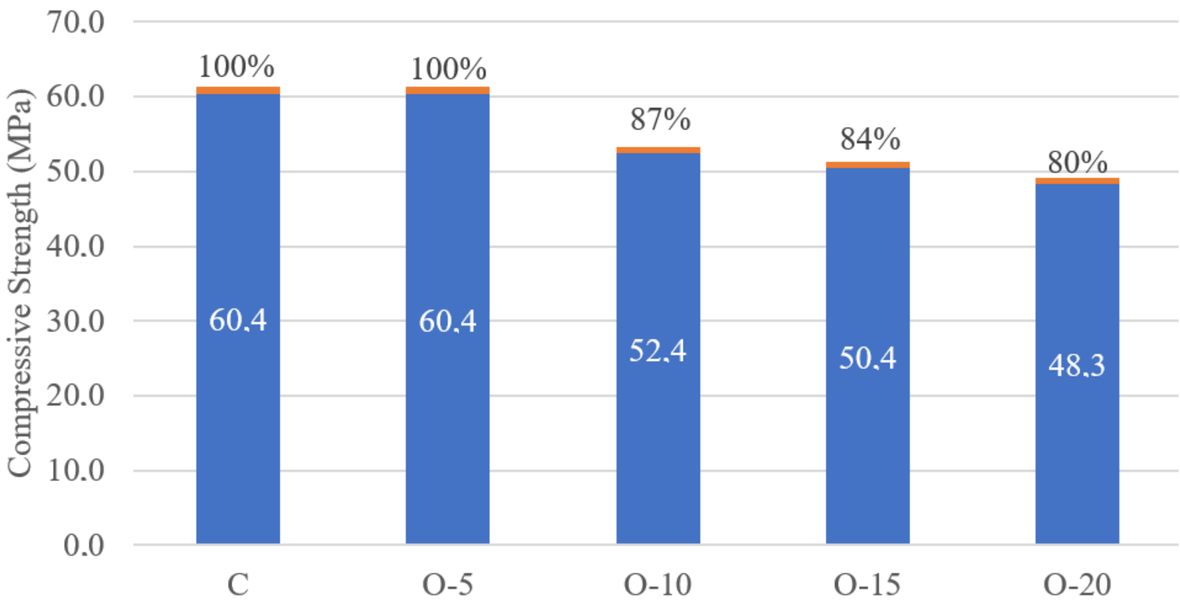

Figure 9. Compressive strength test results.

Flexural strength results demonstrated a gradual reduction with increasing OWA content. Normalized values relative to the control mix were 99% for O-5, 84% for O-10, 80% for O-15, and 68% for O-20. Similarly, compressive strength results followed a similar trend: the control mix exhibited 60.4 MPa strength, while normalized values for O-5, O-10, O-15, and O-20 were 100%, 87%, 84%, and 80%, respectively. Even at the highest replacement level (20%), compressive strength remained within acceptable ranges, confirming the feasibility of OWA incorporation.

The observed reductions in strength are attributed to the partial substitution of cement with OWA, which decreases cement content and alters hydration dynamics. Nevertheless, the results demonstrate that OWA can be effectively utilized as an SCM without compromising performance at moderate replacement levels.

In conclusion, the integration of OWA into mortar mixes offers a promising pathway for reducing environmental impact in the construction industry. Mortars containing up to 15% OWA exhibited mechanical properties suitable for practical applications, while higher replacement levels showed acceptable though reduced performance. Careful optimization of particle size and calcination temperature maximizes OWA's reactivity, enabling sustainable cement substitution while maintaining satisfactory mechanical properties. Further evaluation of long-term performance is recommended for future studies.

3.2.3. Durability performance

3.2.3.1. Accelerated mortar bar tests

Since OWA contains a relatively high amount of K_2O (29.13%), which is known to contribute to alkali–silica reaction (ASR) in cementitious systems, accelerated mortar bar tests (AMBTs) were conducted to evaluate whether its incorporation aggravates or mitigates ASR expansion. The results are presented in Figure 10. At early ages (3 d), all mixes—including the control—exhibited negligible expansion. As the test progressed to day 14, expansion increased across all mixes, but values remained comparable to the control, indicating a limited positive effect of OWA on ASR mitigation. For example, expansion was 0.33% for the control specimen (C) and reduced to 0.23% for O-20. Across all replacement levels (5–20%), no significant deviation in ASR expansion was observed compared to the control.

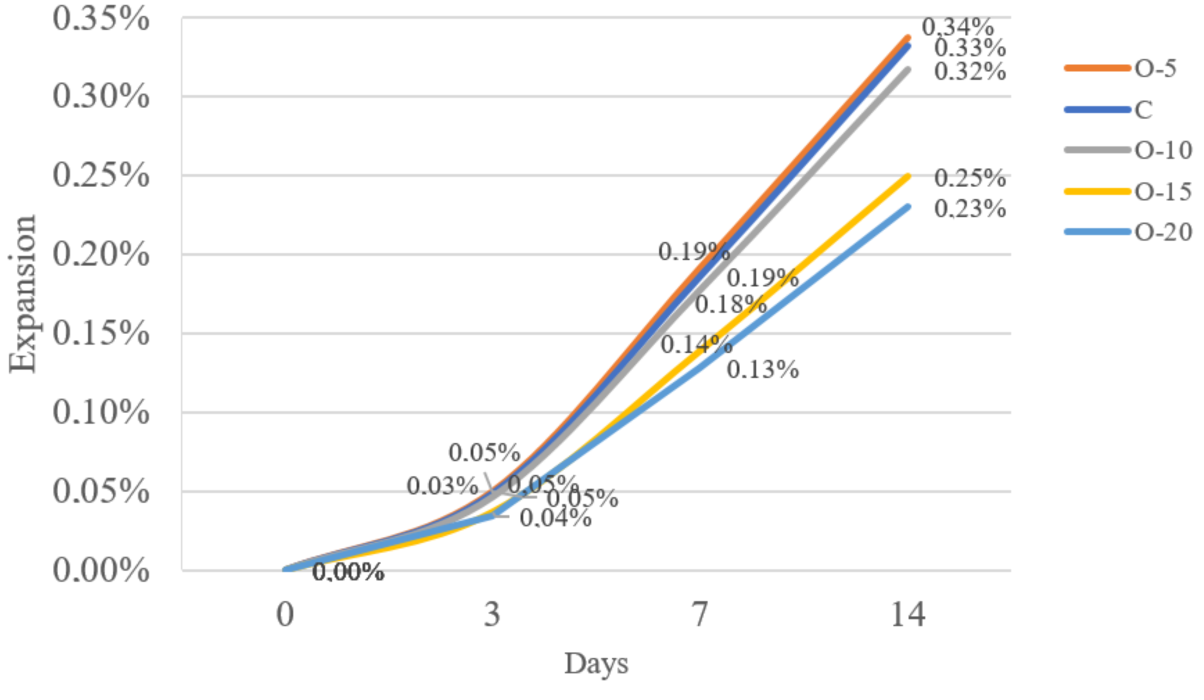

Figure 10. AMBT results for mixes produced with OWA calcined at 600 °C.

Mortars with higher OWA content exhibited slightly lower expansion rates compared to the control, particularly at later ages (days 7–14). This suggests that the incorporation of carefully processed OWA may contribute to lowering ASR-related expansions. Overall, ASR expansion was reduced from highly reactive to moderately reactive levels with increasing OWA content, consistent with the findings of Al-Akhras (2012), who reported that OWA incorporation does not exacerbate ASR and may even contribute to slight reductions in expansion despite its very high alkali content. Further research is needed to clarify the underlying mechanisms and optimize OWA production parameters for enhanced mitigation of ASR expansion in concrete and mortar applications. Additionally, long-term durability studies are essential to validate the performance of OWA-incorporated mixes under real-world conditions.

3.2.3.2. Capillary water absorption tests

Capillary water absorption tests were performed on hardened mortars with varying OWA replacement levels (0–20%). The results, obtained according to British

Standard Institution (2002), are presented in Figure 11. The control mix exhibited the lowest water absorption coefficient (0.043 kg/(m²·min$^{0.5}$), while values increased progressively with OWA content: 0.063 for O-5 and O-10, 0.067 for O-15, and 0.080 for O-20. This increase is attributed to the higher porosity introduced by OWA particles, which create additional voids and pathways for water ingress. As the OWA content increases, so does the volume of these pores, leading to enhanced water absorption capacity. While this behavior raises concerns for long-term durability, the coefficient of water absorption values remained within the general range reported for other SCMs, albeit following a different trend. For instance, Thiedeitz et al. (2022) demonstrated that RHA mortars develop a denser microstructure and reduced capillary suction at comparable levels, reflecting the high silica reactivity of RHA. Similarly, Shaladi et al. (2022) showed that palm oil fuel ash (POFA) treated at 600 °C exhibited reduced porosity and lower water absorption, consistent with its enhanced pozzolanic activity. In contrast, OWA—with its lower silica and higher alkali content—exhibited a progressive increase in absorption with replacement level, highlighting the importance of chemical composition in governing durability performance. This divergence in trends underscores that while OWA values remain comparable in magnitude to those of other SCMs, its distinct chemistry leads to different absorption behavior.

These findings highlight the need for a balanced approach when incorporating OWA into mortar mixes. Moderate replacement levels (≤15%) appear to balance mechanical performance and water absorption behavior, whereas higher replacement levels may necessitate additional measures to control porosity and moisture ingress. In conclusion, OWA incorporation provides sustainability benefits and acceptable durability performance, but careful consideration of replacement levels is required.

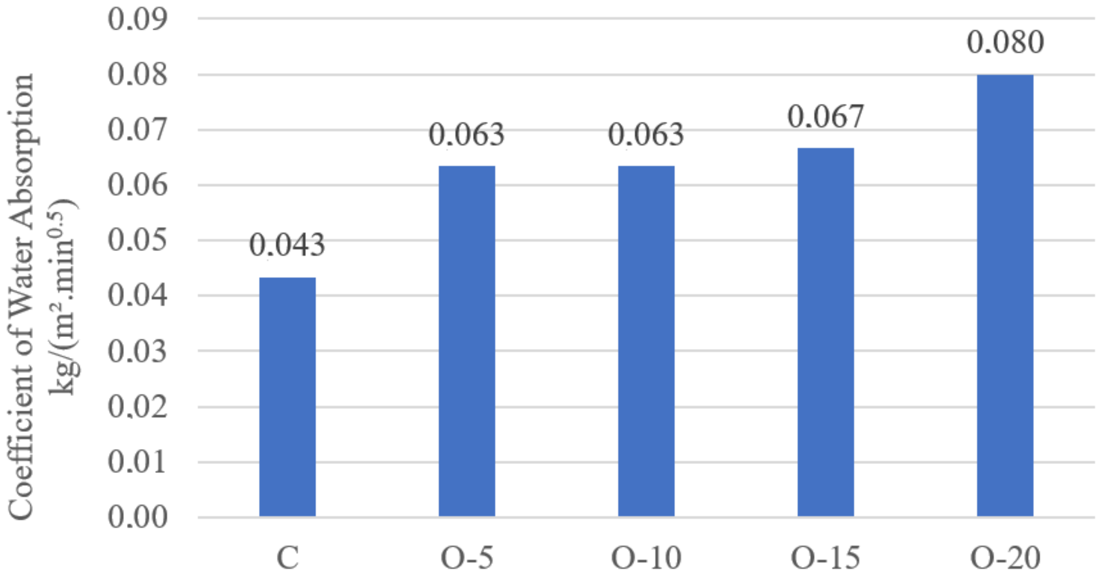

Figure 11. Coefficient of capillary water absorption as per British Standards Institution (2002).

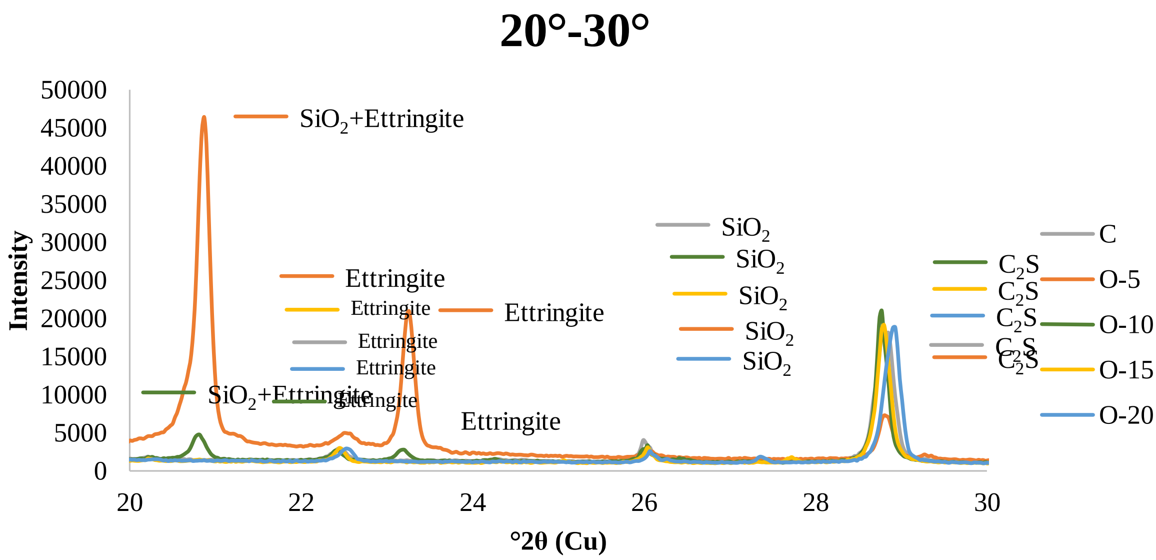

Figure 12. XRD results for mixtures between 20°-30° 2θ.

3.2.4. Microstructural analyses

3.2.4.1. X-Ray diffraction analysis

X-ray diffraction (XRD) tests were conducted on mortar mixes containing varying percentages (0-20%) of OWA calcined at 600 °C for 2 hours as partial cement replacement. The diffraction patterns were analyzed across the 2θ range of 20°–50°, with key phase contributions grouped into three intervals: 20°–30°, 30°–40°, and 40°–50°, as shown in Figures 12–14.

In the 20°–30° 2θ range, the strongest reflections were associated with quartz (SiO_2) and ettringite. A broad hump between 29° and 32° was attributed to poorly crystalline calcium silicate hydrate (C–S–H), which appeared as an amorphous background rather than sharp reflections. In the 30°–40° 2θ range, the broad signal between 30.5° and 32° reflected amorphous C–S–H overlapped by contributions possibly from belite (C_2S) and calcite ($CaCO_3$). Peaks around 35.5° corresponded to tricalcium aluminate (C_3A) and with some dicalcium aluminate (C_2A) polymorphs, while quartz contributed a secondary peak near 39°. In the 40°–50° 2θ range, CH showed medium-intensity reflections at 47.0° and 50.0°, while calcite overlapped with CH around 47.5–48.0°, complicating phase attribution. Quartz was visible near 43°, and C_3A contributed weak reflections at angles of 41–42°.

Across all mixes, CH was consistently detected, but its peak intensity generally decreased with increasing OWA content. This reduction indicates progressive CH consumption via pozzolanic reactions, motivated by OWA's silica (20.19%) and alumina (2.28%) combined with its relatively low CaO (21.90%). The consumption of CH was accompanied by the formation of secondary C–S–H and C–A–S–H gels, which displayed as intensified amorphous signals between 29° and 32°. OWA's exceptionally high alkali content (K_2O 29.13%, Na_2O 6.66%) did not appear as discrete peaks in XRD, because these oxides dissolve rapidly in the pore solution as KOH and NaOH. The presence of KOH in particular raises the pore solution pH, which accelerates the dissolution of silica and alumina phases and thereby enhances secondary C–S–H formation. Although KOH itself is not visible in XRD due to its solubility, its chemical effect is important to the observed reduction in CH and the growth of amorphous gel phases. In addition, part of the alkalis can crystallize as alkali sulfates such as K_2SO_4, which do appear in XRD near 30–32° and 43–44°. These alkalis also influence the stability of ettringite and AFm phases. Phases such as ettringite, $CaCO_3$, and C_3A remained relatively stable across all mixes, indicating that OWA incorporation primarily influenced the CH and C–S–H balance rather than altering early hydration or carbonation-related phases. While enhanced secondary C–S–H formation is beneficial for microstructural densification and strength development, the decline in mechanical performance at higher replacement levels (O-15 and O-20) suggests dilution effects or the formation of less effective hydration products.

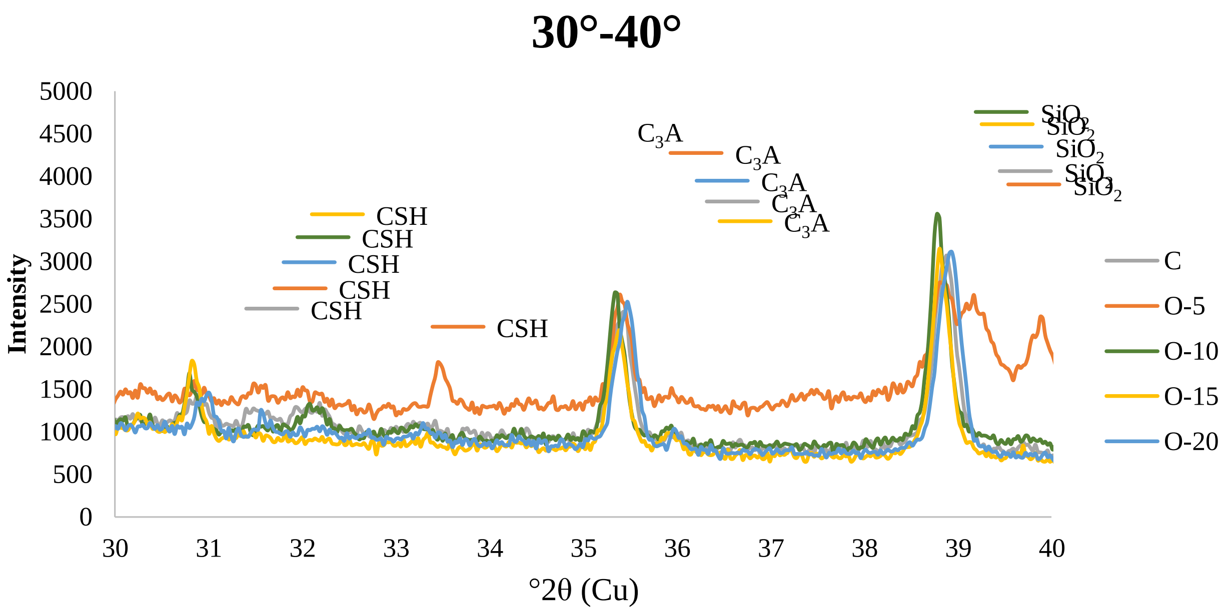

Figure 13. XRD results for mixtures between 30°-40° 2θ.

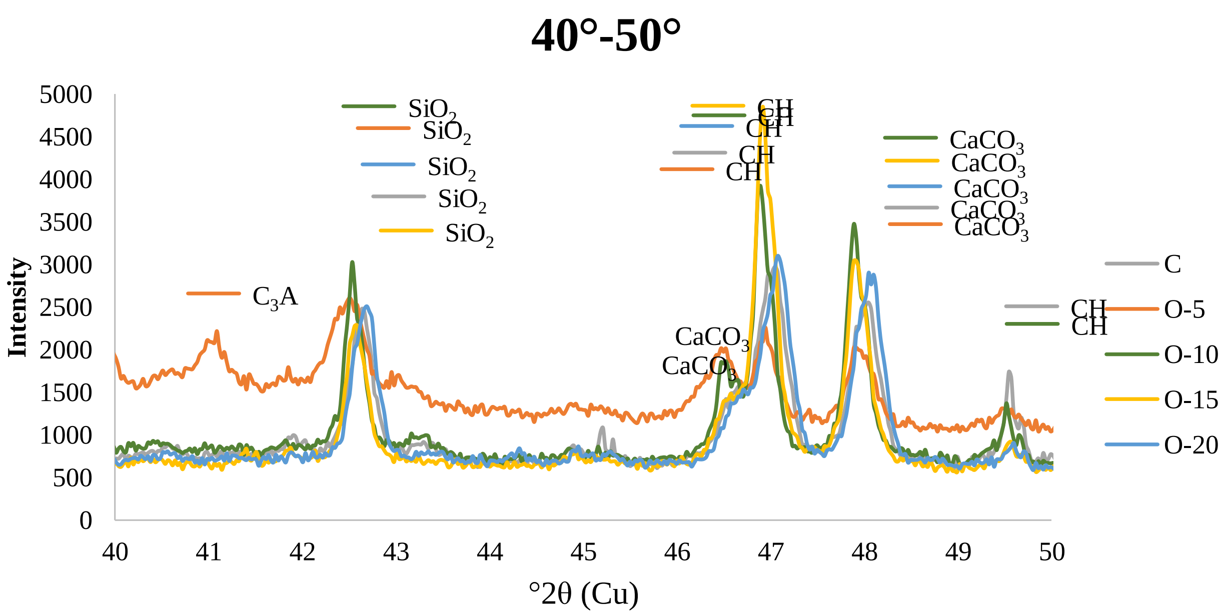

Figure 14. XRD results for mixtures between 40°-50° 2θ.

3.2.4.2. Scanning electron microscopy analysis

SEM imaging was performed to examine the microstructural evolution of mortar mixes incorporating varying percentages of OWA calcined at 600 °C. The images of all five mixes acquired at 5000x magnification are presented in Figure 15. Although lower-magnification (1000×) scans were also available, they were not included due to insufficient clarity. Arrows indicate calcium hydroxide (CH) crystals in Figure 15-a, and the surrounding amorphous matrix is interpreted as calcium silicate hydrate (C–S–H) gel. While the visual interpretation of figures was difficult to assess, corresponding quantitative EDS results are also provided in Table 4 to better explain the SEM results.

The control mix (C) without OWA displayed the typical morphology of Portland cement hydration products, with CH plates surrounded by fibrous C–S–H serving as a baseline for comparison. No ettringite was visible. EDS confirmed Ca (32.7 wt%) and O (54.3 wt%) dominance, consistent with portlandite and C–S–H phases.

At 5% OWA replacement (O-5), subtle morphological changes were observed, including denser C–S–H regions. This suggests that the pozzolanic reaction of OWA consumed part of the CH, leading to secondary C–S–H formation. EDS analysis supported this, showing Ca enrichment (38.3 wt%) increased Al and Fe contributions, consistent with ash incorporation.

At 10% replacement (O-10), CH reduction became more evident, with EDS confirming Ca-rich, Si-poor regions (Ca 33.8 wt%, Si 2.4 wt%), typical of portlandite, while the surrounding matrix showed elevated O signals (62.7 wt%). This indicates that CH remained prevalent, although pozzolanic activity was underway.

At 15% replacement (O-15), CH content diminished further (Ca 27.7 wt%), and C–S–H regions expanded, reflecting sustained CH consumption and enhanced pozzolanic activity. EDS spectra revealed the appearance of Na and K signals, pointing to ash-derived phases entering the matrix. At 20% replacement (O-20), CH was scarcely detected, and the microstructure was dominated by C–S–H/C–A–S–H phases enriched in Si (22.9 wt%) and Al (6.4 wt%), alongside ash-derived inclusions containing K, Fe, and Mg. The sharp decline in Ca (6.5 wt%) and the rise in Fe (8.5 wt%) highlight the transformation of the matrix into ash-rich hydration products, likely due to the presence of high OWA.

Across all replacement levels, SEM/EDS observations revealed a progressive reduction in CH and a corresponding increase in C–S–H, consistent with enhanced pozzolanic reactivity. While this trend is favorable for strength and durability, microstructural factors such as porosity, microcrack formation, and ash particle distribution must also be considered. Excessive replacement (e.g., 20%) may introduce localized weaknesses despite the overall increase in C–S–H.

In summary, SEM/EDS analysis confirms that increasing OWA content promotes late pozzolanic reactivity, evidenced by diminishing CH and expanding C–S–H phases. This microstructural evolution supports the potential for improved mechanical properties and durability, provided that OWA is well integrated into the cement matrix and does not introduce excessive porosity or microstructural heterogeneity.

It should be noted that SEM/EDS observations are inherently localized and reflect specific regions within the sample. While the presented images illustrate consistent trends in CH reduction and C–S–H development, microstructural variability across different areas may influence the interpretation and should be considered when generalizing these findings.

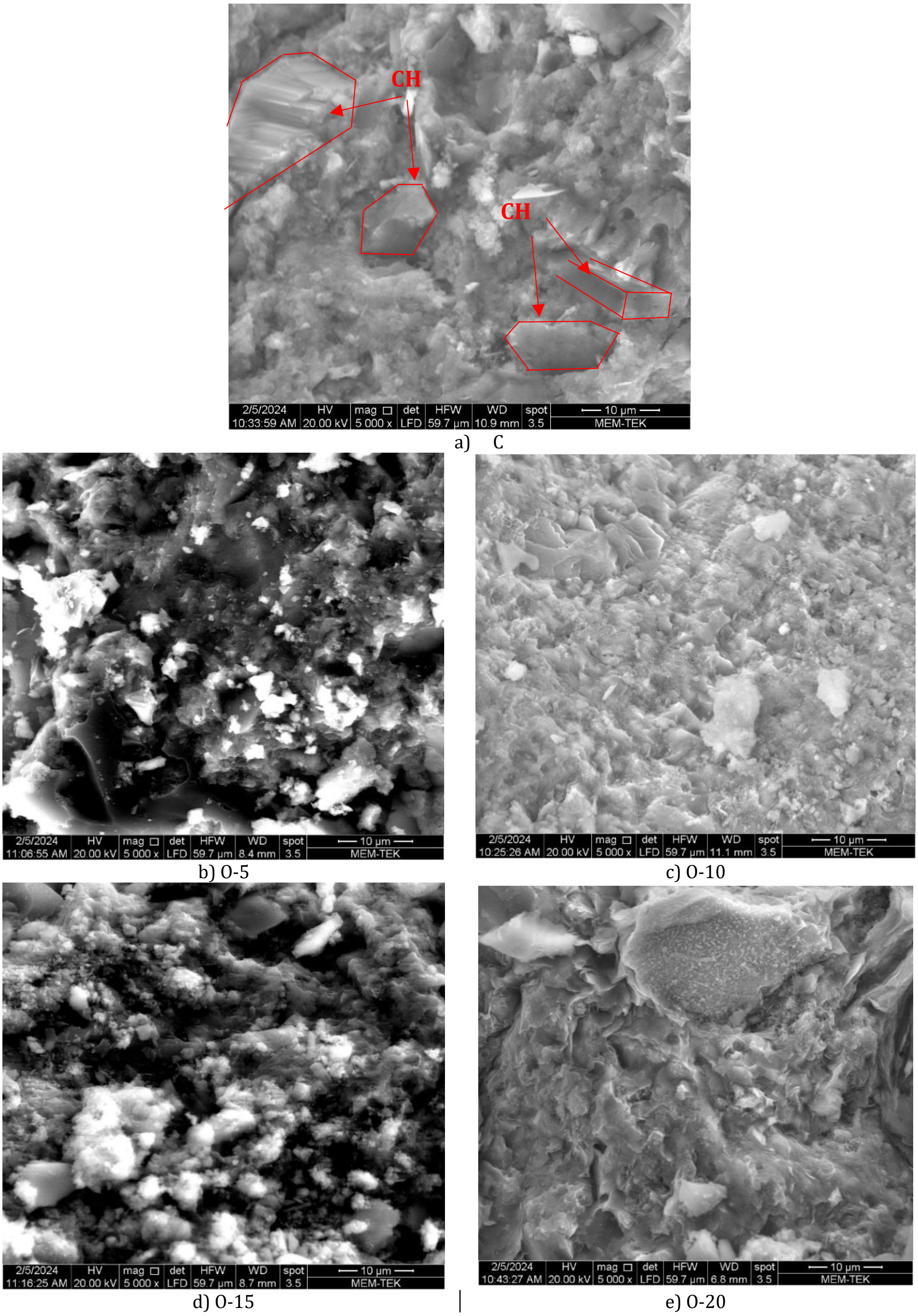

Figure 15. SEM images of mortar mixes with varying OWA replacement levels a) C b) O-5 c) O-10 d) O-15 e) O-20.

4. Discussion

As this was the first study using OWA from the Gemlik region of Türkiye, Phase I was intentionally limited to identifying suitable processing conditions. The primary objective of this phase was to determine a mix that surpassed the ASTM threshold for strength activity index (SAI), which was achieved only under specific conditions (600 °C and <90 µm). In phase II, the mechanical, durability, and microstructural results demonstrated that OWA can be effectively utilized as an SCM when processed under optimized conditions. The observed reduction in CH and the parallel increase in CSH confirm the pozzolanic reactivity of OWA, which contributes to strength development and long-term durability. These findings are consistent with previous studies on agricultural waste ashes, such as RHA and POFA, which similarly reported enhanced pozzolanic activity and strength gains when processed under controlled conditions (Al Akhras, 2012; Aburawi & Al Madani, 2018). This reinforces the potential of OWA to reduce cement consumption and mitigate environmental impact through valorization of waste materials.

Nevertheless, the balance between mechanical strength and durability requires careful consideration. While moderate replacement levels enhance microstructural density and performance, higher OWA contents may lead to increased porosity or microstructural incompatibilities that reduce the benefits of additional C–S–H formation. This highlights the importance of identifying an optimum replacement level that maximizes pozzolanic activity without compromising strength and durability.

It should also be noted that the microstructural examinations in this study were primarily based on SEM/EDS and XRD analyses. While these techniques provided valuable insights into CH consumption and C–S–H development, complementary methods such as thermogravimetric analysis (TGA) and Fourier-transform infrared spectroscopy (FTIR) could further clarify the mechanisms of hydration and pozzolanic reactions. In addition, although the high potassium content of OWA suggests potential effects on setting time, direct setting time measurements were not performed in this study. These limitations have been acknowledged, and future research should incorporate TGA, FTIR, and setting time tests to provide a more comprehensive understanding of OWA's reactivity, phase evolution, and early-age performance.

In summary, SEM/EDS and XRD analyses confirm that increasing OWA content promotes late pozzolanic reactivity, evidenced by diminishing CH and expanding C–S–H phases. This microstructural evolution supports the potential for improved mechanical properties and durability, provided that OWA is well integrated into the cement matrix. Future studies combining mechanical, durability, and advanced microstructural techniques will be essential to establish standardized processing and application guidelines for OWA as a sustainable SCM.

The comprehensive analysis of experimental results regarding the incorporation of OWA in mortar mixes provides valuable insights into its potential as an SCM for enhancing sustainability. Examination of particle size distribution, calcination temperature, OWA content, and their effects on mechanical properties, durability, and microstructural characteristics reveals several outcomes, and key conclusions can be drawn as follows:

Both calcination temperature and particle size significantly influenced OWA's pozzolanic reactivity. Mortars incorporating OWA calcined at 600 °C and ground to finer particles (<90 μm) exhibited improved pozzolanic activity and better mechanical performance compared to coarser fractions and lower calcination temperatures. Finer particles and higher calcination temperatures contribute to more effective pozzolanic reactions and denser microstructures.

In the second phase of the study, varying proportions of OWA (0%, 5%, 10%, 15%, and 20%) were used to replace traditional cement in mortar mixes. The results showed that flexural strengths ranged from 99% of the control at 5% replacement (O-5) to 68% at 20% replacement (O-20), while compressive strengths ranged from 100% of the control at 5% replacement (O-5) to 80% at 20% replacement (O-20). Although higher strength reductions were observed at higher replacement levels, the mixes maintained satisfactory mechanical performance within acceptable ranges.

AMBT results indicated mitigation of the ASR expansion with OWA addition. Expansions reduced from 0.33% (for control) to 0.23% at 20% replacement (O-20) by day 14. Capillary water absorption increased with OWA content due to its higher porosity, but the values remained comparable to those of other agricultural waste ashes. These findings highlight the need for further optimization of OWA processing conditions and exploration of potential additives to enhance long-term durability.

Microstructural analyses provided deeper insights into the chemical and morphological changes induced by OWA incorporation. XRD patterns revealed reduced calcium hydroxide (CH) peak intensities and increased calcium silicate hydrate (C–S–H) content, confirming active pozzolanic reactions in OWA-containing mixes. SEM images verified these findings, showing progressive CH reduction and denser C–S–H regions with increasing OWA content, thereby exhibiting enhanced reactivity and improved microstructural density.

Overall, the integration of OWA as an SCM in mortar mixes demonstrates strong potential for sustainable construction practices, offering the dual benefits of reducing cement consumption and valorizing agricultural waste. When particle size, calcination temperature, and replacement levels are carefully optimized, OWA can maximize pozzolanic reactivity while minimizing drawbacks such as increased porosity. This contributes to lowering the environmental footprint of construction materials while maintaining acceptable performance. However, further investigations into long-term durability, variability in ash composition, large-scale applications, and life-cycle assessments are necessary to further validate its practical viability.

Author Contributions

The percentages of the authors' contributions are presented below. All authors reviewed and approved the final version of the manuscript.

	K.E.	Ö.E
C	50	50
D	50	50
S	0	100
DCP	80	20
DAI	80	20
L	80	20
W	50	50
CR	20	80
SR	20	80
PM	50	50
FA	100	0

C= concept, D= design, S= supervision, DCP= data collection and/or processing, DAI= data analysis and/or interpretation, L= literature search, W= writing, CR= critical review, SR= submission and revision, PM= project management, FA= funding acquisition.

Conflict of Interest

The author declared that there is no conflict of interest.

Ethical Consideration

Ethics committee approval was not required for this study because of there was no study on animals or humans.

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