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# Characterization and Optimization of Olive Pomace Ash as a Partial Cement Replacement

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**ABSTRACT**

In this study, calcined and ground olive pomace ash (OPA) was evaluated as a supplementary cementitious material for the development of blended cements. Portland cement (CEM I 42.5) was partially substituted with OPA at replacement levels of 10%, 20%, and 30% by weight, while a reference mix containing 0% of OPA was used for comparison. Experimental investigations, conducted in accordance with Algerian and European cement standards, included chemical, physical, mechanical, and durability-related tests. The results show that OPA incorporation increases the water demand for normal consistency and reduces the workability of standardized mortars. These effects are mainly attributed to the high capacity of OPA particles to absorb water. The setting times were slightly accelerated. Moreover, cements containing 10% of OPA exhibited significant strength gains exceeding 20 MPa between 28 and 60 days, highlighting the beneficial pozzolanic reactivity of the additive, as supported by XRD and FTIR results. This demonstrates the potential use of OPA as a partial cementitious material.

## 1. INTRODUCTION

The valorization of biomass ashes has attracted significant research interest owing to their pozzolanic and/or hydraulic activity [1-4]. These ashes include wood ash, rice husk ash, coal ash, sugar cane bagasse ash, palm ash, and olive pomace ash.

The use of biomass ashes is gaining attention due to their multiple benefits [5, 6]. These include a decrease in the exploitation of natural resources and energy needed for the manufacturing of cement, a decrease in the ecological impacts associated with uncontrolled ash disposal, and the ability to retain potentially toxic elements.

From a structural standpoint, the replacement of ordinary portland cement with biomass ashes helps to lower the heat of hydration [7]. It also mitigates the occurrence of concrete cracking [7, 8] and ensure satisfactory compressive strength values [9].

Olive oil mill operations generate significant quantities of by-products called olive pomace. After calcination, this waste can be used in the construction sector as building materials, particularly in the cement and concrete industry.

Pomace is the solid waste generated from the initial pressing of fresh olives, consisting mainly of pulp and pits. It constitutes about one-third of the mass of crushed olives. It and typically contains 28.5% water, 41.5% shell, 21.5% pulp, and 8.5% oil. While part of this waste is traditionally valorized for secondary oil extraction or soap production, a considerable amount remains underutilized. Olive production is seasonal (October–December). However, this waste is thrown out in abundance all year round.

Several studies have examined the introduction of OPA in cementitious systems.

Tayeh et al. [10] investigated the use of olive ash in concrete production as a partial substitute for cement. Slump and compressive strength tests at 7, 28 and 90 days were conducted using various percentages of olive ash (0% to 15%) on fresh and hardened concrete. The durability of the mixture was evaluated in a solution containing 5% NaOH and $MgSO_4$ by weight of water. High-temperature resistance was assessed using concrete cubes exposed to temperatures up to 170℃. The findings indicate that increasing the proportion of olive ash leads to reduced compressive strength and workability. However, durability and thermal resistance are enhanced.

Dahim et al. [11], in their research, replaced a part of cement in concrete from 2.5% to 12.5% with an increase of 2.5%. The findings show that the incorporation of olive ash leads to a reduction in concrete workability. This is evidenced by a decrease in slump as the ash content increases. Mechanical strength and durability improve progressively with higher olive ash levels up to an optimum of 7.5%. Beyond this value, strength begins to decline. Therefore, a 7.5 replacement of cement was identified as the optimal dosage in this study. This percentage could produce concrete with superior strength and durability compared to the reference concrete mix. Olive waste ash improves both strength and durability by reducing the effective water-cement ratio in the concrete mix, densifies the void and pore structure in cementitious materials.

Cuenca et al. [12] reported that the incorporation of fly ash from the combustion of olive pomace grains as a filler in self-compacting concrete, gives satisfactory fresh properties and acceptable mechanical performance.

Al-Akhras et al. [13] indicated that the incorporation of olive pomace ash up to 22% as a sand replacement in concrete improves its thermal cycling resistance: few cracks and reduced structural degradation compared with plain concrete after exposure to temperature cycles between 30 and 150°C.

This study contributes to the valorization of agro-industrial by-products while addressing the environmental impact of construction materials. It explores the partial replacement of clinker by OPA as a means of improving the sustainability of cementitious binders and reducing $CO_2$ emissions linked to clinker production. It examines the chemical, physico-mechanical, and durability properties of blended cements containing 10%, 20%, and 30% OPA. The aim is to determine the optimal substitution level and promote the sustainable use of this agro-industrial residue.

## 2. MATERIALS AND METHODS

### 2.1 Raw materials

Portland cement (CEMI 42.5). Its chemical composition determined by X-ray fluorescence (XRF) analysis, reveals 63.96% CaO, 5.06% $Al_2O_3$, 3.70% $Fe_2O_3$, 19.86% $SiO_2$, 2.30% MgO, 0.71% $K_2O$, 0.28% $Na_2O$, 3.09% $SO_3$ and a loss on ignition (LOI) of 2.25%. Its absolute and apparent densities are 3.15 g/cm$^3$ and 0.963 g/cm$^3$, respectively.

The standard sand used in this study complies with the requirements of EN 196-1. It is a siliceous sand containing more than 98% $SiO_2$. Its absolute and apparent densities are approximately 2650 Kg/m$^3$ and 1600 Kg/m$^3$, respectively.

The olive pomace used in this study was collected from the Guelma region in northeastern Algeria.

To prevent microbial growth, the raw material was air-dried under ambient conditions. It was then calcined at around 480°C for 2 hours in an earthenware vessel until ash formation, followed by grinding and sieving through a 100µm mesh. The OPA thus obtained has an absolute and apparent densities of 1440 Kg/m$^3$ and 441 Kg/m$^3$, respectively.

It is also characterized by Fourier-transform infrared spectroscopy (FTIR) to analyze functional groups while mineral phases were identified by X-ray diffraction (XRD).

The FTIR spectrum of olive pomace ash (Figure 1) exhibits characteristic bands associated with both inorganic and residual organic phases. A broad absorption around 3230 cm$^{-1}$ is assigned to O-H stretching [14], related to adsorbed water or surface hydroxyls of silicate and aluminate phases. Weak shoulders near 2900-2850 cm$^{-1}$ (bands at 2921 and 2852 cm$^{-1}$) are due to symmetric and asymmetric CH stretching vibrations (νCH, alkyl groups) [15], indicating traces of unburnt carbonaceous matter.

The band at 1571 cm$^{-1}$ is assigned to the stretching modes of lactone and carbonyl functionalities associated with C=C bands in aromatic ring.

The band around 1160 cm$^{-1}$ is attributed to the C-O stretching in acids or ester groups; it corresponds to oxidized carbons [15]. The bands about 1035 cm$^{-1}$ are assigned to Si-O-Si and Si-O-Al stretching vibrations, confirming the presence of silicate phases. This indicates the potential pozzolanic reactivity of the ash [16]. The band at 866 cm$^{-1}$ is attributed to C-O vibration in carbonate groups. Finally, the bands observed in the 800–500 cm$^{-1}$ region is attributed to Si-O bending modes, consistent with mineral oxides and aluminosilicate structures [17].

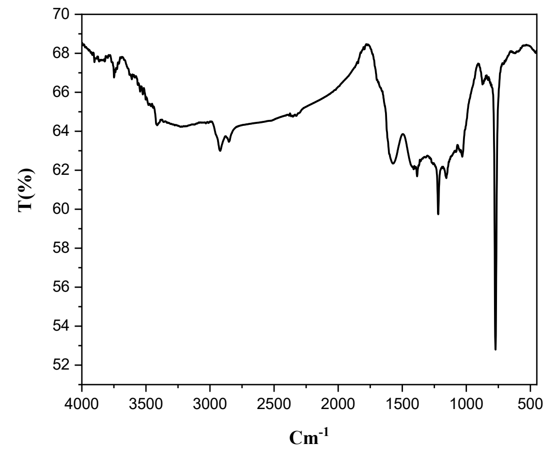


**Figure 1.** Infrared spectrum of the olive pomace ash used

The XRD pattern of the calcined olive pomace ash is presented in Figure 2. Between 15° and 35° (2θ), a large hump was observed indicating the presence of an amorphous aluminosilicate. This feature is favorable for pozzolanic reactivity [16].

In addition, some crystalline peaks were identified. The main diffraction peak at 2θ ≈ 28.26° corresponds to quartz ($SiO_2$) [18]. The smaller peak at 2θ ≈ 40.40° can be attributed to residual calcium oxide (CaO) or aluminosilicate phases, as reported in previous studies on biomass ashes [18, 19].

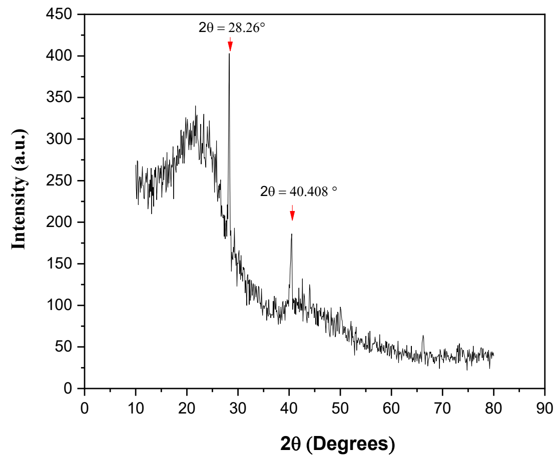


**Figure 2.** X-ray diffraction pattern of the calcined olive pomace ash

The coexistence of both amorphous and crystalline phases suggests that the olive pomace ash possesses a good pozzolanic potential.

The FTIR and XRD results demonstrate that olive pomace ash contains reactive silica, along with carbonate phases and residual organics. This finding support its suitability as a supplementary cementitious material for sustainable binder formulations. These results are consistent with those reported in the literature [6, 19].

### 2.2 Cement preparation

The obtained OPA was subsequently employed as a partial replacement for Portland cement (CEM I 42.5) at substitution levels of 10%, 20%, and 30% by weight.

The choice of these substitution levels was based on previous research conducted on supplementary materials.It also considered the need to remain within the typical replacement range used in the blended cement CEM II.

After batching, the cementitious mixtures were thoroughly homogenized. They were then subjected to physicochemical and mechanical characterization in accordance with the relevant international standards.

A control sample without OPA (0%) was also produced. The mix proportions and codes of all formulations are presented in Table 1.

**Table1.** Cement samples codes and contents of (OPA) and CEMI 42.5

| Cement Codes | C0 | C10 | C20 | C30 |
|---|---|---|---|---|
| CEM I 42.5% | 100 | 90 | 80 | 70 |
| OPA % | 0 | 10 | 20 | 30 |

### 2.3 Preparation of standardized mortars

In accordance with European standard EN 196-1, the tests are performed on standard mortar bars of (40 × 40 × 160) mm$^3$. The new cements were mixed with standardized sand and water according to the following proportion:

450 g ± 2 g of cement, 1350 g ± 5 g of standardized sand, 225 g ± 1 g of water. These masses correspond to the preparation of three test bars (40 × 40 × 40) mm$^3$.

### 2.4 Performed tests

2.4.1 Tests on cement powder

XRF was used to determine the chemical composition of cement powders.

The specific surface area is determined according to the standard EN196-6:2018.

The absolute and apparent density are measured on anhydrous cement powder in accordance with standards NA 2595 and NF EN 1097-3, respectively.

2.4.2 Tests on cement pastes

Normal consistency tests were carried out in accordance with NA 229. Determining the standardized consistency of cement paste is essential for assessing the initial and final setting times. These tests were performed on fresh cement pastes maintained at a laboratory temperature of 21 ± 1°C using a Vicat apparatus.

2.4.3 Tests on cement mortars

The density of samples in both fresh and hardened states was determined according to standard NF EN 1015-6 and standard NF EN 1015-10, respectively.

The water absorption by capillary test was performed according to the EN 13057 (2002) standard. The rate of water absorption by capillary suction was determined on mortar specimens placed in direct contact with water, without applying hydraulic pressure, over time.

Upon demolding, the prismatic mortar specimens (40 × 40 × 160) mm$^3$ were cured in water for 28 days.

The specimens are then extracted and dried to constant mass and sealed on four lateral faces. Then, they are placed on supports in a water tray. The first 5 mm of the specimen`s surface is immersed in the water. Mass measurements were taken at regular intervals.

The absorption of water by total immersion test was performed according to the prescription of standard NBN B15-215 (2018) norm. After drying the specimens in a ventilated oven at 105°C, they were immersed in water for 48 hours.

According to standard NF P18-459 (2010), water accessible porosity was determined by hydrostatic weighing. The test bars are conserved in water for 28 days. The porosity accessible to water is calculated based on the mass difference between the saturated and dry states. To ensure accuracy, the test was performed on three specimens.

Standard NA 234 describes in detail the procedure for measuring mechanical resistance. After 24 hours of drying in the humid chamber at a temperature of 20 ± 1°C, the samples were demolded. They were then stored in a saturated atmosphere in the humid chamber for 28 and 60 days, respectively. At the test age, the specimens were subjected to bending, and the resulting halves were then tested for compressive strength. The value reported for each cement sample is the average of three tests.

To evaluate the resistance of the developed cement to aggressive environments, mortar bars were demolded after 24 hours and stored in water for 28 days. The initial masses of the bars were then recorded. Three specimens from each sample were immersed in a 5% sulfuric acid solution ($H_2SO_4$). The solution was renewed every 15 days. At predetermined intervals, the specimens were removed and weighed to monitor mass changes.

## 3. RESULTS AND DISCUSSION

### 3.1 Characterization of blended cement

The chemical composition of the blended cement samples, as obtained from XRF analysis, is summarized in Table 2. This analysis was conducted to evaluate the effect of OPA incorporation on the oxide composition of the cements.

**Table 2.** Chemical composition of the blended cement samples

| | C0 | C10 | C20 | C30 |
|---|---|---|---|---|
| CaO | 63.96 | 62.59 | 60.69 | 59.30 |
| $Al_2O_3$ | 5.06 | 4.89 | 4.69 | 4.54 |
| $Fe_2O_3$ | 3.70 | 3.66 | 3.60 | 3.58 |
| $SiO_2$ | 19.86 | 19.07 | 18.15 | 17.45 |
| MgO | 2.30 | 2.24 | 2.15 | 2.06 |
| $Na_2O$ | 0.28 | 0.28 | 0.27 | 0.27 |
| $K_2O$ | 0.71 | 0.92 | 1.17 | 1.44 |
| $Cl^-$ | 0.035 | 0.056 | 0.080 | 0.107 |
| $SO_3$ | 3.09 | 2.95 | 2.94 | 2.98 |
| LOI | 2.25 | 6.77 | 10.74 | 15.38 |
| $CaO_{Free}$ | 0.20 | 0.03 | 0.03 | 0.03 |

The specific surface area of the obtained cement C0, C10, C20, and C30 measured using the Blain apparatus are 3470 cm$^2$/g, 3296 cm$^2$/g, 3000 cm$^2$/g, and 2647 cm$^2$/g respectively. It is observed that increasing the OPA substitution rate leads to a decrease in the specific surface area of the cement. This behavior can be explained by the lower fineness of the ash compared to CEM I 42.5 cement.

According to the Figure 3, the blended cements exhibit lower apparent and absolute densities than the reference sample. This reduction can be attributed to the lower intrinsic density of the waste material incorporated in the mixtures compared to that of the reference cement.

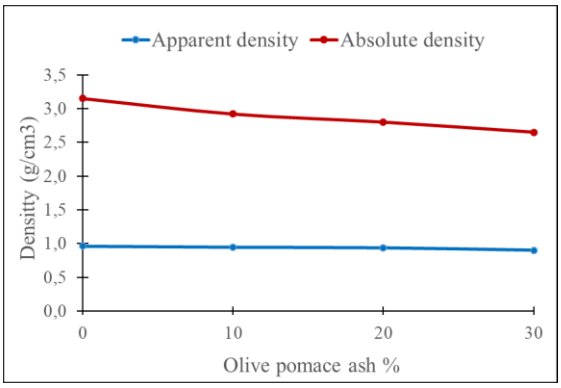


**Figure 3.** Apparent and absolute densities of the cement powder sample

### 3.2 Characterization of prepared cement pastes

As shown in Figure 4, the ratio of water to cement (W/C) required to achieve normal consistency increases from 29.4% for the reference cement to 42% for mixtures incorporating olive pomace ash. This rise reflects the higher water demand of olive pomace ash, likely due to its finer particle size and porous nature [6].

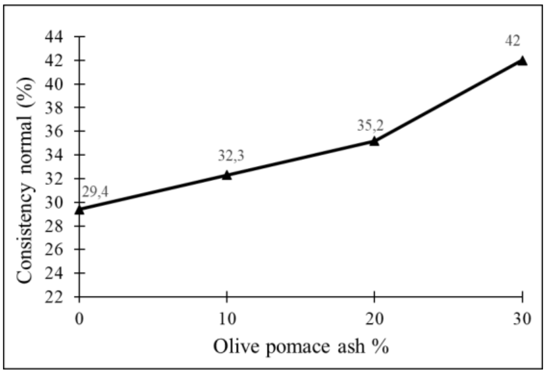


**Figure 4.** Normal consistency of blended cement pastes

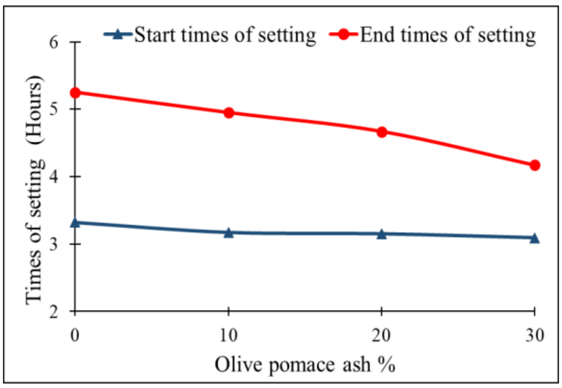


**Figure 5.** Results of the setting times

The prepared cements generally exhibited comparable setting times (Figure 5). However, the incorporation of olive pomace ash was found to slightly accelerate both the initial and final setting times compared to the reference cement. This behavior is attributed to the potassium oxide ($K_2O$) content in OPA, which contributes to accelerating the setting time.

All measured values remain within the limits recommended by the NA 442 standard, which specifies a minimum setting time of 75 minutes.

### 3.3 Characterization of prepared cement mortars

3.3.1 Workability

The test of workability was carried out in accordance with the EN 1015-3 standard. This method provides a reliable assessment of the workability of standardized mortars. It also allows for the evaluation of the influence of OPA incorporation on the rheological behavior of mortars prepared with the new cements.

As shown in Figure 6, the introduction of olive pomace ash into cement leads to a progressive reduction in the spreading value with increasing replacement levels. However, the spreading diameters remain within the limits specified by the standards. These findings are consistent with the results obtained for normal consistency. This phenomenon is attributed to the physical characteristics of OPA. It exhibits a highly porous structure, low density (Figure 3), and possibly its morphology. OPA may also contain a residual organic or carbonic fraction, which promotes significant adsorption and absorption of mixing water.

In line with the requirements of NF EN 1015-6, all prepared mortars can be classified as plastic mortars (F ≤ 200 mm).

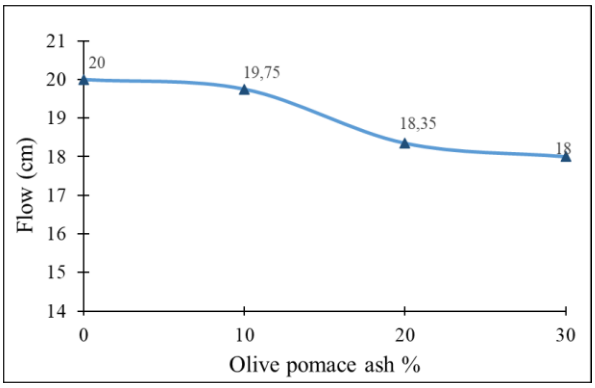


**Figure 6.** Results of workability test

3.3.2 Density

Concerning the densities of the prepared cement mortars in both the fresh and hardened states (Figure 7), the incorporation of olive pomace ash into portland cement leads to a decrease in density. The magnitude of this reduction is proportional to the amount of ash incorporated.This behavior can be attributed to the comparatively lower density of olive pomace ash relative to portland cement.

3.3.3 Absorption of water by capillary

The results of the capillary water absorption test are illustrated in Figure 8. As shown in this figure, it is observed that:

The water absorption by capillarity increases progressively with time throughout the test period (up to 1440 min).

The reference sample exhibited the highest absorption compared to the ash-based blended cements, both at 28 and 60 days of curing. Samples incorporating 20% and 30% olive pomace ash generally displayed stable absorption values between 28 and 60 days. This stability can be attributed to the stabilization of their pore structure from 28 days onward. Furthermore, it must be noted that the capillary absorption values measured at 60 days were lower than those recorded at 28 days for all cements containing olive pomace ash. This phenomenon is explained by the densification of the cementitious structure induced by olive pomace ash [20] and the formation of secondary C-S-H, which ensures the refinement of existing pores.

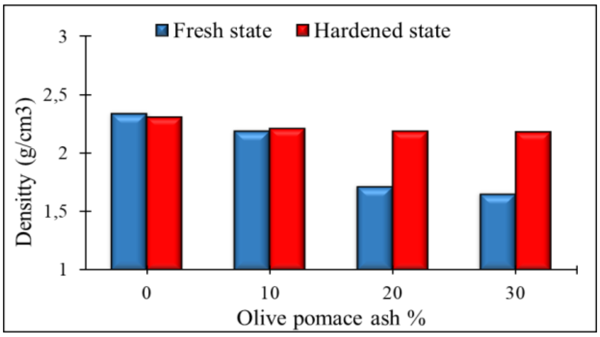


**Figure 7.** Density measurements of the fresh and hardened states of prepared cement mortars

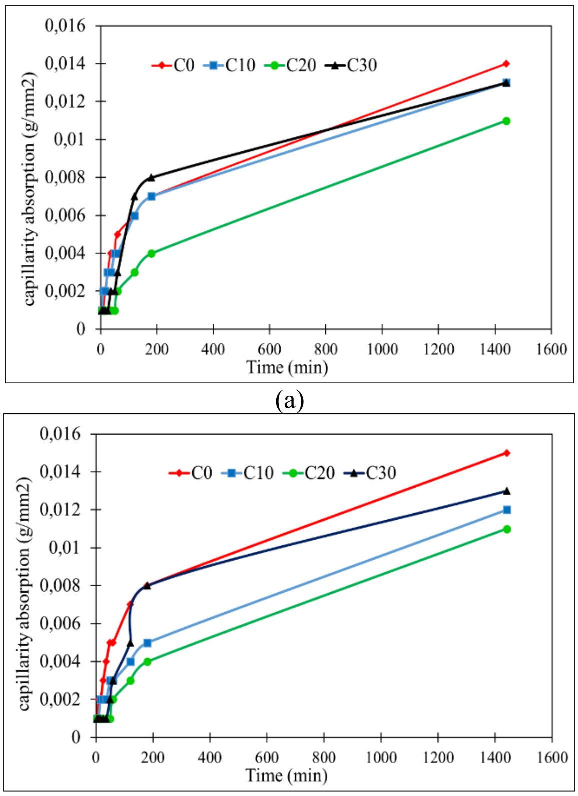


**Figure 8.** Capillary water absorption of prepared cement mortars: (a) 28 days, (b) 60 days

3.3.4 Water absorption by total immersion and porosity

As illustrated in Figure 9, the incorporation of olive pomace ash affects water-accessible porosity. This parameter follows a trend similar to total water absorption for all tested mortars. It can be detected that a 20% replacement level of olive pomace ash appears to be the most effective in reducing porosity.

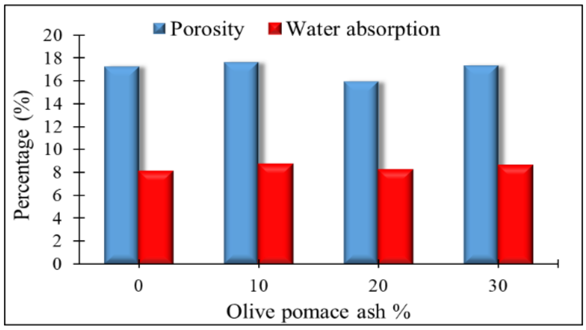


**Figure 9.** Water accessible porosity and total immersion absorption

3.3.5 Mechanical properties

In terms of mechanical performance (Figures 10 and 11), a continuous increase in strength was observed for all mixtures with extended curing, reflecting the progressive advancement of hydration reactions. Blended cement mortars containing olive pomace ash demonstrated higher compressive strength gains between 28 and 60 days compared to the reference cement (Figure 10). For example, the sample prepared with 10% ash recorded a strength increase of more than 20 MPa over this period. This gain in strength is explained by the prolonged pozzolanic effect of OPA. XRD analyses confirm the presence of both amorphous and crystalline components. The amorphous phase may contribute to long-term strength development through secondary hydration reactions with calcium hydroxide, while the crystalline phases act mainly as inert fillers [20, 21]. These results are consistent with capillary absorption measurements conducted at the same storage ages.

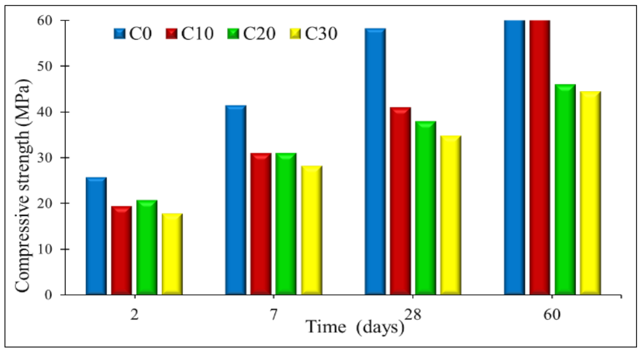


**Figure 10.** Compressive strength of cement mortars with time

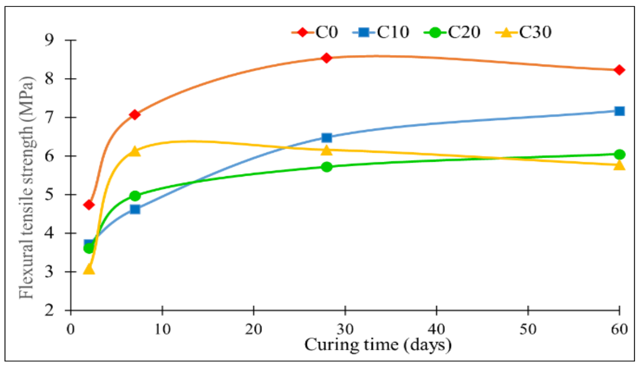


**Figure 11.** Flexural strength of prepared cement mortars

From Figure 11, the control mix (C0) shows the highest flexural strength, reaching about 9 MPa after 60 days. The

10% substitution (C10) causes only a slight reduction in early strength, while higher levels (C20, C30) significantly decrease early strength due to clinker dilution and the slow reactivity of the ash. Beyond 28 days, strength recovery is observed, confirming the delayed pozzolanic effect of olive pomace ash. This effect enhances long-term performance through microstructural densification and secondary C-S-H formation [22, 23].

3.3.6 Attack acid

From Figure 12, it can be seen that, the extension of the duration of exposure of the specimens in the acidic environment leads to increasingly significant mass losses for all the prepared cement mortars. After 45 days of immersion, the C30 sample exhibited lower mass loss compared to C20, suggesting improved resistance at higher substitution levels.

The weight loss results show a progressive increase with immersion time for all mixtures (C0, C10, C20, and C30), reflecting continuous degradation under the aggressive environment. Substituting cement with OPA led to greater mass loss compared to the C0 sample, due to increased porosity and reduced matrix cohesion. However, at a low substitution rate (10%), the weight loss remained moderate. This indicates that limited incorporation of OPA can enhance sustainability without significantly affecting durability.

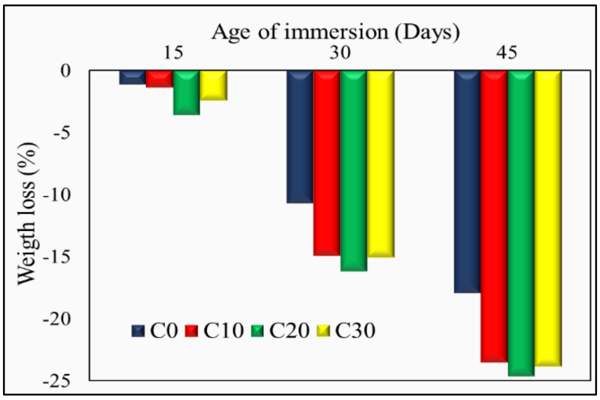


**Figure 12.** Mass loss in $H_2SO_4$ acidic

The higher porosity and lower density of OPA (Figure 3) likely create a more permeable matrix, facilitating acid ingress. Furthermore, the elevated $K_2O$ content in OPA (Table 2) could influence the stability of hydration products in an aggressive environment.

## 4. CONCLUSIONS

This study evaluated the potential of olive pomace ash (OPA) as a supplementary cementitious material. It offers both technical and environmental benefits. Through chemical, physical, mechanical, and durability tests conducted in accordance with Algerian and European standards. The main findings can be summarized as follows:

- Increasing the substitution rate of portland cement with OPA results in a reduced density of cement and mortar which may be advantageous for heavy structure applications.
- At moderate replacement levels around 10 to 20% of OPA, performance is optimized. A 10% OPA substitution recorded a notable increase in compressive strength between 28 and 60 days, highlighting the beneficial pozzolanic reactivity of the additive. Meanwhile, 20% OPA contributed to reduced porosity and improved overall performance of the cementitious materials.
- Although higher OPA content increases water demand to achieve normal consistency, it also slightly accelerates setting times and decreases the workability of standardized mortars.
- Cements containing OPA exhibit higher mass loss when exposed to aggressive environments compared to the reference cement. However, at a low substitution rate (10%), weight loss remaines moderate, indicating that limited incorporation of olive pomace ash can enhance durability.
- Overall, this study demonstrates that olive pomace ash can be incorporated into cement at a substitution level of 10%, resulting in materials suitable for the production of ordinary mortars and concretes. This approach contributes to sustainable construction practices while valorizing an abundant agro-industrial by-product.

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