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## THE EFFECT OF PISTACHIO VERA SHELL ASH (PSA) ON CONCRETE PERFORMANCE

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### ABSTRACT

Cement production is a major contributor to global energy consumption and CO<sub>2</sub> emissions, prompting the need for sustainable alternatives in the construction industry. This study investigates the potential of Pistachio Vera Shell Ash (PSA), an agricultural waste, as a partial replacement for cement in concrete. PSA was substituted at 3%, 5%, and 10% by weight of CEM I 42.5 R cement in concrete mixtures, and its effects on the physical, mechanical, and microstructural properties of concrete were evaluated. The results indicate that as the PSA substitution rate increased, the water demand of the concrete mixtures rose, leading to reduced workability. At the 3% replacement level, the compressive strength of the concrete remained comparable to that of plain concrete and only a 10% reduction was observed at 28 days. However, higher substitution rates (5% and 10%) resulted in significant strength losses, with reductions of 21% and 42.7%, respectively. Despite its non-pozzolanic nature, PSA demonstrated potential as a supplementary cementitious material (SCM), particularly in regions with abundant pistachio production. The study concludes that PSA can contribute to sustainable construction practices by reducing the environmental impact of concrete production and promoting the recycling of agricultural waste. Further research is recommended to optimize PSA substitution levels and enhance its compatibility with concrete matrices.

**Keywords:** Supplementary cementitious materials (SCM)s, Pistachio vera shell ash (PSA), Concrete compressive strength, Sustainability, Biomass.

## 1 INTRODUCTION

Concrete is the world's most popular construction material [1,2]. Cement, the main material of concrete, uses a lot of energy during its production and causes significant CO<sub>2</sub> emissions [2,3]. It is estimated that 5 to 8 % of global CO<sub>2</sub> emissions are caused by the cement sector [4]. The development of sustainable materials that could reduce the financial and environmental costs associated with cement production is essential [5].

In recent years, the utilisation of agricultural and industrial wastes in the concrete industry has emerged as a promising approach both environmentally and economically. Agricultural wastes (e.g. rice husk ash, coconut husk, wheat straw) and industrial wastes (e.g. fly ash, blast furnace slag, silica fume, waste glass) can be used as cement or aggregate substitutes in concrete mixes, reducing natural resource consumption and alleviating waste management problems. These materials also offer an attractive option due to their generally low cost and abundant availability. The world has entered a rapid industrial development with the twenty-first century. Although this development provides great benefits in terms of civilization, considering the negative effects of industrial wastes on the environment, major problems arise [6–12]. The utilization of these waste materials with useful recycling mechanisms is important in terms of cost and at the same time in terms of improving environmental impacts. Reuse of waste materials reduces raw material procurement costs. The use of recycled materials also earns points in green building certifications such as BREEAM, LEED and DGNB [13]. Recycling of industrial and agricultural waste materials such as silica fume, fly ash [3,14], waste glass [15] and rice-husk ash [16], which is used in the construction sector, which is environmentally problematic, will contribute greatly to humanity in terms of both environmental aspects and improving the properties of concrete.

According to the 2019 data of the European Ready Mixed Concrete Association (ERMCO), the European Union (EU) produces approximately 0.60 m<sup>3</sup> of concrete per year, while this amount is 0.8 m<sup>3</sup> in Türkiye [17]. In this way, researches on increasing the performance and economizing of concrete are constantly ongoing [17].

There are many studies to increase the compressive strength and durability of concrete. The use of mineral additives in the concrete mixture is one of these methods. Mineral additives fly ash (FA), silica fume (SF), blast furnace slag (BFS) play an active role in increasing the mechanical properties and durability of concrete when used in appropriate proportions [18–21].

Recently, there are studies investigating the effect of agricultural wastes on concrete performance, rice husk ash is added to the concrete mixture and gives positive results [22,23]. The use of pistachio shell ash, which is a pozzolanic material, in cement and concrete provides advantages such as reducing the permeability of concrete, resistance to chemical effects, resistance to alkali-silica reaction, improvement in workability properties, and thus strength and durability of concrete [24]

According to TUIK data, Türkiye ranks third after Iran and the USA in pistachio production. The amount of pistachio produced in 21 countries in the world is 1.158.519 tons [25]. Pistachio production in Turkish cities including Gaziantep, Şanlıurfa, and Siirt is increasing due to growing demand. According to TUIK data, it has been stated that pistachio production has doubled in the last 15 years [25]. According to global statistical review data, world pistachios production (in shell basis) reached more than 638.000 tons in 2014 [18]. Burning rate of pistachio shell is 99 %. The rate of ash is 1.2 % [26–28]. Shell obtained from pistachio, one of the most important production resources of Siirt province, is used as fuel in furnaces, but the ash formed remains as waste.

In the study by Baran et al. (2020) [29] Hazelnut shell ash was substituted up to 30 wt. % in normal CEM I 42.5R ordinary Portland cement (OPC) mixtures. Compared to OPC, the addition of hazelnut shell ash reduced the setting time by 96% and increased the water requirement for standard consistency by 59%. As the ash content increased, the compressive strength results decreased significantly. The addition of more than 5% hazelnut shell ash resulted in an unsatisfactory 28-day compressive strength value.

In the study by Tekin et al. (2021) [24], the substitution of PSA up to 30 wt. % instead of OPC in cement mortars caused a significant increase in the water requirement of the mortar and a significant delay in the setting time of the cement due to the presence of carbon-based structures. PSA substitution up to 10% gave similar early strength development (2 days) to plain cement mortar. With longer curing times, the compressive strength values of plain cement mortars can be increased by up to 17% with the use of PSA (10%). A 30% PSA substitution significantly reduced the compressive strength value of cement mortars, which was significantly aided by higher porosity properties. Between 500 and 900 ° C, PSA has a higher specific surface area [30]. Using a variety of techniques, the possible synthesis of graphitic carbon structures from waste and bio-recycles has recently been reported [31]. The partial replacement of cement with biomass ash after incineration has been proposed as an important solution due to the possibility of hazardous heavy metal and substance contamination leaching

into the environment [32]. Furthermore, these carbon-based materials have recently gained popularity as a research topic for cement-based composites to improve their properties [33]. Therefore, the use of PSA as an SCM can bring significant economic and environmental benefits to the cement industry, particularly in countries with higher pistachio production [24].

This study aims to investigate the potential of PSA as a partial replacement for cement in concrete, focusing on its impact on workability, compressive strength, and microstructural properties. While previous studies have explored the use of other agricultural wastes in concrete, the unique chemical composition of PSA, characterized by high alkali content and low pozzolanic activity, presents both opportunities and challenges. By substituting PSA at 3%, 5%, and 10% by weight of cement, this research seeks to determine the optimal substitution level that balances environmental benefits with mechanical performance. Advanced microstructural analyses, including SEM, EDX, and XRD, were employed to understand the interaction between PSA and the concrete matrix.

The findings of this study are expected to contribute to the growing body of knowledge on sustainable construction materials, particularly in regions with high pistachio production, such as Türkiye. By recycling PSA, this research not only addresses waste management issues but also offers a viable solution to reduce the carbon footprint of concrete production. Furthermore, the study provides insights into the potential of PSA as a SCM, paving the way for future research on its long-term durability and performance in concrete applications.

## 2 MATERIAL AND METHOD

In this study, CEM I 42.5 R Portland cement and pistachio vera shell ash (PSA) obtained from the furnace flue filter of the pistachio shell burned at 300–350°C in an industrial oven were used. Approximately 1.2 % of the burnt pistachio shell was obtained as ash. The sodium oxide (Na<sub>2</sub>O), potassium oxide (K<sub>2</sub>O), sulphite (SO<sub>3</sub>), magnesium oxide (MgO) ratios in the structure of pistachio vera shell ash are higher than cement, while the amounts of calcium mono oxide (CaO) and silicon dioxide (SiO<sub>2</sub>) are lower Table 1. While this has an effect on reducing the initial and final setting times, the low aluminum trioxide (Al<sub>2</sub>O<sub>3</sub>) and silicon dioxide (SiO<sub>2</sub>) amount in pistachio vera shell ash has an effect on increasing the initial and final setting times. PSA is a cementitious waste material with a high specific surface area consisting of graphene, alumina, quartz and calcite-based structures [24]. According to ASTM C618 [34], for pozzolanic activity, the amount of silicon dioxide + aluminum trioxide + iron trioxide (SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub>) should be more than 70 % in natural pozzolan [35]. However, the sum of SiO<sub>2</sub>

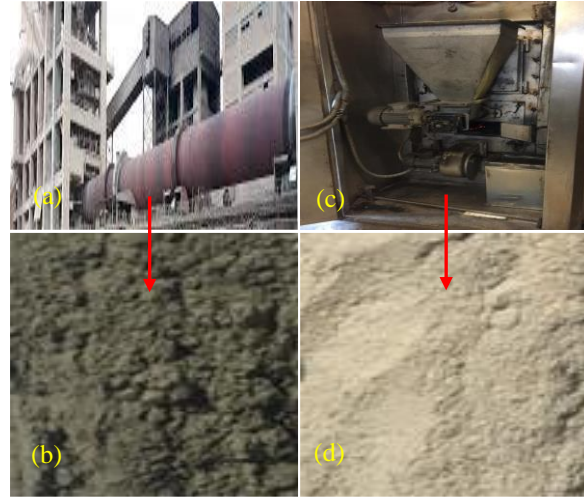
+  $\text{Al}_2\text{O}_3$  +  $\text{Fe}_2\text{O}_3$  components in the structure of PSA is approximately 4 %. This shows that PSA is not pozzolanic. The results reported by Shakouri et al. [36] and Kamau et al. [37] are consistent with the possibility that the aggregation of particles is due to high  $\text{K}^+$  content. Shakouri et al. [36] state that the  $\text{K}^+$  content of crops is influenced by plant species and fertilizers applied. Therefore, since the total alkali content of PSA is high ( $\text{Na}_2\text{O}_{\text{eq}} > 0.60\%$ ), it is important not to use it with aggregates containing reactive silica such as andesite, dacite, rhyolite, etc. in order not to cause alkali-silica reaction with PSA substitute [38]. Loss on ignition 18.33 % indicated that PSA was not fully calcined and should be burnt at higher temperatures. The fact that the Blaine value of PSA is similar to that of cement shows that it can be physically substituted for cement. In addition, the fact that the specific density value of PSA is lower than cement may enable the production of lightweight concrete (Table 1).

Natural stone in the range of 0–5 mm, 5–15 mm and 15–25 mm was used as aggregate. The density of sand consisting of fine aggregate is  $2.62 \text{ g/cm}^3$ , medium aggregate is  $2.69 \text{ g/cm}^3$ , and coarse aggregate is  $2.72 \text{ g/cm}^3$ . The sieve analysis of the aggregate mixture used in the experiment is given in Figure 3 according to the limit values. In addition, chryso delta brand superplasticizer was used. The density of the superplasticizer is  $1153 \text{ kg/m}^3$ .

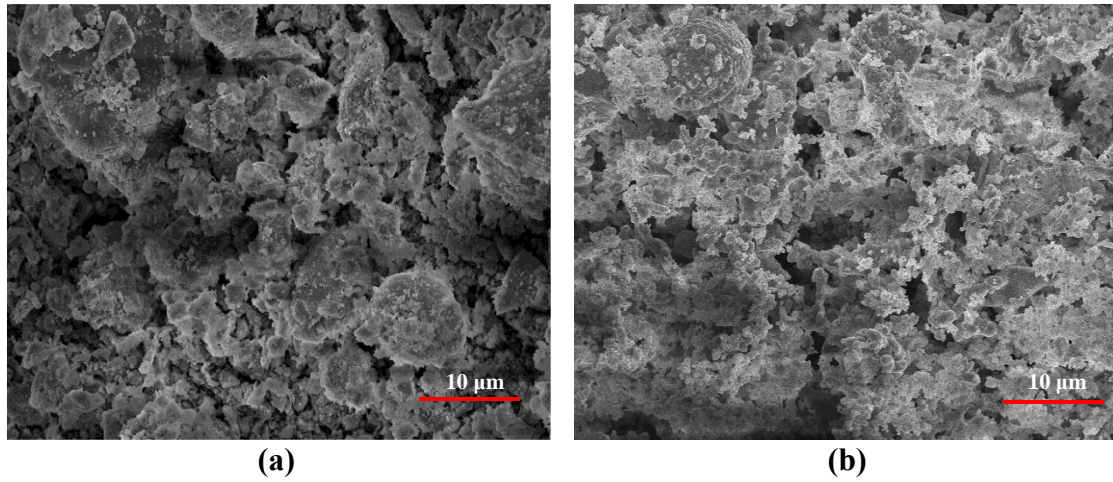
**Table 1. Physical and chemical values of PSA and Portland cement.**

		Portland Cement	PSA
Components (%)	$\text{SiO}_2$	19.61	2.4
	$\text{Al}_2\text{O}_3$	4.92	0.65
	$\text{Fe}_2\text{O}_3$	3.15	0.77
	CaO	63.66	17.07
	MgO	2.19	5.92
	$\text{SO}_3$	2.54	12.96
	$\text{K}_2\text{O}$	0.75	33.77
	$\text{Na}_2\text{O}$	0.29	8.13
	$\text{Na}_2\text{O}_{\text{eq}}$	0.78	30.35
	Loss on ignition	2.89	18.33
	(S+A+F)	27.68	3.82
	Density ( $\text{kg/m}^3$ )	3130	2350
		Blaine ( $\text{cm}^2/\text{g}$ )	3274
		Fineness (45 $\mu$ )	26.6

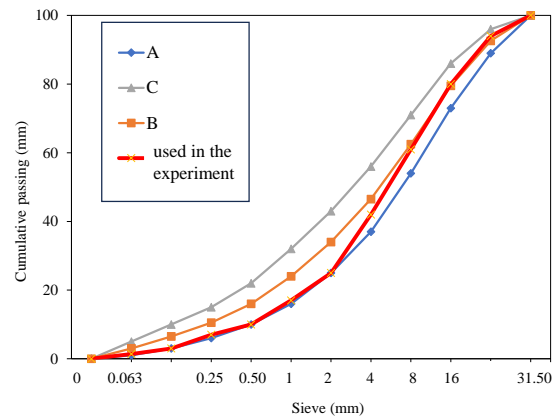
$$\text{Na}_2\text{O}_{\text{eq}}^* = \text{Na}_2\text{O} + 0.658 \text{ K}_2\text{O}$$



**Figure 1. (a) Cement plant (b) macro images of Portland cement (c) Pistachio Shell Burning Industrial Furnace (d) macro images of PSA.**



**Figure 2. SEM images of (a) Portland cement (b) PSA.**



**Figure 3. Sieve analysis.**

In order for the materials in Figure 1, where macro images are presented, to be considered interchangeable, the chemical and physical properties presented in Table 1 must be similar. The close Blaine values of both materials are supported by the similar granules shown in the SEM images in Figure 2.



## 2.1 Method

In this study, mix proportions for 1m<sup>3</sup> target concrete C25/30 design are given in Table 2 according to TS EN 196-1 [39]. In concrete production, 0, 3, 5 and 10 % by weight of PSA was substituted for Portland cement. The first mix was plain concrete (denoted by PL) and was used as the control group. PSA replaced cement at 3% (denoted by PSA3), 5% (denoted by PSA5) and 10% (denoted by PSA10) of the cement mass, respectively. In order to determine the 7-day and 28-day compressive strength of PSA-substituted concrete and plain concrete, 6 cube specimens of 150 mm × 150 mm × 150 mm<sup>3</sup> were cast for each mix (see Figure 4). Specimens were demoulded after 24 hours and cured in saturated lime solution at 20 ± 2 ° C and R.H. ≈ 95% for 7, 28 days.

After determining the physical and mechanical properties of the concrete specimens, the specimens were pulverised and the hydration products were observed by scanning electron microscopy (SEM), energy dispersive spectrometry (EDS) and X-ray diffraction (XRD) analyses.



**Figure 4. Sample preparation.**

**Table 2. Mix ratio of 1 m<sup>3</sup> concrete and slump values.**

Material	PL	PSA3	PSA5	PSA10
Portland cement, kg	300	291	285	270
PSA, kg	-	9	15	30
Water, kg	190	190	190	190
Admixture, kg	4.2	4.2	4.2	4.2
Fine aggregate 0-5mm, kg	983	981	981	978
Coarse aggregate 5-15 mm, kg	369	368	368	367
Coarse aggregate 15-25 mm,kg	404	403	403	402
Water/Binder	0.63	0.63	0.63	0.63
Unit weight, kg/m <sup>3</sup>	2250	2247	2245	2241

Cube specimens with dimensions of  $150 \times 150 \times 150 \text{ mm}^3$  was subjected to compressive tests in accordance with TS EN 12390-2 [40] after 28 days of curing in accordance with TS EN 12390-3 [41]. Compressive strength values were calculated by averaging three specimens from each design group after testing. The compressive strength was measured using a hydraulic concrete press machine with a load of  $0.6 \text{ N/mm}^2/\text{sec}$ .

For SEM, XRD analyses, samples were taken from the samples exposed to ambient and high temperature and brought into a form suitable for analysis. Firstly, the samples were made suitable for the SEM device. In order to take the images of the prepared samples, the surfaces to be imaged were made conductive with the help of Au coating device. The samples were placed in the chamber of the SEM device and then the process started. The surface morphologies of the samples were characterized at various scales by scanning electron microscopy (SEM, Hitachi SU3500).

XRD analyses were performed at a wavelength of  $1.5406 \text{ (\AA)}$  between  $10$  and  $90^\circ$  at a step rate of  $0.02^\circ$  and a scan rate of  $2^\circ$  per minute.

### 3 RESULTS AND DISCUSSION

#### 3.1 Slump values

Slump classification of the specimens was performed according to TS EN 206-1 [42] and EN 12350-2.

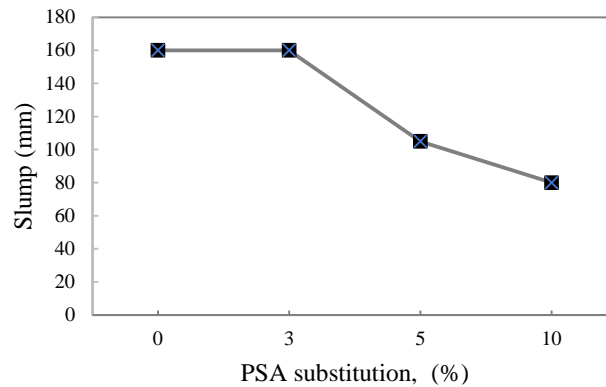


**Figure 5. Slump values of (a) PL; (b) PSA10.**

As the PSA substitution rate in the concrete increased, the water requirement of the specimens increased and workability decreased (see Figure 5). The slump value of plain concrete was 160 mm (slump class S4). Although the workability of the concrete did not change at 3% PSA substitution, the slump values decreased to 105 (slump class S3) and 80 mm (slump class S2) at 5% and 10% substitution, respectively (see Figure 6). The reason for the increase



in the need for water is thought to be the increase in surface area because of the small particle size of PSA. This increase in the amount of water resulted in an increase in the initial and final setting time.



**Figure 6. Slump values.**

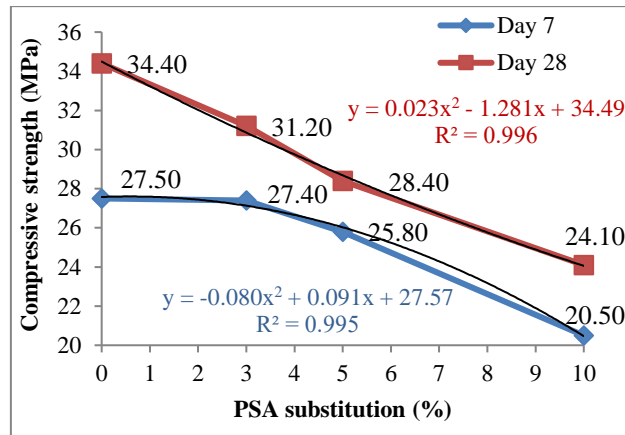
### 3.2 Compressive strength

This study examines the effects of agricultural waste PSA on the early and long-term compressive strength of concrete. According to the 7-day early strength results, the compressive strength of the PSA3 sample (27.4 MPa) is nearly identical to that of the reference concrete (PL) (27.5 MPa). This indicates that low substitution ratios of PSA do not significantly affect the early strength of concrete. However, it was observed that strength losses increased as the substitution ratio increased. In the 28-day strength values, compared to the reference concrete (34.40 MPa), the strength of PSA-containing concrete decreased with each increase in the substitution ratio. Particularly, the strength of the PSA10 sample dropped to 24.1 MPa, suggesting that high substitution ratios of PSA may negatively impact the mechanical properties of concrete Table 3. These results demonstrate that the use of agricultural waste PSA in concrete can be considered a sustainable solution, but the substitution ratio needs to be optimized. Additionally, investigating chemical or physical modifications to enhance the compatibility of PSA with the concrete matrix could be beneficial in minimizing strength losses.

**Table 3. The effect of PSA substitution on the compressive strength values of concrete.**

Day 7				Day 28			
Measured		$\sigma^1$	Strength Effectiveness <sup>2</sup>	Measured		$\sigma^1$	Strength Effectiveness <sup>2</sup>
(MPa)			(%)	(MPa)			(%)
PL	27.5	1.20	-	34.4	1.05	-	
PSA3	27.4	1.25	-0.1	31.2	1.15	-10.1	
PSA5	25.8	1.30	-6.6	28.4	1.20	-21.1	
PSA10	20.5	1.32	-34.1	24.1	1.35	-42.7	

<sup>1</sup> standard deviation, <sup>2</sup>Strength-effectiveness =  $\frac{\text{PSA cs} - \text{PL cs}}{\text{PL cs}} \times 100\%$



**Figure 7. The effect compressive strength of PSA substitution.**

Eq. (2) was obtained by applying regression analysis to the compressive strength value in Figure 7.

$$f'_{cf} \text{ (MPa)} = f'_c + aV_s + bV_s^2 \quad (1)$$

$$f'_{cf} \text{ (MPa)} = 34.49 - 1.281V_s + 0.023V_s^2 \quad (2)$$

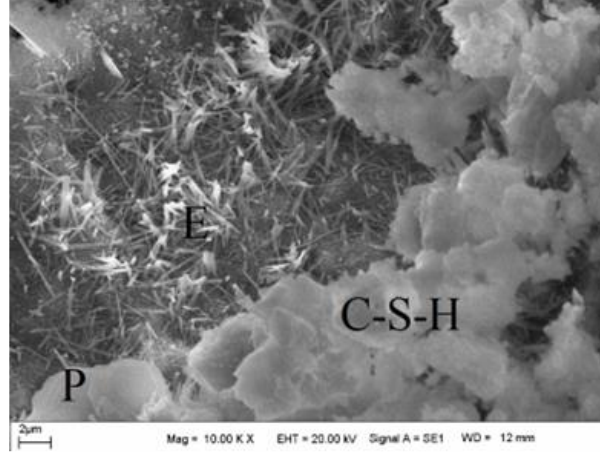
Figure 7 was obtained by using the regression analysis and substituting  $f'_c = 34.40$  MPa in Equation. (1). Table 3 shows a favourable agreement between the test results and the compressive strength (day 28) predictions made using Equation. (2). Prediction errors are less than 1.07%.

### 3.3 Microstructure analyses

In this study, the interactions of PSA with the concrete matrix were investigated in detail using advanced microstructural analysis techniques such as SEM, EDX and XRD to better understand the mechanical effects.

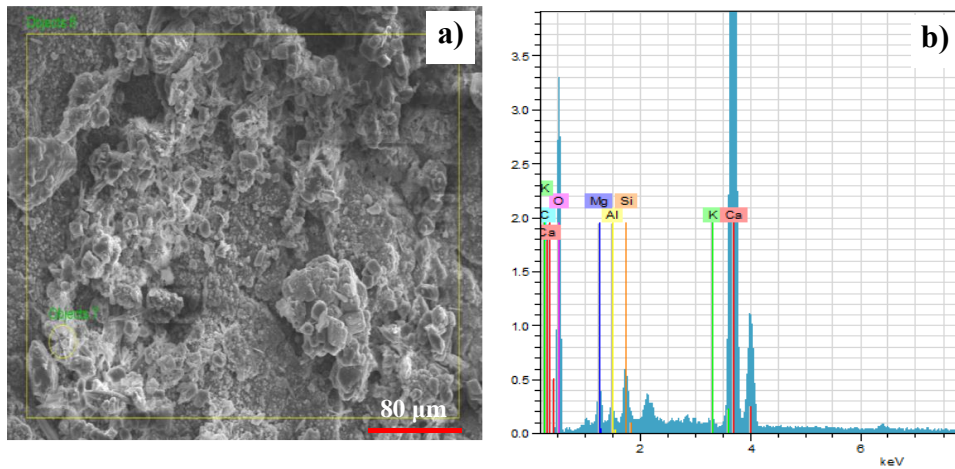
Cement hydration products were observed with the SEM test of the 3 % of PSA-substituted sample. X-ray diffraction and energy dispersive spectrometry experiments show the interaction of calcium, chlorine and alkali ions in oxidised cement concrete with cement PSA, alite (Ca-Si-O), combeite (Na-Ca-Si) and wadeite (K-Si-O) compositions in cement.

The hydration products formed in concrete, portlandite (P), calcium hydroxide (Ca (OH)<sub>2</sub>) crystals in the structure of calcium silicate hydrate gel (C-S-H) bind with pozzolans and form products with pozzolanic properties. Slab portlandite (P) crystals and ettringite structures (E) in the concrete sample were observed by scanning electron microscopy, Figure 8.



**Figure 8.** Hydration products of PSA3 P: portlandite (CH), E: ettringite ( $C_6A\dot{S}_3H_{32}$ ), calcium silicate hydrate gel (C-S-H).

The high alkali content of PSA added in the cement paste caused the increase. The high alkali content of PSA caused the formation of calcium belit ( $2CaSO_4.K_2SO_4$ ) in the cement paste. Thus, ettringite crystals in a needle-like shape, calcium alumina formation hydrates, and calcium monosulfoalumina hydrates are thought to reduce the viscosity of cement paste [29]. Additionally, ettringite crystals are quite stable and cause an enormous expansion in the cement paste, Figure 9.

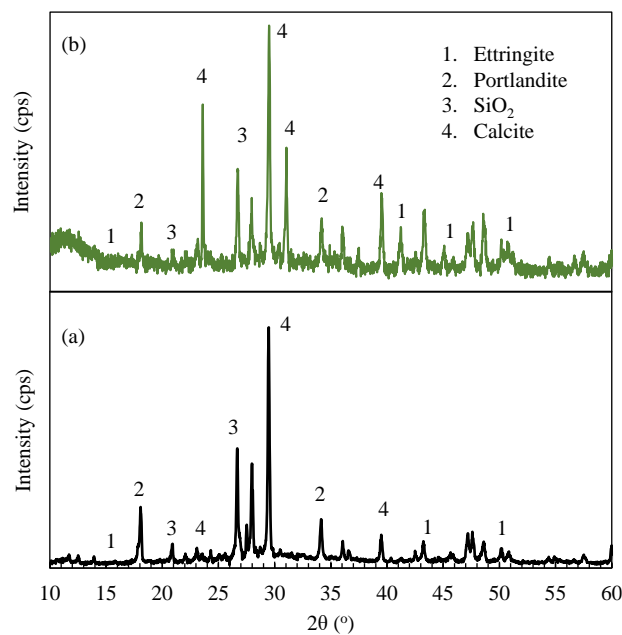


**Figure 9.** Analysis of PSA3 (a) SEM image; (b) EDS.

According to Figure 5, which shows the SEM-EDX analysis of 3% PSA-substituted concrete, calcium (Ca), silicon (Si), calcium (C) and oxide (O) ratios are high. Accordingly, the presence of calcium (Ca), silicon (Si) and oxide (O) is evidence of calcium silicate hydrate gel (C-S-H) formation. In addition, the presence of calcium (Ca) may have caused the formation of calcium carbonate (calcite).

According to Figure 10, Indicates the results of the XRD analysis of the concrete, The peak of quartz was observed at the position of  $2\theta = 29.04^\circ$ . In addition, predominant mineral phases (calcite) at different Bragg angle positions ( $2\theta = 23.62^\circ, 31.02^\circ, 39.46^\circ$ )

stand out in XRD diffraction patterns. It would be correct to say that higher calcite peaks occur in PSA-substituted concretes since shell ash is used. Portlandite ( $\text{Ca}(\text{OH})_2$ ) peaks can give us an idea for the strength product C-S-H in the amorphous structure. The similar intensity of the free-moving  $\text{Ca}(\text{OH})_2$  peaks in the two samples indicates that the samples have similar strength [43]. PSA substitution increased the amount of ettringite. The viscosity lowering effect of ettringite may be the reason for the decrease in slump analysis consistency in Figure 5 with increasing PSA substitution [44]. In addition, the volumetric expansion caused by the Ettringite needles can cause capillary cracks on the concrete surface due to internal forces and reduce the durability of the concrete [45,46].



**Figure 10. XRD analysis of (a) PL (b) PSA3.**

## 4 CONCLUSIONS

This study highlights the importance of using agricultural waste as a cement substitute in concrete production from a sustainability perspective. Although pistachio shell ash (PSA), an agricultural waste, has no binding properties, it has significant potential to reduce the environmental impact of concrete and increase resource efficiency. The use of these wastes in concrete mixes helps to conserve natural resources, reduce the carbon footprint and alleviate waste management problems.

1- The workability of concrete showed an inverse relationship with PSA replacement rates. While a 3% PSA substitution maintained the original workability, a 10% substitution caused a 50% reduction in slump values. This high water demand resulting from the loss of

workability due to PSA can potentially promote crack formation and adversely affect the long-term durability of concrete.

2- The experimental results show that concrete with 3% PSA substitution achieved a compressive strength comparable to that of plain concrete at early age. However, there was a 10% decrease in compressive strength at 28 days. The decrease in compressive strength became more pronounced with higher substitution rates, showing strength reductions of 10%, 21% and 42% for 3%, 5% and 10% PSA substitutions, respectively, compared to the control mix.

3- SEM-EDX and XRD analyses revealed intense calcite peaks in the 3% PSA-substituted concrete specimen, which can be attributed to its agricultural waste origin. However, the observed intense ettringite formation is probably responsible for the decrease in both slump and compressive strength values.

4- Since the amount of silicon dioxide + aluminium trioxide + iron trioxide ( $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ ) = 3.87 % < 70.00 % is low, it was that PSA is not a pozzolanic material. However, it is thought to have pozzolanic properties when used with cement.

5- This study demonstrates that incorporating PSA as a SCM —specifically at a 3% cement replacement level—delivers notable environmental and economic advantages by valorizing agricultural waste in alignment with global sustainability objectives. Beyond reducing reliance on conventional cement, the adoption of PSA contributes to natural resource conservation and mitigates CO<sub>2</sub> emissions inherent in cement manufacturing. This approach is particularly beneficial for regions with high pistachio cultivation, offering a dual solution for sustainable waste management and eco-conscious construction practices.

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## Conflict of Interest Statement

There is no conflict of interest between the authors.



## Statement of Research and Publication Ethics

The study is complied with research and publication ethics.

## Artificial Intelligence (AI) Contribution Statement

This manuscript was entirely written, edited, analyzed, and prepared without the assistance of any artificial intelligence (AI) tools. All content, including text, data analysis, and figures, was solely generated by the authors.

## Contributions of the Authors

M.Doğruyol: Formal analysis, Investigation, Data curation, Conceptualization, Methodology, Resources, Writing - review & editing, Visualization. M.Durmaz: Conceptualization, Methodology, Formal analysis, Investigation, Writing - review & editing.

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