





# Challenge Journal

## OF CONCRETE RESEARCH LETTERS

### Research Article

## Tea waste ash: Characterization, pozzolanic activity and effects on the fresh properties of cement pastes

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### ABSTRACT

It is a well-known fact that the use of agricultural wastes in cementitious composites by turning them into ash provides both ecological and economic benefits. Tea factories in Türkiye are located in the Eastern Black Sea region. Tea waste generated in factories locally causes environmental problems. This study focuses on the usability of the wastes left over from the products processed in the tea factory. Tea waste from Artvin, Türkiye was turned into ash at 850 °C. Physical, chemical, mineralogical, thermogravimetric and morphological properties of tea waste ash were examined. Pozzolanic activity test was performed to determine its usability in cement systems. Blended cement pastes were prepared by substituting up to 10% tea waste ash. Physical and chemical properties of blended cement pastes were determined. As a result, the tea waste ash was found to contain a high proportion of K<sub>2</sub>O. The waste material does not show pozzolanic properties. The hydration of cement pastes has accelerated with tea waste ash. Tea waste ash substitution has increased standard consistency water of blended cement pastes. Tea waste ash substitution up to 8% provided the initial setting time included in the standard. It was concluded that tea waste ash could be used as a setting accelerator.

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### 1. Introduction

Expansion of the construction sector driven by population growth has confronted humanity with unprecedented challenges. One major consequence of these developments is the significant increase in waste production (Çelik et al. 2024; Şengel et al. 2022; Canbaz et al. 2021). The utilization of waste materials in concrete structures plays a critical role in promoting environmental sustainability and supporting recycling efforts (Bulut 2024; Karalar and Çavuşlu 2022). Agricultural waste is contingent upon the nature of the agricultural activities undertaken, which can be generated from crop residues in the field, or from agro-based industries (Bekem Kara 2024). After the harvest and consumption of some agricultural products such as corn, peanuts, sugar cane;

wastes such as corn cobs, peanut shells and sugar cane pulp are generated (Raheem and Ikotun 2020). Safe disposal of agricultural wastes is an important issue in terms of reducing the volume of waste and eliminating storage problems and costs, as well as preventing environmental pollution. In recent years, it has been ascertained that the use of these wastes in cementitious composites can be beneficial both economically and ecologically.

Studies on agricultural wastes point that the agricultural product that leaves the most residues is rice. The annual production of rice worldwide is 600 million tons (Younes et al. 2018). While only 17% of this amount is used, it is known that 83% is rice husk waste. Burning rice husk in open areas for disposal causes soil and water pollution (Khan et al. 2020). In recent years, the most no-

table agricultural waste is rice husk ash. It is known that rice husk ash has pozzolanic properties. Rice husk ash, which has a high silica content, is substituted for cement. (Mohseni et al. 2016; Younes et al. 2018; Zahedi et al. 2015).

Moreover, studies on the usability of regionally produced agricultural wastes in cementitious composites are available in the literature (Baran et al. 2020; Tekin et al. 2021). One of the agricultural wastes with regional production is tea, which is the most consumed beverage after water. In this context, studies published in recent years are summarized. Anjum et al. (2020) washed the tea wastes they collected with distilled water 4-5 times to separate them from sugar and milk components and used them in brick production by sieving. Jakhrani et al. (2019), on the other hand, collected the tea waste from student cafes and local restaurants in the Seoul area and used it in the production of mortar. Nasr et al. (2019) obtained tea waste ash by burning domestic waste tea at 600 °C and replaced it with cement up to 10% in mortar

production. They found that the 7.5% substitution increased the compressive strength compared to the reference. Gupta et al. (2015) named the tea powder obtained after the preparation of home tea as “tea waste” and collected it from the university cafeteria for use in brick production. The common point of the mentioned studies is that the tea wastes used are the residues from the brewed tea. It is troublesome to collect these wastes from various places. In addition, milk, sugar, sweetener, etc. of collected tea wastes must also be removed. It is also known that the quality of teas differs from region to region. In this context, it will not be possible to talk about the same characteristics and homogeneity of domestic tea wastes. Therefore, this study focused on the factories that are the source of tea waste. In Türkiye, where there are 274 tea factories in total, the region that is suitable for tea cultivation in terms of climate is the Eastern Black Sea, and the planted tea areas and the amount of tea produced increase every year. Tea production map of Türkiye is seen in Fig. 1.

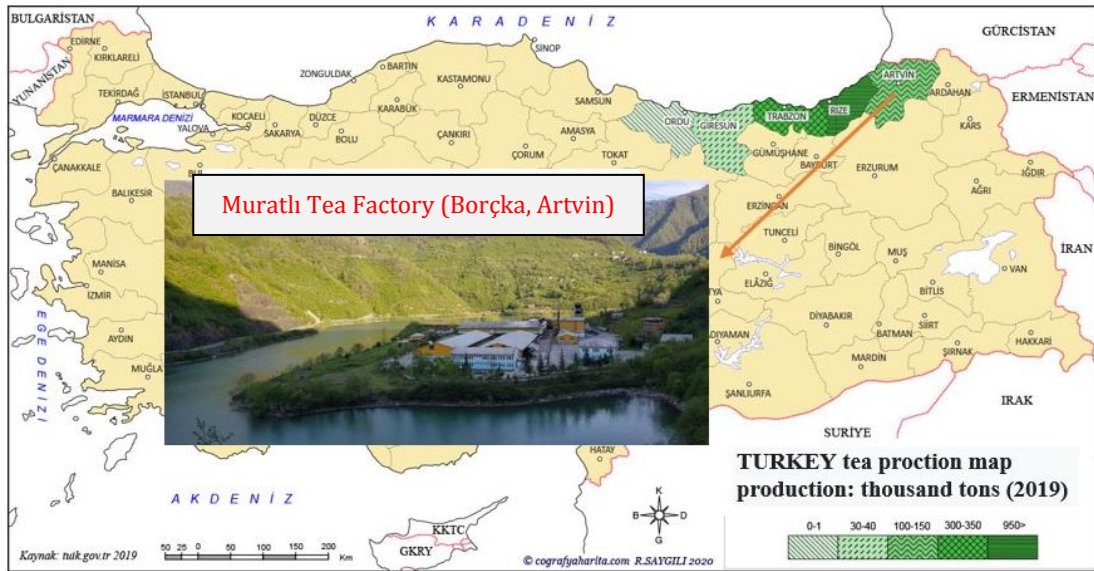


Fig. 1. Tea production amounts in Türkiye, and Muratlı Tea Factory (TEBGE 2024).

The total product obtained annually in the Eastern Black Sea region consists of around 1.5 million tons of fresh tea, so approximately 650 thousand tons of tea waste is generated every year (BEP A 2024). The collection of waste tea fibers in a closed volume creates storage problems and additional costs. Kara (2018) concluded that tea waste can be used as natural fiber up to 7 kg in 1 m<sup>3</sup> of concrete, but it would be appropriate to use it up to 5 kg in places that will be exposed to abrasion (Kara 2018). One of the methods of destroying waste tea fibers is incineration. However, burning this waste together with coal does not provide the limit values specified in the Regulation on Control of Industrial Air Pollution. In this context, it is more advantageous to burn waste tea fibers in a separate incineration unit (RTB 2013). Terzi et al. (2025) used tea ash together with blast furnace slag. Tea ash was substituted for cement at rates of 1.25%, 2.5%, 3.75%, 5%, 7.5% and 10%. Tea ash

used with blast furnace slag exhibited lower strength than the control sample on days 2, 7, 28, and 90. As far as the author knows, the waste tea fibers obtained from the factory in the production of cement composites have not been turned into ashes and replaced with cement until now. In this regard, the potentiality of ash obtained from the incineration of tea waste to usability in cement systems is investigated in the present study.

## 2. Materials and Method

In this study, CEM I 42.5 R Portland cement (PC), Ca(OH)<sub>2</sub>, tea waste and water were used. Cement was obtained Oyak Cement Plant (Ordu, Türkiye). The cement's specific surface area was 3,316 cm<sup>2</sup>/g, its specific gravity was 3.13 g/cm<sup>3</sup>. Chemical properties of PC is given in Table 1.

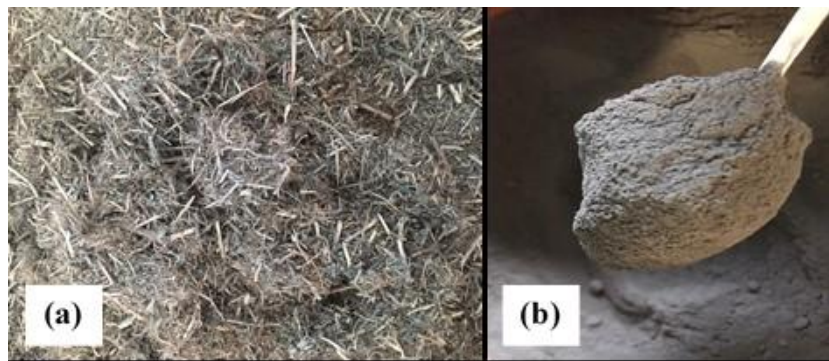
**Table 1.** Chemical properties of cement.

Oxide	CaO	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	SO <sub>3</sub>	MgO	Na <sub>2</sub> O	Loss of ignition	Insoluble matter
Value (%)	62.64	4.56	3.36	19.05	2.88	2.98	0.15	3.02	0.30

### 2.1. Preparation of TWA

Tea wastes taken from Muratlı Tea Plant (Artvin, Türkiye) storage area were first dried at  $100 \pm 5$  °C. According to Tekin et al. (2021) The incineration of agricultural wastes plays a crucial role in reducing their volume, enabling relevant industries to utilize smaller land areas and incur lower disposal costs (Tekin et al. 2021). Then the tea waste was exposed to 850 °C for three hours and

coded as TWA (Fig. 2). Upon reviewing the literature, it was observed that tea waste was burned at 600-700 °C. However, the loss of ignition values of the ash obtained in these studies are on average 13%. To minimize the loss of ignition, the temperature was increased in this study (Nasr et al. 2019; Terzi et al. 2025). No grinding process was performed on the ash after high temperature. TWA was sieved using a 250-micron sieve. Material passing through the sieve was used.

**Fig. 2.** Tea waste from tea factory and TWA.

### 2.2. Characterization of TWA

The elemental composition of TWA was determined using the X-ray fluorescence (XRF) method. Specific gravity and specific surface area were determined using the surface area analysis (BET) method. In addition, the particular size distribution of TWA was determined by laser particle size analyzer (Malvern, model 'Mastersizer Hydro 2000 MU'). Mass losses of TWA at increasing temperatures were determined with the help of thermogravimetric analysis (TG) in Perkin Elmer STA-6000 device. In this study, TG of TWA was performed at 10 °C / min heating rate up to 900 °C. X-Ray diffraction patterns (XRD) of TWA were obtained at room temperature, in Bruker brand Discover-D8 model powder diffractometer system and using copper radiation [ $\lambda$  ( $CuK\alpha$ ) = 1.54056 Å] in the scanning range of  $2\theta = 5.0-80.0^\circ$ . SEM was used to investigate morphology of TWA.

### 2.3. Pozzolanic activity of TWA

The hydraulic property determined in terms of compressive strength with the mortar prepared by mixing ground natural pozzolan, water,  $Ca(OH)_2$  and sand is defined as pozzolanic activity (TS 25 2008). The pozzolanic activity of The TWA utilized in the study was assessed in accordance with the TS 25 standard (Table 2).

The prepared mortar sample was poured into  $40 \times 40 \times 160$  mm<sup>3</sup> sized molds (Fig. 3). It was demoulded the next day and kept at  $55 \pm 2$  °C for 6 days.

### 2.4. Preparation of blended cement pastes with TWA

In this study, reference cement paste (M1) was prepared with 100% Portland cement. In addition, 2, 4, 6, 8, and 10% TWA was replaced by cement by weight. Thus, five different blended cement pastes were produced according to TS EN 196-3 (2017) standard. Blended cement pastes were labelled M2, M3, M4, M5 and M6.

**Fig. 3.** Mortar samples for pozzolanic activity.

**Table 2.** Amount of materials for pozzolanic activity.

Materials	TWA	TS 25
Ca(OH) <sub>2</sub>	150 g	150 g
Pozzolan	218.30 g	$T = 2 \times 150 \times$ (density of pozzolan/ density of Ca(OH) <sub>2</sub> )
Standard aggregate	1,350 g	1,350 g
Water	$184.15 + 90 = 274.15$	$0.5 \times (150 + T)$

### 2.5. Preparation of blended cement pastes with TWA

In this study, reference cement paste (M1) was prepared with 100% Portland cement. In addition, 2, 4, 6, 8, and 10% TWA was replaced by cement by weight. Thus, five different blended cement pastes were produced according to TS EN 196-3 (2017) standard. Blended cement pastes were labelled M2, M3, M4, M5 and M6.

### 2.6. Experimental methods

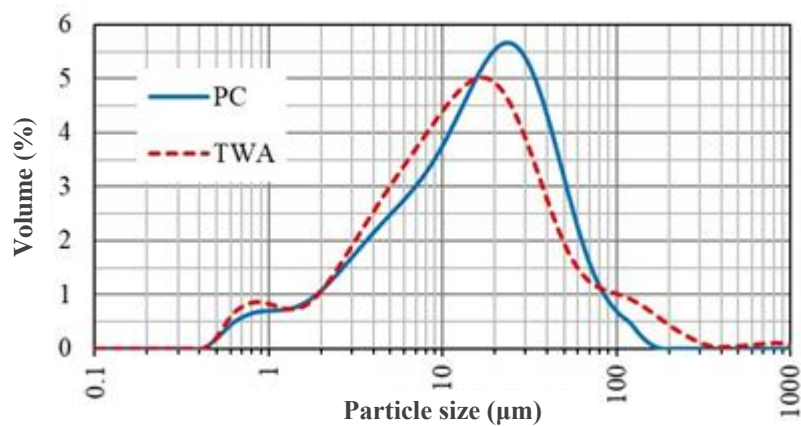
The compressive strength of the mixture prepared for pozzolanic activity was measured on the 7<sup>th</sup> day in accordance with TS EN 196-1 (2016) standard. The standard consistency water and setting times of the produced cement pastes were determined with the Vicat device. The volume expansion of the mixtures was found with the Le Chatelier mold. The setting times were performed in accordance with the TS EN 196-3 (2017) standard, with the ambient temperature being  $20 \pm 2$  °C and the

mixing water temperature being  $20 \pm 2$  °C, under experimental conditions. The volume expansion that may occur in the cement paste with the Le Chatelier mold was determined in accordance with the TS EN 196-3 (2017) standard.

## 3. Results and Discussion

### 3.1. Characterization of TWA

Specific gravity of TWA was  $1.63 \text{ g/cm}^3$ , and the specific surface area (BET) was  $11,810 \text{ cm}^2/\text{g}$ . Cumulative particle size distribution of TWA is seen in Fig. 4. The high peak was in the 10–100  $\mu\text{m}$  range. There are two smaller peaks below 1  $\mu\text{m}$  and above 100  $\mu\text{m}$ . In addition,  $d_{90}$ ,  $d_{50}$  and  $d_{10}$  values of TWA were 54.52  $\mu\text{m}$ , 12.663  $\mu\text{m}$ , and 2.400  $\mu\text{m}$ , respectively. It was seen that PC has more homogeneous distribution than TWA.  $d_{90}$ ,  $d_{50}$  and  $d_{10}$  values of PC were 46.265  $\mu\text{m}$ , 15.404  $\mu\text{m}$ , and 2.605  $\mu\text{m}$ , respectively.

**Fig. 4.** Cumulative particle size distribution of TWA.

The chemical compositions of PC, TWA and blended cements are shown in Table 3. The chemical components of the blended cements were theoretically calculated according to the replacement amount of TWA with cement. Values were evaluated according to some TS EN 197-1 (2012) limitations. When the chemical compositions of TWA were examined, it was established that the SO<sub>3</sub>, MgO and glow loss amounts were above the limit value required by the standard. However, it was observed that the SO<sub>3</sub>, MgO and glow loss amounts of the cements blended with up to 10% TWA substitute used in this

study did not exceed the standard value. The limit value of Cl<sup>-</sup> for all cements is 0.1% (TS EN 197-1 2012). When the oxides of the blended cements are calculated theoretically, up to 4% TWA substitution provides a limit value.

However, in 6, 8 and 10% replacements, the Cl<sup>-</sup> limit value is exceeded, which may cause rebar corrosion for reinforced concrete. The alkali content depends on the amount of K<sub>2</sub>O and Na<sub>2</sub>O in the cement (Zhang 2011). Alkalines, a minor but inevitable component of PC clinker, originate from the raw materials used in cement produc-

tion. Owing to variations in the composition of raw materials, the alkali content in cement produced by different factories shows significant differences across various regions (Huang and Yan 2019). When the alkali presence of TWA is examined, it is observed that its main component is  $K_2O$  (31.31%). Due to the high alkali con-

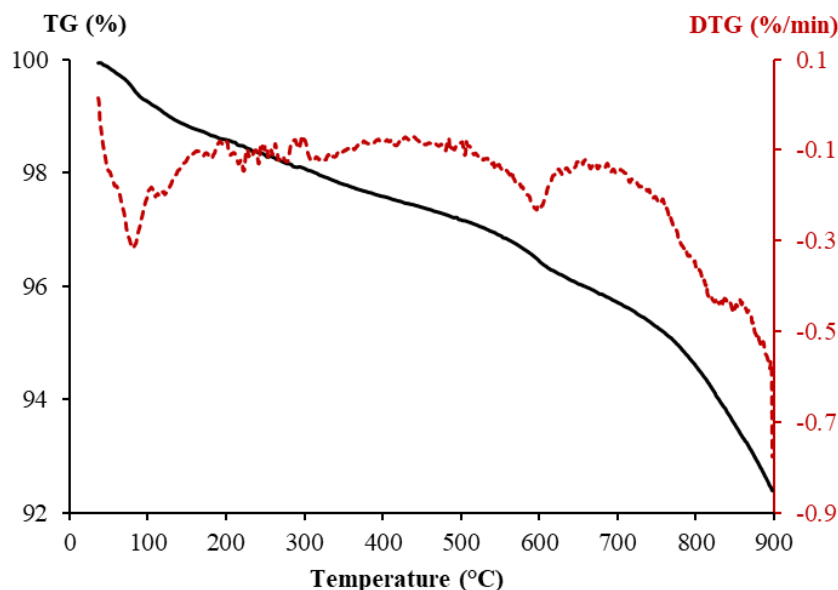
tent of TWA and low content of  $SiO_2+Fe_2O_3+Al_2O_3$ , the alkali content of the blended cement increased significantly with the increase of TWA substitution. The total alkali content (%) for M1, M2, M3, M4, M5 and M6 mixtures was calculated as 0.88, 1.28, 1.67, 2.07, 2.47 and 2.86, respectively.

**Table 3.** Chemical properties of blended cements.

Oxide (%)	TWA	M1 (OPC)	M2	M3	M4	M5	M6
CaO	13.95	63.10	62.12	61.13	60.15	59.17	58.19
SiO <sub>2</sub>	3.13	19.56	19.23	18.90	18.57	18.25	17.92
Al <sub>2</sub> O <sub>3</sub>	8.24	4.41	4.49	4.56	4.64	4.72	4.79
Fe <sub>2</sub> O <sub>3</sub>	1.18	3.16	3.12	3.08	3.04	3.00	2.96
K <sub>2</sub> O	31.31	0.79	1.40	2.01	2.62	3.23	3.84
Na <sub>2</sub> O	0.10	0.36	0.35	0.35	0.34	0.34	0.33
P <sub>2</sub> O <sub>5</sub>	8.24	–	0.16	0.33	0.49	0.66	0.82
MnO	3.90	–	0.08	0.16	0.23	0.31	0.39
SO <sub>3</sub>	5.64	2.99	3.04	3.10	3.15	3.20	3.26
MgO	5.42	2.36	2.42	2.48	2.54	2.60	2.67
Cl	1.57	0.02	0.05	0.08	0.11	0.14	0.18
Loss of ignition (LOI)	9.47	2.70	2.84	2.97	3.11	3.24	3.38

Mass loss values of TWA at increasing temperatures are presented in Fig. 5. According to the DTG curves, endothermic peaks formed for TWA under 100 °C indicate that the adsorbed water in the material structure evaporates. The second endothermic peak, occurring at around 600 °C, is believed to signify the loss of water that is more strongly bonded to the structure. It was found that the total mass loss of TWA at 900 °C amounted to 7.61%. In XRD analysis, 77% of TWA was found to be crystalline and 23% amorphous. Sylvite is potassium chloride in natural mineral form (Fig. 6). It is known

from the literature that crystalline materials do not exhibit pozzolanic properties (Bekem Kara 2022; Erdem et al. 2007). When the diffraction patterns are examined, it is seen that all minerals are of potassium origin. This situation is parallel to  $K_2O$ , which emerged as the main component in the XRF analysis. When the TWA image was magnified 5000X, it was found to have a needle-like structure (Fig. 7). This situation is attributed to the presence of  $K_2O$ , which has a high content in TWA. In addition, the acicular image supports the high specific surface area (11,810 cm<sup>2</sup>/g) determined by BET.



**Fig. 5.** TG-DTG analysis of TWA.

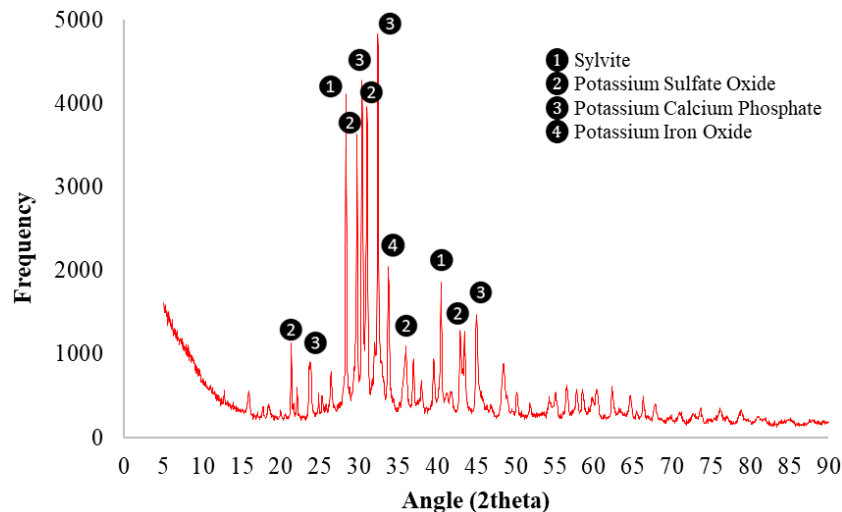


Fig. 6. Diffraction patterns of TWA.

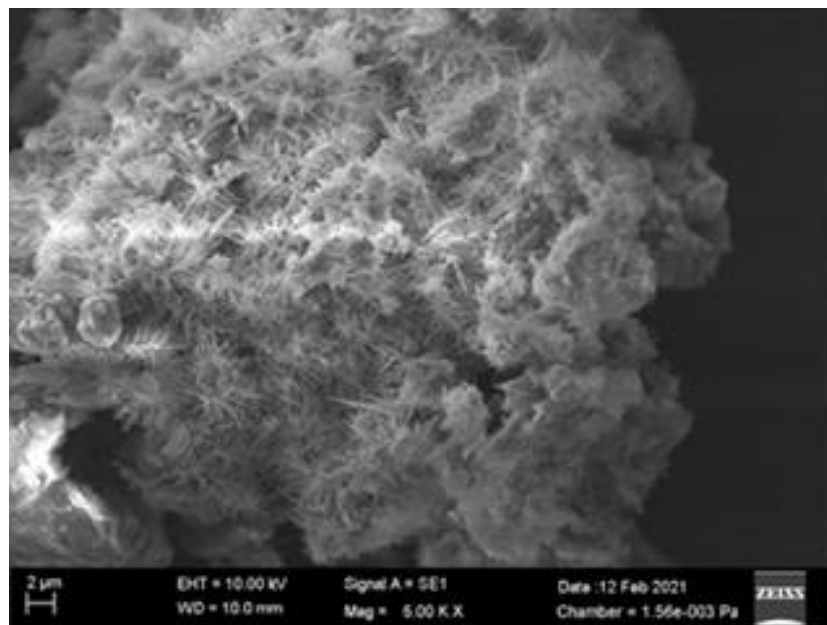


Fig. 7. SEM image of TWA (x5000).

### 3.2. Pozzolanic activity of TWA

As a result of the pozzolanic activity test, the compressive strength of the TWA substituted mortars was obtained as 2.20 MPa on the 7th day. In this context, the waste ash material does not show pozzolanic properties. This result supports each other with XRD diffraction patterns, which is one of the characterization features.

### 3.3. Standard consistency water of blended cement pastes

The amount of water determined for the standard consistency of cement pastes produced with TWA is presented in Fig. 8. The amount of water required for standard consistency of blended cement pastes increased significantly with TWA substitution. While 2% TWA substitution increased the consistency water requirement by 14.07%, 10% TWA substitution increased the consistency

water requirement by 71.36%. The fact that the specific surface area of TWA is greater than that of cement can be explained as the reason for the increased water requirement with the increase in the replacement ratio. As ashes are hygroscopic in nature (Ganesan et al. 2007) and the specific surface area of TWA is three times higher than cement it needs more water for proper consistency. Tekin et al. (2021) found that the amount of water required for consistency increased as the substitution rate increased in the cement pastes they produced with pistachio shell ash substitution. In another study, an increase in water requirement was observed with the use of hazelnut shell ash (Baran et al. 2020). This situation was explained in the literature by the increase in total surface area after modification with ash particles that are finer than cement. Furthermore, it has been noted that irregular shapes and pores contribute to an increased water requirement (Garcés et al. 2008).

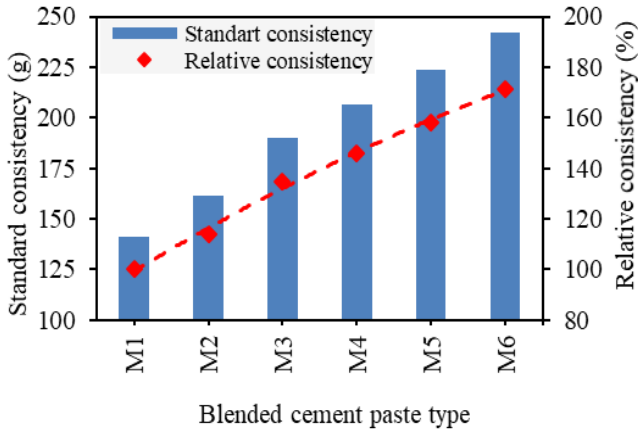


Fig. 8. Standard consistency water of the blended cements.

3.4. Setting time of blended cement pastes

Initial and final setting times of the mixtures are shown in Fig. 9. It is seen that the setting times decrease as the amount of TWA increases. While the initial setting time was 230 min. in the M1, it was measured as 215, 195, 130, 80 and 45 min. with 2, 4, 6, 8 and 10% TWA substitution, respectively. It is observed that the initial

setting decreased by 80.43% compared to the reference sample when the TWA substitution was 10%. Therefore, it is thought that TWA can be used as a setting accelerator additive in addition to its other properties explained in more detail in this study. It is known that the alkalis (Na<sub>2</sub>O and K<sub>2</sub>O) in TWA are higher than cement, which has an effect on the setting time. Because alkalis accelerate the setting of cement-based materials (Jawed and Skalny 1978). It is seen in Table 3 that there is an increase in the K<sub>2</sub>O amounts of cement pastes with TWA substitution. While the K<sub>2</sub>O amount in the theoretical chemical composition of M1 is 0.79%, the K<sub>2</sub>O amount of M6 was calculated as 3.84% with 10% TWA substitution. As a result of the detected changes, the conformity value of the initial setting time is specified as 60 minutes and above in the relevant standard (TS EN 197-1 2012). Except for the M6 mix, all of the mixes were found to have a set onset time above the specified value (Fig. 6).

When the final setting times are examined, the final setting accelerating effect of TWA is again seen. The final setting times of the samples with 0, 2, 4, 6, 8 and 10% TWA substitution were measured as 270, 260, 235, 175, 120 and 75 minutes, respectively. With the use of 10% TWA, the final setting time decreased by 72.22% compared to the reference. There is no value for the final setting time in the relevant standard (TS EN 197-1 2012).

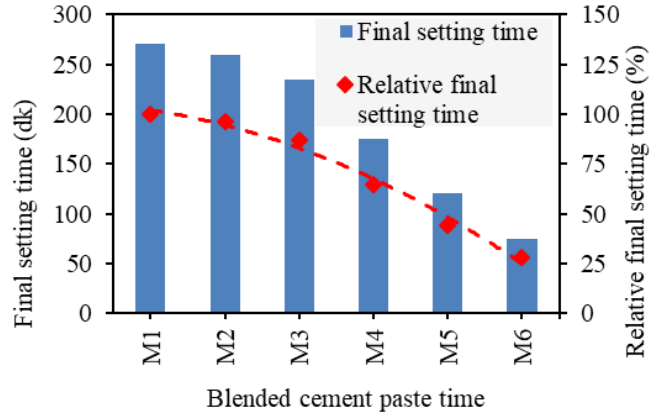
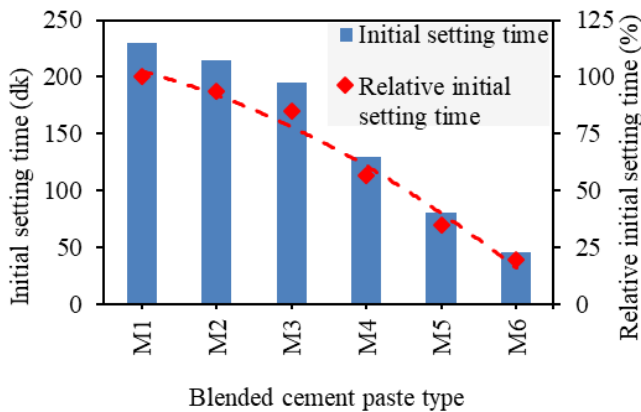


Fig. 9. Initial and final setting time values of the blended cements.

3.5. Volume expansion of blended cement pastes

The amount of possible expansion that may occur as a result of the reaction of free CaO and MgO in TWA-substituted cement samples with water should be below 10 mm according to the TS EN 197-1 (2012) standard.

As a result of the study, it was observed that the expansion values did not exceed the limit value as seen from Table 4. While the expansion amount of the M1 sample is 1 mm, it is seen that the expansion amount is 3

mm in the M6 sample with 10% TWA substitution. The increase in MgO ratios in the theoretically calculated chemical analyzes of the mixtures formed with TWA is given in Table 3.

In the theoretical calculation, the MgO ratio in the M1 mixture is 2.36%, while the MgO ratio in the M6 mixture is 2.67%. Therefore, it is possible to explain the increase in volume expansion with the increase in the MgO ratio that reacts. This circumstance aligns with the existing literature (Bekem Kara 2022).

Table 4. Chemical properties of cement.

Mix ID	M1 (OPC)	M2	M3	M4	M5	M6
Expansion (mm)	1.0	1.0	1.0	1.5	2.0	3.0

#### 4. Conclusions

Studies on the recyclability of agricultural wastes in cementitious systems have attracted attention in recent years. In this study, tea waste ash was characterized in detail and the properties of cement pastes blended with tea waste ash were investigated. In accordance with the experimental findings, the following results were obtained:

- Tea waste ash is of potassium origin, mostly in crystalline form. The specific surface area is quite high due to the needle-like structure obtained in SEM image. The main component of ash in chemical composition is  $K_2O$ .
- Tea waste ash substitution has increased standard consistency water of blended cement pastes. 10% tea waste ash reduces the initial setting time by up to 80.43% and the final setting time by up to 72.20%. Ash substitution up to 8% provided the initial setting time included in the standard. It is thought that tea waste ash can be used as a setting accelerator additive in cement systems, even in small amounts, due to its high  $K_2O$  content.

Waste tea fibers are one of the agricultural wastes that need to be disposed of in tea production areas. It is thought that the use of these wastes, which is an important issue for the Eastern Black Sea Region where tea farming and tea factories are located in Türkiye, is an important issue. It is recommended to investigate the strength, thermal and durability performance properties of mortars and concretes with tea waste ash substitute. In addition, tea waste ash can be used in geopolymer production due to their high alkali content.

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#### Conflict of Interest

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this manuscript.

#### Author Contributions

All of the authors made substantial contributions to conception and design, or acquisition of data, or analysis and interpretation of data; were involved in drafting the manuscript or revising it critically for important intellectual content; and gave final approval of the version to be published.

#### Data Availability

The datasets created and/or analyzed during the current study are not publicly available, but are available from the corresponding author upon reasonable request.

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