

## Effect of Fly Ash and Metakaolin Substituted Forms on Structural Properties in Light Mortar with Pumice Aggregate

Fatime Zehra ÇİÇEK<sup>1</sup>, Recep Kadir PEKGÖKGÖZ<sup>1\*</sup>, Sümeyye Büşra KAZANASMAZ<sup>1</sup>, Ali SARIŞIK<sup>1</sup>, Fatih AVCİL<sup>2</sup>



<sup>1</sup>Department of Civil Engineering, Harran University, Şanlıurfa, Türkiye

<sup>2</sup>Department of Civil Engineering, Bitlis Eren University, Bitlis, Türkiye

(ORCID: [0000-0002-0599-410X](https://orcid.org/0000-0002-0599-410X)) (ORCID: [0000-0002-3083-2241](https://orcid.org/0000-0002-3083-2241)) (ORCID: [0009-0009-3255-8213](https://orcid.org/0009-0009-3255-8213))

(ORCID: [0000-0001-7698-6134](https://orcid.org/0000-0001-7698-6134)) (ORCID: [0000-0001-6550-550X](https://orcid.org/0000-0001-6550-550X))

**Keywords:** Pumice, Fly ash, **Abstract**

Metakaolin, Composite mortar, Standard Sand

In this study, experimental tests were conducted on composite mortars in different forms prepared using lime, pumice, fly ash, and metakaolin. This research aims to recycle fly ash produced as industrial waste and produce a building material that is durable, lightweight, and provides good insulation. In the composite structure mortar mixture designs prepared for the experiments, CEN standard sand was replaced with Nevşehir pumice (0.5-4 mm) at the rates of 10%, 20%, and 30%, respectively, and fly ash-metakaolin was replaced with the cement at the rates of 10%, 20%, and 30%, respectively. A total of 13 different mixtures were prepared. Unit volume mass test, Ultrasonic test, water absorption test, compressive strength test, flexural strength, thermal conductivity coefficient test, scanning electron microscope (SEM) analysis, and thermogravimetric - differential thermal (TGA/DTA) analysis were performed on the prepared samples. As a result of the research, it was determined that the mortar mixture obtained by substituting 30% pumice with standard sand and 30% fly ash and metakaolin with cement provided the highest compressive strength, lowest dry unit volume weight, and thermal conductivity coefficient compared to other mortar mixtures.

### 1. Introduction

In recent years, it has been observed that cement mortars in our country are insufficient to provide the required performance in construction applications in many respects. For this reason, the use of new-generation composite cement mortars is becoming widespread. Mortars used in the construction industry are generally normal-weight mortars prepared with cement, aggregate, water, and various chemical additives [1]. However, many R&D (Research and Development) studies are ongoing to improve the technical properties of new-generation composite mortars with lightweight aggregates with different characteristics and different binder uses [2]. Studies continue to improve the properties of plaster mortar by substituting alternative materials for both cement

and sand [3]. One of these alternative materials is pumice, a lightweight aggregate. Pumice aggregate is generally used in the construction industry in the world and in Türkiye. The main reason for this is its low unit volume weight, high heat and sound insulation, air-conditioning feature, excellent acoustic feature, elasticity against earthquake load and behavior, and being more economical than its alternatives. The use of lightweight aggregates in cementitious mortars is increasing significantly due to the energy savings they provide in production, transportation, heat, and sound insulation [4]. Since the earthquake forces affecting the structures vary in proportion to the weight of the structures, it is extremely important to use lightweight materials to reduce the loss of life and property, so that the structures are less affected by earthquakes. For this

\*Corresponding author: [recepkdir@harran.edu.tr](mailto:recepkdir@harran.edu.tr)

Received: 06.05.2024, Accepted: 28.06.2024

reason, the importance of using lightweight aggregate in cementitious mortars is better understood. Particularly porous lightweight aggregates are an important raw material in the development of thermal insulating mortars [5]. In this study, pumice was substituted with CEN standard sand, and fly ash, and metakaolin were substituted with cement. The materials we produce are intended to be economical, provide heat and sound insulation, have high strength, and are lightweight. Rahman et al., in their study with pumice, showed that the samples produced had 9.8% higher compressive strength and 36% lower porosity than the control sample [4].

Lime was one of the most common building materials used as a binder in the production of mortar until the 1990s. It is known that lime was used as a binder in all historical buildings that have survived to the present day [7, 8]. It is known that Pozzolanas, which consist of natural and artificial substances that give hydraulic properties to mortars when mixed with lime, have been used in the construction of water-resistant structures in water and humid regions since ancient times [8]. In their study, Gülbe et al. examined the effect of white cement on the properties of lime mortars. They determined that the mechanical strength of the mortar increased significantly as the amount of cement in lime mortar mixtures increased [9]. Veiga et al. created separate mortar mixtures with air lime, natural hydraulic lime, white cement, silica fume, metakaolin, and natural pozzolana from the Cabo Verde region. They investigated the experimental application and performance evaluation of mortars with mortars with added additives. As a result, they observed the highest compressive strength in lime-white cement mortars in hardened mortar samples [10].

With the studies carried out in the academic world, due to environmental protection and sustainable construction requirements in the future; It has been shown that the use of by-products such as fly ash, granulated blast furnace slag, silica fume, rice husk ash and metakaolin in cement and concrete has gained great importance [11]. Turk et al. stated that generally the use of these mineral additives reduces and improves permeable voids through micro-filling effect, pozzolanic reaction, and densification of the cement paste-aggregate interface area [12]. Using fly ash instead of some cement also contributes to the protection of the environment by reducing the amount of natural raw materials used in cement production [13]. In this paper, fly ash and metakaolin waste materials were used as binders. Fly ash is the waste material formed by burning hard coal in thermal power plants built to produce energy and kept in chimneys with the help of filters. Fly ashes alone have

little to no binding properties. For this reason, they are combined with slaked lime to create a chemical reaction and gain hydraulic binding properties. If fly ashes contain lime components in their structure, their strength increases as a result of their interaction with water, and they can gain durability. The increases in strength values that may occur very slowly in fly ashes form the basis of the pozzolanic properties of fly ashes [14]. Fly ashes, which have very high surface area values, do not conduct heat and electricity well because they are empty and spherical in micro dimensions. Therefore, fly ashes are known to be good insulators. Metakaolin is a substance obtained as a result of the calcination of clay and used in many areas of concrete production. [11]. Ahmed et al., stated in their study that metakaolin is an environmentally friendly alternative binder to Portland cement [15]. Various studies have proven that metakaolin contributes to the improvement of the properties of mortars by combining with calcium hydroxide, thanks to the high silica and alumina content it contains [15], [16].

## 2. Material and Method

In the research conducted, in the production of the control mortar series; CEM II A-LL 42.5 R Portland calcareous composite cement, lime, and CEN standard sand complying with TS EN 196-1 [17] were used. It is known that hydrated lime improves the workability and adhesion properties of mortars. Many studies have been conducted on how the use of hydrated lime affects the water requirement in mortars. It has been determined that in the mortars created by mixing a small amount of lime (0%~20%) with natural pozzolans, the lime particles fill the gaps between the natural pozzolan particles by acting as a filler. According to this evaluation, the water needs of lime-pozzolan mixtures are not higher than natural pozzolan mixtures. However, it is assumed that after the lime rate exceeds 20%, more water will be needed to wet the lime particles and fill the gaps between the particles [18]. For these reasons, the lime rate was kept constant at ~14% in this study. The basic characteristics of the lime used are given in Table 1.

**Table 1.** Basic characteristics of lime

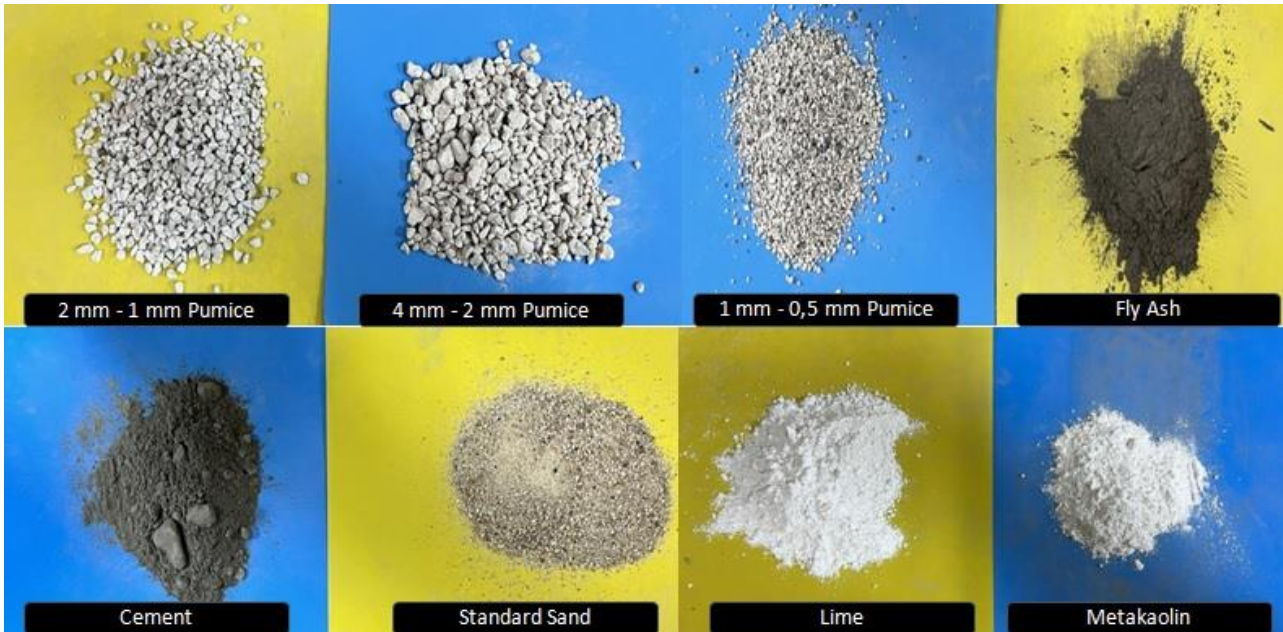
Basic Characteristics	Performance
Grain Size Distribution	
0.009 mm	$\leq 7$
0.2 mm	$\leq 2$
Volume Constancy	$\leq 2$
Penetration/Water requirement	$\geq 10$ and $\leq 50$
CaO+MgO	$\geq 80$
MgO	$\leq 5$
CO <sub>2</sub>	$\leq 7$
SO <sub>3</sub>	$\leq 2$
Free Lime	$\geq 65$

physical and chemical properties of the cement used are given in Table 2.

**Table 2.** Physical and mechanical properties of cement

Fineness (45 $\mu$ m, above sieve, %)	5.7	
Specific mass (g/cm <sup>3</sup> )	3.00	
Specific surface (cm <sup>2</sup> /g)	3997	
The initial setting of cement	2h-50 min	
The final setting of cement	3h-30 min	
Compressive strength (MPa)	Day 2	22.00
	Day 28	51.70

As shown in Figure 1, Nevşehir pumice (0.5-4 mm), fly ash, and metakaolin were used in the proportions of 10%, 20%, and 30% in the mortar mixture designs in the composite structure. The

**Figure 1.** Materials used in mixtures

To provide lightness to the produced composite mortars, 3 different mixtures were prepared by substituting pumice at 10%, 20%, and 30% of the sand volume instead of standard sand. The Nevşehir pumice used is between 0.5 and 4 mm in size and the physical properties of each granulometry have been determined by experiments and are shown in Table 3. The sieving method of Nevşehir pumice was made in accordance with TS EN 933-1 [19] standard. TS EN 1097-3 [20] and TS EN 1097-6 [21] standards were used to determine the physical properties of aggregates. 9 different mixtures were

prepared by substituting fly ash, metakaolin, and fly ash-metakaolin as binders in the proportions of 10%, 20%, and 30% of the cement volume instead of cement. A total of 13 different composite mortar mixtures were produced.

**Table 3.** Physical Properties of Nevşehir Pumice

	Aggregate Size (mm)		
	1-0.5	2-1	4-2
Saturated unit volume mass (g/cm <sup>3</sup> )	1.13	1.08	1.00
Dry unit volume mass (g/cm <sup>3</sup> )	0.91	0.85	0.77
Water absorption (%)	23.59	26.35	30.11
Compact bulk density (g/cm <sup>3</sup> )	0.485	0.475	0.469
Loose bulk density (g/cm <sup>3</sup> )	0.422	0.448	0.451
Aggregate moisture content (%)	0.30	0.30	0.40

City tap water was used in all mixtures. The chemical properties of pumice, fly ash, and metakaolin used in the mixtures are given in Table 4, and their specific mass is given in Table 5.

**Table 4.** Chemical Properties of Pumice, Fly ash, and Metakaolin

Contents(%)	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	P <sub>2</sub> O <sub>5</sub>	K <sub>2</sub> O	SO <sub>3</sub>	SrO	Na <sub>2</sub> O	TiO <sub>2</sub>	SiO <sub>2</sub>	NiO	Loss of ignition
Metakaolin	53.0	43.1	0.7	0.5	0.1	0.1	0.1	0.0	0.0	0.1	2.0	0.0	-	0.2
Fly Ash	28.4	14.6	11.4	23.3	2.2	0.1	1.4	6.7	0.1	0.4	0.6	0.1	0.1	10.6
Pumice	73.2	12.3	1.1	0.7	0.1	-	4.2	0.0	-	3.6	0.1	0.0	-	4.5

## 2.1. Mixture Calculation

The water-solid ratio in the mixtures was kept constant at 0.20. In the mixtures, Nevşehir pumice 4-2 mm was used at a rate of 50%, 2-1 mm at a rate of 30%, and 1-0.5 mm at a rate of 20%. They are named "Control mortar "CS", Pumice "P10, P20, P30", Fly Ash "FA10, FA20, FA30", Metakaolin "MK10, MK20, MK30" and All (Pumice + Fly Ash + Metakaolin) "ALL10, ALL20, and ALL30".

**Table 5.** A specific mass of Fly ash, Metakaolin, and Lime

	FA	MK	Lime
Specific mass (g/cm <sup>3</sup> )	2.18	2.03	2.064

In mortar mixtures containing pumice (P10, P20, P30) and containing all components (ALL10, ALL20, ALL30), pumice was used as lightweight aggregate instead of sand by substituting 10%, 20%, and 30% of the sand volume, respectively. In mortar mixtures containing fly ash (FA10, FA20, FA30), metakaolin (MK10, MK20, MK30) and all components (ALL10, ALL20, ALL30), binders were used by substituting 10%, 20% and 30% of the cement volume. Thus, a total of 13 different mixtures (CS, P10, P20, P30, FA10, FA20, FA30, MK10, MK20,

MK30, ALL10, ALL20, ALL30) were prepared. The materials used in the mortar mixture are given in Table 6.

For mortar mixtures in which only sand was used as aggregate, sand, lime, and binders were taken into the mixing bowl and mixed dry for 3 minutes. Then, 2/3 of the mixture water was added and mixed at high speed for 2 minutes. Finally, all the mixing water was added and mixed at high speed for 1 minute. In the mortar mixtures using sand and pumice as aggregates, sand and pumice aggregates of different sizes were taken into the mixing bowl, 1/3 of the mixture water was added and mixed for 3 minutes. Then, lime and binders (cement, fly ash, and metakaolin) were added and mixed at high speed for 2 minutes, using all the mixing water. For all mixtures, fresh mortar taken from the mixer was poured into 40x40x160 mm prism size and 50x50x50 mm cube molds according to TS EN 998-1 [22]. Mortar samples were compressed on the vibration table for 2 minutes and kept in the mold for 24 hours. It was then removed from the molds and left to cure in water for 7, 14, and 28 days. Unit volume mass analysis, compressive strength, flexural strength, thermal conductivity analysis, ultrasound test, water absorption test, SEM analysis, and TGA/DTA analysis were performed on the samples that completed the curing period following the standards.

**Table 6.** Materials Used in 1 m<sup>3</sup> Mortar

Code	Standard sand (kg)	Lime (kg)	Cement (kg)	Water (kg)	Pumice (kg)	Metakaolin (kg)	Fly ash (kg)
CS	1000	280	400	340			
P10	900	280	400	340	100		
P20	800	280	400	340	200		
P30	700	280	400	340	300		
MK10	1000	280	360	340		40	
MK20	1000	280	320	340		80	
MK30	1000	280	280	340		120	
FA10	1000	280	360	340			40
FA20	1000	280	320	340			80
FA30	1000	280	280	340			120
ALL10	900	280	360	340	100	20	20
ALL20	800	280	320	340	200	40	40
ALL30	700	280	280	340	300	60	60

### 3. Results and Discussion

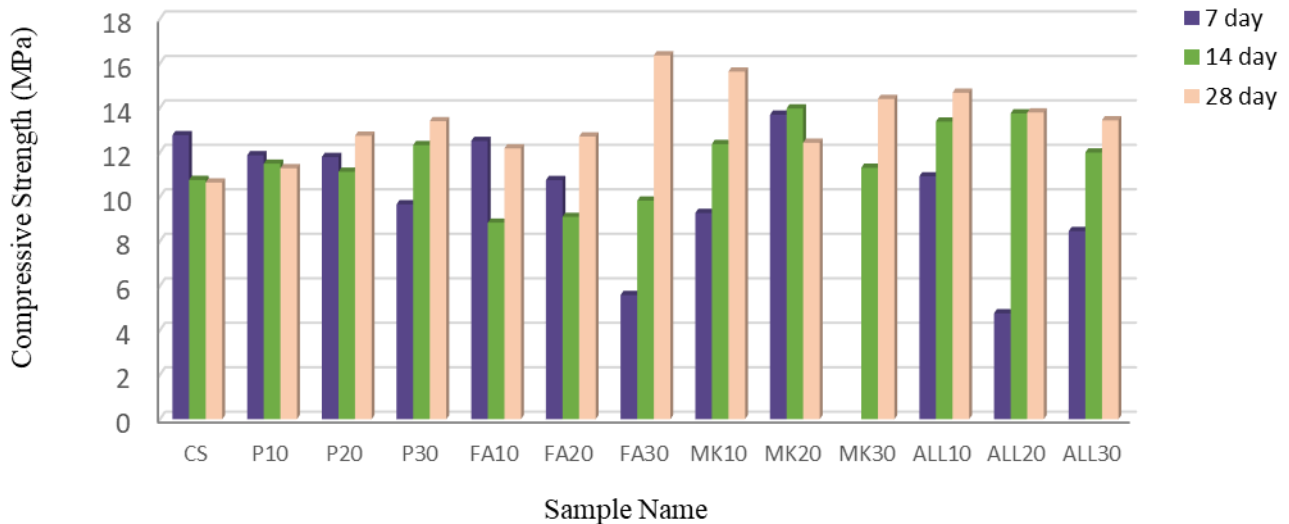
#### 3.1. Compressive Strength Analysis

For each of the 13 different composite mortar mixtures prepared, the compressive strength tests of 3 cube samples of 50x50x50 mm dimensions after 7, 14, and 28 days of curing were carried out following the TS EN 1015-11 [23] standard. In these tests, the crushing operations were carried out perpendicular to the mortar casting direction in accordance with the relevant standards. In the study, the 7, 14, and 28-day compressive strength of the samples was calculated

according to Equation 1. The arithmetic average of the obtained values was taken as shown in Figure 2.

$$f_c = \frac{F}{A_c} \quad (1)$$

In the statement given above;  $f_c$  is the compressive strength (MPa),  $F$  is the maximum load reached at the moment of fracture (N) and  $A_c$  is the cross-sectional area (mm<sup>2</sup>) where the pressure load is applied on the sample.

**Figure 2.** Compressive strength of the samples

In the TS EN 998-1 standard, four separate strength classes are prescribed for the 28-day compressive strength values of mortar groups (CS I - CS IV). The strength limits in these classes are given in Table 7:

**Table 7.** 28-day compressive strength values of mortar groups [1]

The strength class	28-day mortar strength range (MPa)
CS I	0.4 – 2.5
CS II	1.5 – 5.0
CS III	3.5 – 7.5
CS IV	≥ 6

When Figure 2 is examined, the highest compressive strength result of the 7-day samples belongs to the MK20 mortar mixture, and it is seen that the metakaolin used increases the compressive strength. When the results of 14-day samples were examined, the compressive strength value of the MK20 mortar mixture gave the best result with 13.97 MPa. However, the results for ALL10 and ALL20 mortar mixtures were 13.38 MPa and 13.75 MPa, respectively, and a lighter and higher compressive strength building material was obtained. When previous studies were examined, it was observed that the compressive strength increased in samples produced by substituting 10-20% metakaolin with cement in mortar production [24], [25]. However, when replacing metakaolin with cement, it has been observed that the compressive strength decreases above 30% MK [26]. Among the samples, the highest compressive strength was obtained with 16.36 MPa in the FA30 mortar mixture in the 28-day curing results. Many studies have shown that the use of fly ash deteriorates the early-age concrete strength, but generally increases the strength and durability of composites over time, as it consumes the  $\text{Ca(OH)}_2$  produced during the hydration of cement and forms secondary hydrates such as CSH [27, 28, 29]. It was also observed that it had a positive effect on the 28-day compressive strength of the samples in which fly ash was used instead of cement. When the evaluation of lightweight aggregates for the production of composite mortar is analyzed, it is envisaged in the TS EN 998-1 standard that mortars with low unit volume mass values will be included in the CS I and CS II classes in terms of 28-day compressive strength class [2]. When the 7, 14, and 28-day compressive strength values of the ALL20 mortar mixture, in which pumice was used as lightweight aggregate,

were examined, it was seen that they were 4.76 MPa, 13.75 MPa, and 13.79 MPa, respectively. When this situation was evaluated, it was determined that all of the mortars we produced were included in the CS IV mortar strength class.

### 3.2. Flexural Strength Analysis

In the study, beam samples of 40x40x160 mm were prepared from composite mortar mixtures for flexural strength tests. When the samples completed their 14-day cure in the curing pool, flexural strength analysis was performed following TS EN 1015-11 [23] standard. The longitudinal axis of the sample was placed perpendicular to the longitudinal axis of the lower and upper loading cylinders and was placed exactly centered as shown in Figure 3. Flexural strength was calculated using Equation 2.

$$f = \frac{3 \times F \times L}{2bh^2} \quad (2)$$



**Figure 3.** Three-point flexural test

In this expression;  $f$  is the flexural strength ( $\text{N/mm}^2$ ),  $F$  is the ultimate load (N),  $L$  is the distance between the support cylinders (mm),  $b$  is the beam width (mm) and  $h$  is given as the beam height (mm).

In the study, the test results of the composite mortar mixtures that completed their 14-day cure are shown in Figure 4. Flexural strength is an important mechanical property in exterior plaster applications. When Figure 4 is examined, the highest flexural strength of the 14-day-old samples was obtained in the P30-coded samples. It was concluded that as the ratio of pumice used as lightweight aggregate in the mixtures increased, the flexural strength improved. When looking at the mortar mixtures using metakaolin, it was observed that their flexural strengths were lower compared to other mortar mixtures.



**Figure 4.** Flexural strength of the composite mortar mixtures

### 3.3. Water Absorption Analysis

In the water absorption test, the prepared composite mortar samples with dimensions of 50x50x50 mm were kept in the oven at 105°C for 24 hours. The composite mortar samples taken out of the oven were left to cool at room temperature. After the cooled samples were weighed and their weights were recorded, they were left to cure in 20°C water for 24 hours. The samples were removed from the water after 24 hours and placed on a dry towel to remove moisture. The samples were then weighed, and their saturated weights were recorded. The weight water absorption percentages of the samples were calculated using Equation 3.

$$\delta_k = \frac{G_d - G_k}{G_k} \times 100 \quad (3)$$

In this equation,  $\delta_k$  denotes the water absorption percentage by weight (%),  $G_d$  denotes the water-saturated sample weight, and  $G_k$  denotes the oven-dry sample weight.

In the study, experiments were carried out on three cube samples each to obtain the 14 and 28-day water absorption test results of the samples. These results were then averaged and shown in Figure 5.

When the graphs in Figure 5 showing the test results are examined, according to the water absorption percentage, the highest water absorption value in the 14-day samples was obtained in the ALL30 mixture at 19.77%. It has been observed that this value is related to the dry unit volume mass value of the 14-day T30 mixture. It was observed that the water absorption value increased as the dry unit volume mass decreased. It can be said that the reason for this is that lightweight materials are more porous. Similar results were also observed in 28-day samples.

When the 14-day results of the water absorption test are examined, the water absorption amount increases by 20% in the ALL30 sample compared to the control samples, and it has the highest water absorption amount. The P10 sample gave the lowest value, measuring 6.7% less water absorption than the control sample. When evaluated regarding control samples for 28-day results, the FA30 sample gave the highest water absorption amount with a 20% increase, while the P10 sample gave the lowest value with a 3.8% decrease.

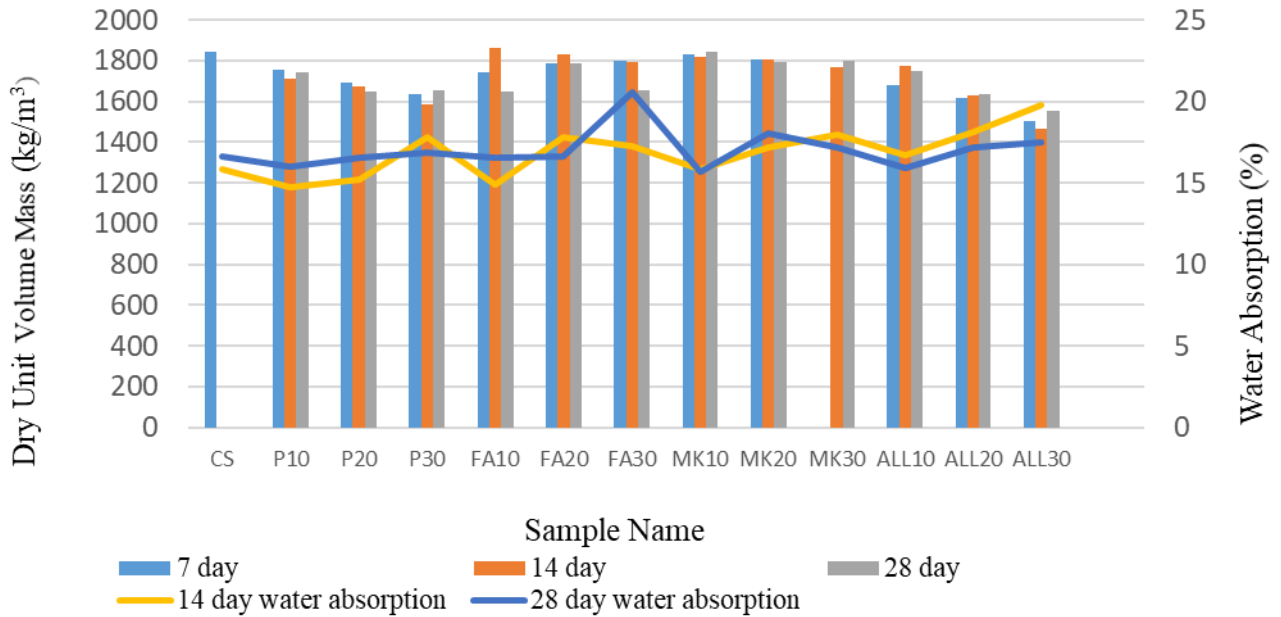


Figure 5. Water absorption test results

### 3.4. Dry Unit Volume Mass Analysis

In this experiment, which was carried out following the TS EN 1015-10 [30] standard, cube samples of 50x50x50 mm were left in the oven at 105 °C for 24 hours, then removed from the oven and allowed to cool at room temperature. The oven-dry weights of the cooled samples were weighed on a scale and the readings were recorded. The dry unit volume mass of the samples was calculated using Equation 5.

In this expression,  $\rho$  is the unit volume mass of the sample (kg/m<sup>3</sup>),  $M_{dry}$  is the oven-dry mass of the sample at the end of 24 hours (kg), and  $V$  is the volume of the cube samples of 50×50×50 mm size (m<sup>3</sup>).

$$\rho = \frac{M_{dry}}{V} \quad (5)$$

When the results of the unit volume mass values of the control mortar samples after 7, 14, and 28 days of curing, determined according to TS EN 12390-7, were examined, the unit volume mass value of the control sample (CS) was higher because it did not contain light aggregate. It can be seen in the graph in Figure 5 that the unit volume mass value decreases in the ALL-mixture series in which pumice, fly ash, metakaolin, and all materials are used. This is a result of reducing the amount of cement and sand in the mixtures by substituting less dense fly ash and metakaolin with cement, and standard sand with

pumice. It has been observed that the use of pumice provides lightness to a certain extent in mortar series. Binici, [31] in his study, showed the positive effect of using pumice in composite mortars on unit volume mass results. Today, both sustainability and economy will be achieved by choosing light aggregate mortars instead of traditional mortars in the interior and exterior spaces of buildings.

### 3.5. Thermal Conductivity Analysis

The thermal conductivity test was carried out following the ISO 22007-2 standard [32]. In this experiment, 50x50x50 mm cube samples, which were kept in the curing pool for 28 days, were kept in the laboratory until they reached the appropriate temperature before starting the experiment. The sensor was contacted to the flat and smooth surface of the 50x50x50 mm sample placed on the 3 cm diameter sensitive sensor of the Hot Disk brand device called TPS 500 S, and the temperature between the sensor and the sample was balanced and the heat conduction value ( $k$ ) was obtained in W/mK. Analysis results are given in Figure 6.



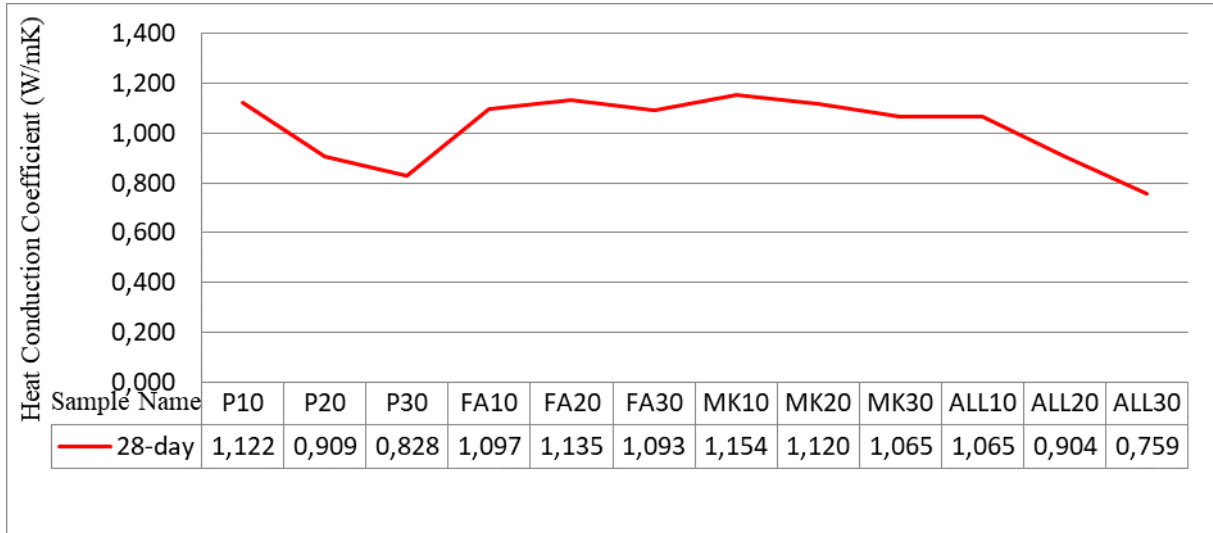


Figure 6. Thermal conductivity coefficient test results

When the results were examined, it was observed that the thermal conductivity coefficients of the samples using pumice aggregate were lower than the other samples. The lowest thermal conductivity coefficient was observed in the sample coded ALL30, and the positive effect of pumice aggregate was observed. It was understood that the reason why the thermal conductivity coefficient of the samples using pumice was lower than the other samples without pumice was due to the porosity of the pumice aggregate.

### 3.6. Ultrasonic Test

Ultrasound tests were carried out on the produced samples after 7, 14, and 28 days of curing, according to the TS EN 12504-4 [33] standard. First of all, the sample is placed between the probes of the UPV

device, with the casting surface of the sample to be tested on top. At this location, the sound velocities of the samples were measured. In order to make the measurement results more precise, measurements were made on three cube samples of 50x50x50 mm from two different directions, and the average value was taken. Sound transmission speed was calculated by dividing the sample length by the transition speed time according to Equation 6.

$$v = \frac{l}{t} \tag{6}$$

In this expression,  $v$  is expressed in terms of sound speed (m/s),  $l$  is expressed in sample size (m) and  $t$  is expressed in transition speed time (s). The results are summarized in Figure 7.

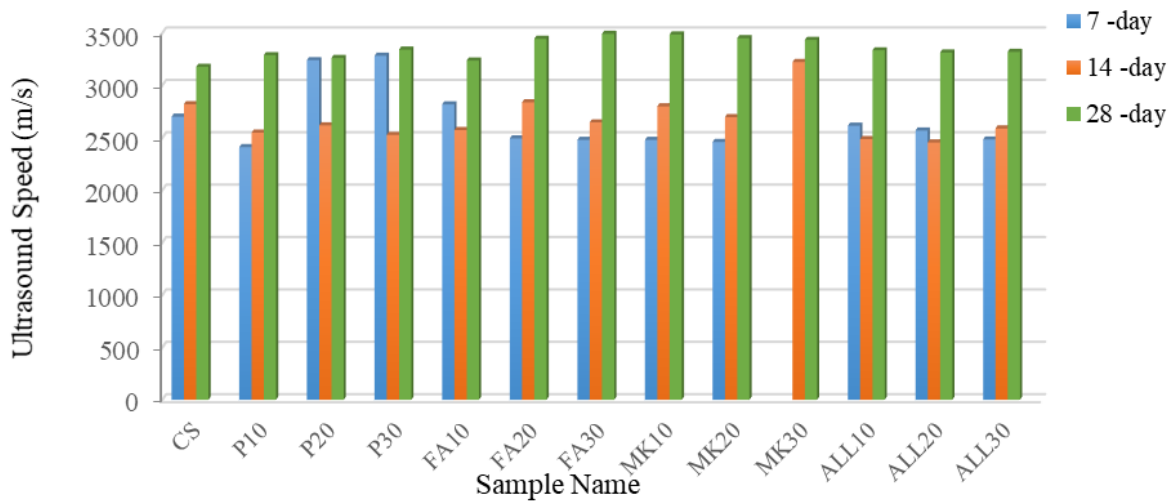


Figure 7. Ultrasound speed test results

Ultrasound testing is a method that gives an idea about the strength of materials in terms of existing voids and cracks [34]. When the ultrasound transmission rate results were examined, it was observed that the 14-day cure result was lowest in the ALL20 mortar mixture. It has been observed that the porous structure of pumice as an aggregate positively affects the sound insulation properties of the mortar. When the results of the ultrasound test are examined, taking the control samples as reference, the MK20 sample gave the lowest value with a 9% decrease for the 7-day results, while the P30 sample gave the highest value with a 17.7% increase. For 14-day results, the ALL20 sample gave the lowest value with a 13% decrease, while the MK30 sample gave the highest value with a 12.5% increase. Although there was no significant difference between the 28-day results of the samples compared to CS, the most significant increase was 9% in the FA30 sample.

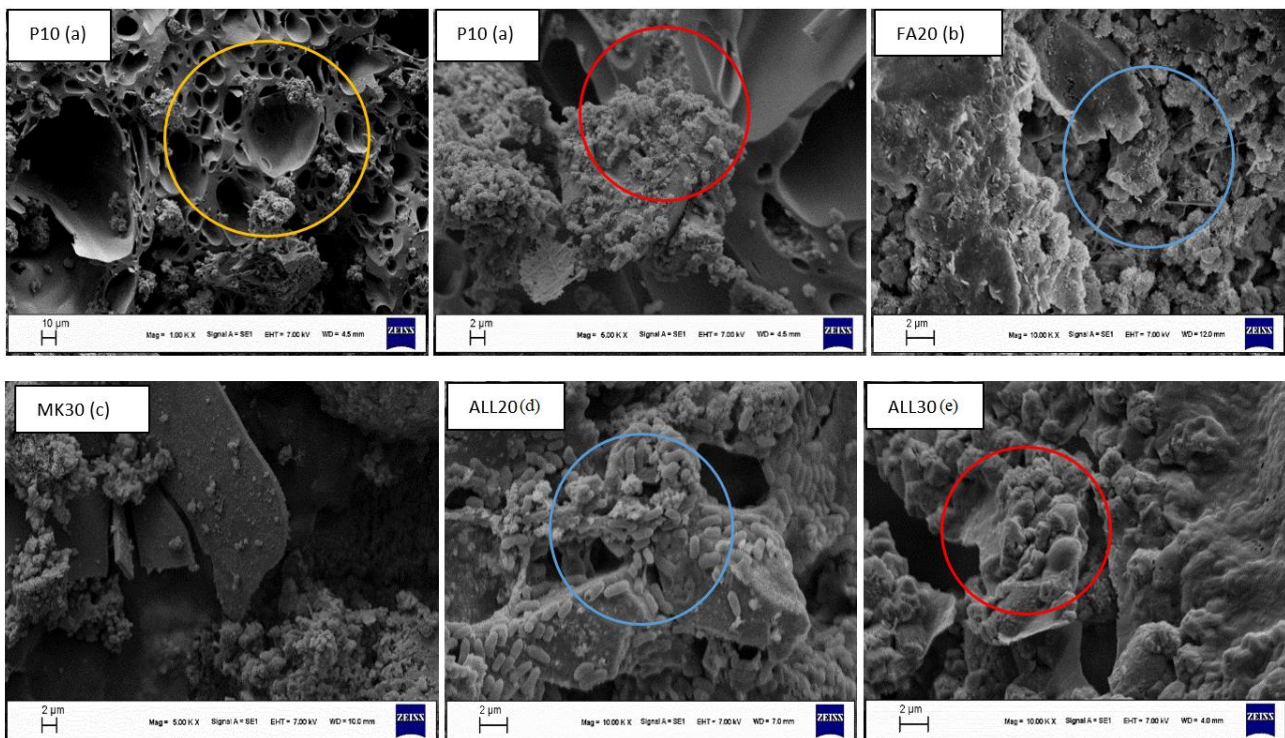
In general, from the graph given in Figure 7, it can be seen that the ultrasound transmission rate values of the 28-day-old samples are higher than the 7- and 14-day-old samples. This shows that after 28 days of curing, the samples have largely completed their hydration, and their compressive strength has increased.

### 3.7. SEM Analysis

The morphological features of the samples were characterized using a scanning electron microscope

(ZEISS EVO50 SEM) on a Secondary Electron (SE) detector at a distance of approximately 4-11 Work Distance (WD) at an acceleration voltage of 7 kV. After the test samples were subjected to the pressure test, the sample samples taken from the broken surface were carefully taken and packaged in ziplock bags, and then the samples were made conductive at a thickness of 5 nm in a gold plating device (electron microscope system) for electron microscope imaging. Images of the test samples were taken from regions where the materials used (pumice, fly ash, metakaolin, lime, standard sand, and cement) were dense and where they contacted each other and formed interfaces. Analyzes were carried out at Harran University Science and Technology Application and Research Center (HUBTAM). The magnification ratio was chosen as 5.00KX and 10.00KX, and the cross-sectional images have a magnification ratio of 250X.

When looking at the SEM analysis in Figure 8 (a), the porous structure of the pumice draws attention to the mixture containing 10% pumice, while lime and cement grains stuck to the pores on the surface of the pumice were observed in the second image. In Figure 8 (b), samples with 10%, 20%, and 30% fly ash added, it was observed that the ashes were shaped in a spherical shape and the pores were completely covered with ashes. As the amount of fly ash increases, it is seen in the SEM images that it covers the surface of the cement in a spherical manner.



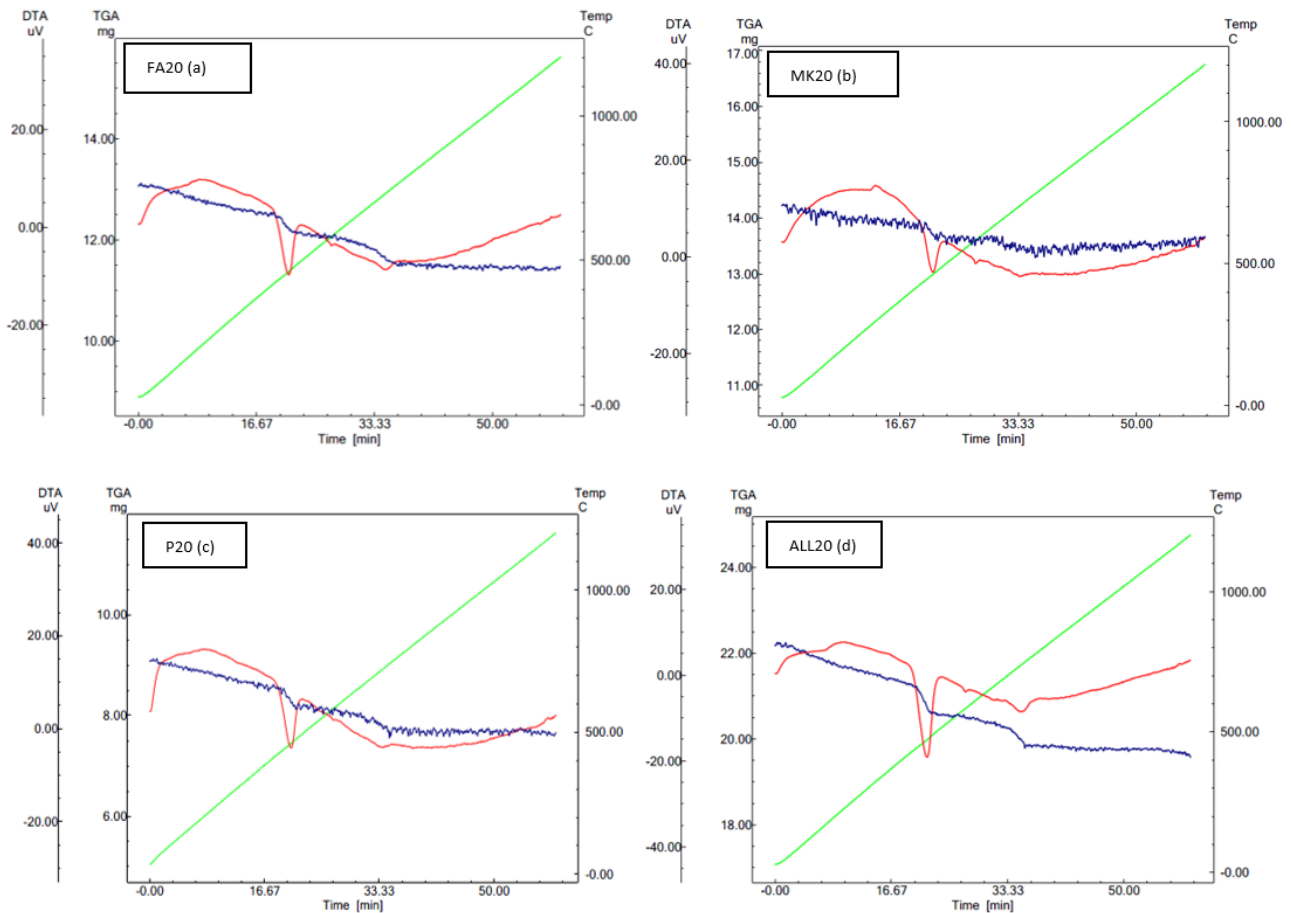
**Figure 8.** SEM Analysis of the samples coded P10 (a), FA20 (b), MK30 (c), ALL20 (d), and ALL30 (e)

Since pumice aggregate is a light material with a rough and porous structure, it is possible to adhere to the aggregate in the paste phase. This situation caused the interface region to not be clearly understood in the SEM images of pumice-added samples, as in some previous studies [34], [35], [36].

In the SEM images of the MK30 sample in Figure 8 (c), as the amount of metakaolin increased, metakaolin deposits were seen in the sample, and the interface that emerged when the metakaolin material seen in this mass was combined with cement was photographed. The difference in clarity in the photographs is due to the difference in elevation on the surface of the sample. Due to the presence of pumice, fly ash, and metakaolin in ALL20 and ALL30 mixtures, together with the spherical structure of fly ash and the thick and short structure of metakaolin, porosity increases were observed in the SEM images in Figure 8 (d-e) as the ratio of pumice increased.

### 3.8. TGA/DTA Analysis

TGA/DTA Analysis is performed to determine the changes that occur within the material as the temperature increases. The TGA experiment was conducted at Harran University Science and Technology Application and Research Center (HÜBTAM). For this analysis, samples that completed their 28-day cure in the cure pool were exposed to pressure testing. Then, the pieces taken from these samples were ground into powder. The powder pieces that were ready for testing were placed in thermo balance, the temperature was increased by 20°C per minute, and the mass losses (TGA) and energy losses (DTA) occurring in their structures were monitored for 1 hour as shown in Figure 9.



**Figure 9.** TGA/DTA analyzes samples coded FA20 (a), MK20 (b), P20 (c), and ALL20 (d)

With TGA/DTA analysis, endothermic and exothermic reactions of the sample against the heat it is exposed to in the experiment are determined. The red line seen in the graphs is the DTA (energy loss) line, the blue line is the TGA (mass loss) line, and the green lines are the temperature change line.

When the TGA analysis of the FA20 coded sample was examined, as seen in Figure 9(a), it was understood that there was a mass decrease in the temperature range of 680-485 °C, and in a similar case, in the MK20 coded sample, in the temperature range of 700-520 °C, as shown in Figure 9(b).

When the TGA analysis was examined, as seen in Figure 9 (c), in the P20-coded sample, a mass decrease was observed in the temperature range of 650-560°C, and a two-stage mass loss was observed in the ALL20-coded sample, as in Figure 9 (d). In the first stage, there was a mass decrease in the temperature range of 650-550°C. In the second stage, the mass decrease occurred in the temperature range of 550-430°C.

In TGA analyses, the highest mass loss occurred in the P10-coded sample and the least mass loss occurred in the MK20-coded sample. It was observed that pumice-added samples had more mass loss compared to other composite mortar samples. It is thought that the behavior of pumice aggregate against temperature is effective in this result.

According to the DTA analysis results of composite mortars, it is seen that there is an endothermic peak between 400°C and 500 °C and an exothermic peak between 750°C and 1000 °C. This peak arises from the free moisture in the system and the loss of water in the C-S-H structure. These peaks correspond to the removal of water molecules bound or free to structural compounds. When temperatures exceed 300 °C, chemically bound water begins to move away [36].

#### 4. Conclusion and Suggestions

Within the scope of this study, Nevşehir pumice (0.5-4 mm) was replaced with CEN standard sand at the rates of 10%, 20%, and 30%, respectively, and fly ash-metakaolin was replaced with cement at the rates of 10%, 20%, and 30%, respectively. Thus, a total of 13 different mixtures (CS, P10, P20, P30, FA10, FA20, FA30, MK10, MK20, MK30, ALL10, ALL20, ALL30) were prepared. The results of the experiments are summarized below.

When the results of 28-day-old samples were evaluated according to their dry unit volume mass, as the amount of pumice increased, the dry unit volume

mass decreased, while as the amount of metakaolin increased, the dry unit volume mass increased.

When the compressive strength results were evaluated, it was observed that as the ratio of pumice, fly ash, and metakaolin increased, the compressive strength increased, according to the 7, 14, and 28-day results. ALL30 sample provides the required compressive strength according to mortar strength classes and is the lightest composite mortar material.

According to flexural strength test results, P30 showed the highest flexural strength value in the beam samples, and the ALL30 sample showed the lowest value. It has been understood that pumice material increases flexural strength, while metakaolin decreases it.

It was observed that the water absorption value increased as the dry unit volume weight decreased. This is because lightweight materials are more porous.

According to the thermal conductivity coefficient results, the high thermal conductivity coefficient of the samples containing only metakaolin caused low thermal insulation, while the samples containing pumice and fly ash had a low thermal conductivity coefficient and provided high thermal insulation. Therefore, the ALL30 sample provided the best thermal insulation value.

Ultrasound transmission rate results show that the samples that completed their 28-day cure are higher than the samples that completed their 7 and 14-day cure. This shows that after 28 days of curing, the samples have largely completed their hydration, and their compressive strength has increased.

When the SEM images were examined, it was observed that there were minerals and light aggregates with a porous glassy structure, and hydrated phases such as portlandite, ettringite, and C-S-H. In the mixtures, the spherical structure of fly ash, the thick and short threadlike structure of metakaolin, and the porous structure of pumice were observed.

According to the TGA-DTA analysis results of composite mortars, the samples deteriorated in more than one stage with the use of pumice, fly ash, and metakaolin.

In light of these evaluations, it was determined that the ALL30 composite mortar mixture has high compressive strength, low dry unit volume mass, and the lowest thermal conductivity coefficient (highest thermal insulation value). Therefore, considering these features, it is thought that it can be used in the supply of mortar for the construction industry.

## Acknowledgment

This study was supported by Undergraduate Project No. 22242.

## Conflict of Interest Statement

There is no conflict of interest between the authors.

## Statement of Research and Publication Ethics

The study complies with research and publication ethics

## References

- [1] O. Uğurer, Ş. O. Kalkan, and L. Gündüz, "Poli vinilalkol (PVA) lif boyutunun çimento esaslı hafif harcın özelliklerine etkisi üzerine bir inceleme", *BAUN Fen. Bil. Enst. Dergisi*, vol. 25, no. 2, pp. 543–563, 2023, doi: 10.25092/baunfbed.1126102.
- [2] E. Çapın and N. Şapcı, "Vermikülit Agreganın Kompozit Yapılı Hafif Harç Üretiminde Kullanımı Üzerine Teknik Bir Analiz", *DÜFED*, vol. 11, no. 2, pp. 307–323, 2022, doi: 10.55007/dufed.1096993.
- [3] Y. T. Altuncı and C. Öcal, "Pamuk Küspesinin Sıva Harcı İçerisinde Agregası Olarak Kullanılabilirliğinin Araştırılması", *SDÜ Fen Bil. Enst. Der.*, vol. 25, no. 3, pp. 558–563, 2021, doi: 10.19113/sdufenbed.880877.
- [4] L. Gündüz, and Ş. O. Kalkan. "Pirinç Kabuğu Külünün Çimentolu Hafif Kompozit Harçlarda Dayanım Arttırıcı Katkı Olarak Kullanılması Üzerine Teknik Bir İnceleme", *Beton 2017 Kongresi Bildirileri, 13-14 Nisan, 2017, İstanbul, Türkiye: Türkiye Hazır Beton Birliği*, 2017. pp. 575-585
- [5] N. Şapcı, & M. Sivri, "Isı Yalıtımlı Harç Üretiminde Bazı Etken Parametrelerin SPSS Yöntemi İle İncelenmesi", *KSÜ, Müh. Bil. Dergisi*, vol. 27, no. 1, pp. 1-15, 2024, doi: 10.17780/ksujes.1269727.
- [6] F. Rahman, W. Adil, M. Raheel, M. Saberian, J. Li and T. Maqsood, "Experimental investigation of high replacement of cement by pumice in cement mortar: a mechanical, durability and microstructural study" *J. Build. Eng.* 49, 104037, 2022, doi: 10.1016/j.job.2022.104037
- [7] H. Y. Aruntaş, M. Şahinöz, ve M. Dayı, "Çimento hamur ve harçlarında kireç kullanımının incelenmesi", *Politeknik Dergisi*, vol. 24, no. 3, pp. 1045-1054, 2021, doi: 10.2339/politeknik.801346.
- [8] T. Uğur and A. Güleç, "Harç, Sıva ve Diğer Kompozit Malzemelerde Kullanılan Bağlayıcılar ve Özellikleri", *Restorasyon ve Konservasyon Çalışmaları Dergisi*, no. 17, pp. 77–91, December 2016.
- [9] L. Gulbe, I. Vitina, and J. Setina, "The influence of cement on properties of lime mortars", *Procedia Engineering*, vol. 172, pp 325-332, 2017, doi: 10.1016/j.proeng.2017.02.030
- [10] M. R. Veiga, A. Velosa, A. Magalhães, "Experimental applications of mortars with pozzolanic additions: characterization and performance evaluation", *Construction and Building Materials*, vol. 23, no. 1, pp 318-327, 2009, doi: 10.1016/j.conbuildmat.2007.12.003
- [11] R. Siddique and J. Klaus, J. "Influence of metakaolin on the properties of mortar and concrete: A review" *Appl. Clay Sci.*, vol. 43, no. 3–4, pp. 392-400, 2009, doi: 10.1016/j.clay.2008.11.007
- [12] K. Turk, C. Kina, M.L. Nehdi, "Durability of Engineered Cementitious Composites Incorporating High-Volume Fly Ash and Limestone Powder", *Sustainability* vol. 14, 10388. 2022, doi: 10.3390/su141610388
- [13] P. Turgut, F. Demir, and K. Türk, "Kendiliğinden Yerleşen Yüksek Dayanımlı Portland Çimentosuz Briket Malzemesi Üretimi", *Dokuz Eylül Üniversitesi Mühendislik Fakültesi Fen ve Mühendislik Dergisi*, vol. 24 no. 72, pp. 703-716, 2022, doi: 10.21205/deufmd.2022247202
- [14] M. Çetin, *Uçucu kül ve cam atıklarından üretilen seramiklerin özelliklerine ZnO katkısının etkisinin incelenmesi*, Yüksek lisans tezi, Sakarya Üniversitesi, 50, 2019.
- [15] A Ahmed, S. I. Bajahry, I. Garba, J. Abubakar, A. Lawan, A. Ocholi, and J. M. Kaura, "Strength Optimization of Metakaolin-Based Geopolymer Concrete Modified with Metabentonite Using Response Surface Method". *International Journal of Research Findings in Engineering, Science and Technology* vol. 5, pp. 21-36, 2023, doi: 10.48028/iiprds/ijrfest.v5.i1.03.
- [16] D. Gür, *Tarihi yapıların onarımında kullanılmak üzere puzolan katkılı hidrolik kireç esaslı onarım harcı üretilme olanaklarının araştırılması*, Yüksek lisans tezi, İstanbul Teknik Üniversitesi, 51, 2021.
- [17] TS EN 196-1, "Çimento Deney Metotları - Bölüm 1: Dayanım Tayini", Türk Standartları Enstitüsü, Ankara, Türkiye, 2016.

- [18] C. Shi, “Studies on Several Factors Affecting Hydration and Properties of Lime-Pozzolan Cements” *Journal of Materials in Civil Engineering*, vol.13 no. 6, pp. 444-445, 2001, doi:10.1061/(asce)0899-1561(2001)13:6(441)
- [19] TS EN 933-1, “Agregaların geometrik özellikleri için deneyler bölüm 1: Tane büyüklüğü dağılımı tayini- Eleme metodu”, Türk Standartları Enstitüsü, Ankara, 2012.
- [20] TS EN 1097-3, “Agregaların fiziksel ve mekanik özellikleri için deneyler bölüm 3: Gevşek yığın yoğunluğunun ve boşluk hacminin tayini”, Türk Standartları Enstitüsü, Ankara, 1999.
- [21] TS EN 1097-6, “Agregaların mekanik ve fiziksel özellikleri için deneyler - Bölüm 6: Tane yoğunluğunun ve su emme oranının tayini”, Türk Standartları Enstitüsü, Ankara, 2022.
- [22] TS EN 998-1, “Kâgir harcı - Bölüm 1: Kaba ve ince sıva harcı”, Türk Standartları Enstitüsü, Ankara, 2017.
- [23] TS EN 1015-11, “Kagir harcı- Deney yöntemleri Bölüm 11: Sertleşmiş harcın eğilmede çekme ve basınç dayanımının tayini”, Türk Standartları Enstitüsü, Ankara, 2020.
- [24] E. Güneyisi, M. Gesoğlu and K. Mermerdaş, “Improving strength, drying shrinkage, and pore structure of concrete using metakaolin”, *Mater Struct*, vol. 41, pp. 937–949, 2008, doi: 10.1617/s11527-007-9296-z
- [25] G. Görhan and G. Kürklü. "Farklı Sınıf Çimento Harcı Üretiminde Metakaolin Katkısı Kullanımının Araştırılması", *IJERAD*, vol. 7, no. 3, pp. 7–14, 2015.
- [26] J. M. Khatib, E.M. Negim and E. Gjonbalaj, “High volume metakaolin as cement replacement in mortar”, *World J. Chem*, vol. 7, no. 1, pp. 7–10, 2012.
- [27] G. Li, C. Zhou, W. Ahmad, K. I. Usanova, M. Karelina, A. M. Mohamed and R. Khallaf, "Fly Ash Application as Supplementary Cementitious Material: A Review" *Mater*, vol. 15, no. 7, pp. 2664, 2022, doi:10.3390/ma15072664
- [28] A. B. Harwalkar and S.S. Awanti, “Laboratory and field investigations on high-volume fly ash concrete for rigid pavement”, *Transp. Res. Rec*, vol. 2441, no. 1, pp. 121–127, 2014.
- [29] U. A. Khan, H. M. Jahanzaib, M. Khan, M. Ali, “Improving the Tensile Energy Absorption of High Strength Natural Fiber Reinforced Concrete with Fly-Ash for Bridge Girders” *KEM*. vol. 765, pp. 335-342, 2018, doi: 10.4028/www.scientific.net/kem.765.335
- [30] TS EN 1015-10 “Kâgir harcı-Deney metotları- Bölüm 10: Sertleşmiş harcın boşluklu kuru birim hacim kütlelerinin tayini”, Türk Standartları Enstitüsü, Ankara, 2001
- [31] H. Binici, “Atık Mukavva, Alçı, Pomza, Perlit, Vermikülit ve Zeolit ile Yapılan Kompozitlerin Yangın Direncinin Araştırılması”, *cukurovaummfd*, vol. 31, no. 1, pp. 1–10, 2016, doi: 10.21605/cukurovaummfd.317714.
- [32] ISO 22007-2 Plastics- Determination Of Thermal Conductivity and Thermal Diffusivity- Part 2: Transient Plane Heat Source (Hot Disc) Method, 2015.
- [33] TS EN 12504-4. Yapılarda beton deneyleri-Bölüm 4: Ultrasonik atımlı dalga hızının tayini. Türk Standartları Enstitüsü, Ankara, Türkiye, 2021
- [34] Ş. Ekmen, *Uçucukül esash hafif geopolymer harçların taze ve sertleşmiş özelliklerinin incelenmesi, modellenmesi ve optimizasyonu*, Doktora tezi, Harran Üniversitesi, 129, 2021.
- [35] İ. B. Topçu, and T. Uygunoğlu, “Properties of autoclaved lightweight aggregate concrete”, *Built and Environ.*, vol. 42 no. 12, pp. 4108-4116, 2007, doi: 10.1016/j.buildenv.2006.11.024
- [36] Nergis, D. D. B., Abdullah, M. M. A. B., Sandu, A. V., & Vizureanu, P. “XRD and TG-DTA study of new alkali activated materials based on fly ash with sand and glass powder,” *Mater*, vol. 13, no. 2, pp. 343, 2020 doi: 10.3390/ma13020343