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# Research Article **Properties of self-curing and self-cooling eco-friendly novel green cement-based mortar**

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**Abstract:** In this study, mullein plant (MP) (Verbascum Thapsus) was replaced with cement at a dosage of 300 (the amount of cement in mortar for producing 1 m<sup>3</sup> mortar) in cement-based mortars at different weight ratios (0%, 1.5%, 3%, 4.5% and 6%). To investigate the impact of MP on the hydration (internal) temperature and the formation time of hydration products in mortars, the mortars' interior temperature and moisture ratios were measured and recorded every minute for one day. It was concluded that the MP had a high-water absorption capacity and delayed the initial and final setting times. The optimum ratio of MP as a replacement for cement was found to be 1.5%. The study also investigated the effects of direct current (DC) application on fresh mortars (both with and without MP). The results showed that the 7-day mechanical strength of the reference mortars exhibited a significant increase with the application of DC. Previous studies have not explored the use of MP in cementitious composite materials. This study concluded that adding a specific amount of MP to cement-based materials can provide self-curing and self-cooling properties. This research is critical for water conservation as it develops a novel self-curing method.

Keywords: Self-cooling, self-curing, eco-friendly, direct current, green mortar.

# 1. Introduction

Cement is the most preferred hydraulic binder in the world. In cement-based materials, extra water is needed for the hydration reactions to continue after the material has hardened. Curing is an essential factor for the continuation of the hydration process, especially at an early age in cement-based composite materials (Chand et al., 2016). The curing process of cement-based materials is often time-consuming and economically inefficient. To address this problem, researchers have conducted extensive studies aimed at identifying more efficient curing methods. One promising solution involves preparing a buried water source for the curing process (Bentz and Weiss, 2010). After the cement-based material hardens, the first 7 days are very important for the material to gain mechanical strength. Shrinkage cracks may occur in cement-based materials, especially in concrete castings produced under hot weather conditions or high-volume concrete productions such as mass concrete, when proper precautions are not taken. Various measures are taken to eliminate this effect, such as adding mineral and chemical additives to cement-based materials. The highest mechanical strength and durability can be achieved by curing the concrete and mortar in lime water (Ravikumar et al., 2011).

Various studies have been conducted on the self-curing of cementitious materials. It has been determined in previous studies that various additives can be used to increase the hydration water content in hardened concrete (Ghiasvand et al., 2022). Self-curing cement-based composite systems can be produced using polymers, super absorbent, lightweight aggregates, wood powders, and shrinkage-reducing additives (Lokeshwari et al., 2021; ACI (308-213) R-13, 2022; Kamal et al. (2018). Ramalingam et al. (2022) observed that the water absorbed during the hydration process of concrete could be provided as an internal water source using polymer balls. Jieting et al. (2022) observed that when the superabsorbent polymer was added to the concrete, more  $C_2S$  and  $C_3S$  formed compared to the control sample. Self-curing is a novel method that can increase the internal moisture ratio in cement-based systems for a more effective cement hydration (Bentz et al., 2005; Bilek et al., 2002; El-Dieb and El-Maaddawy, 2020), which highlights the importance of using self-curing additives in cementbased systems to protect water resources (El-Dieb, 2007). Bashandy et al. (2017) concluded that self-curing concretes exhibited better mechanical properties than no cured concrete. Seongwoo et al. (2022) substituted kenaf cellulose microfibers instead of cement in cement-based composites at 0, 0.3, 0.6, 1.2 and 3% by weight. They observed that an increase in relative moisture ranging from 3% to 30%. The mullein plant (MP) grows almost all over the world (all over Europe, temperate Asia, North America, etc.) (Riaza et al., 2013). The MP is the largest genus of the Scrophulariaceae family (Tatli and Akdemir, 2016). It has been seen that the MP has been often used in the health and pharmaceutical industries (Turker and Camper, 2002; Turker and Gurel, 2005).

MP has high water absorption capacity. Therefore, this research aims to produce self-curing and self-cooling cement-based mortars using natural MP addition. This study also investigates the effects of MP on the hydration temperature. In the literature, the studies on the use of MP in cementitious materials are limited. It was investigated whether a novel self-curing method could be developed by adding MP to cement mortars. It was observed that adding MP to mortars significantly reduced the hydration temperature. However, it is also undesirable to excessively reduce the hydration temperature in cement-based materials. To eliminate this adverse effect, it was aimed to accelerate the hydration reactions by applying DC to the fresh mortars that MP added. This study is considered a significant advantage due to its cost-effectiveness compared to other self-curing methods.

# 1.1. Scope and significance

The initial days following the hardening of the cement-based materials are crucial for the material to gain mechanical strength. The curing process of cement-based materials is often time-consuming and economically inefficient. To overcome this problem, the researchers conducted extensive studies to identify more efficient curing methods. In previous studies, different methods, such as the use of polymers, lightweight aggregates, shrinkage-reducing chemical additives, and wood dust addition, have been applied to gain the self-curing properties of cementitious materials. However, these materials are often costly.

This study demonstrates both ecological and economic benefits, as it utilizes a natural plant to provide self-curing properties to cementitious materials. Furthermore, the positive impact of the results presented in this research is of critical importance in conserving limited water resources. In the literature, the studies on the use of MP in cement-based systems are limited. It was observed that when MP was added to mortars, it significantly reduced the hydration temperature. However, it is also undesirable to excessively reduce the hydration temperature in cement-based materials. To eliminate this adverse effect, it was aimed to accelerate the hydration reactions by applying DC to the fresh mortars that MP added. This study is critical for water savings due to developing a new self-curing method. However, this research should be improved as it causes significant decreases in compressive strength.

# 2. Materials and methods

# 2.1. Materials

In the experimental study, tap water, cement, crushed sand (0-4mm) and MP were used for self-cooling and self-curing green mortar production. A view of the cement, sand and MP is shown in Figure 1.



Figure 1. The view of (a) cement, (b) crushed sand and (c) mullein plant.

# 2.1.1. Cement

CEM I 42.5 R type cement, which complies with TS EN 197-1 (2011), produced by Çimsa company was used in all experiments. The components, content and physical-chemical properties of Portland cement are shown in Table 1.

	1
Components	Content (%)
CaO	63.5
Al <sub>2</sub> O <sub>3</sub>	4.72
$SiO_2$	19.6
MgO	1.91
Fe <sub>2</sub> O <sub>3</sub>	3.26
K <sub>2</sub> O	1.05
Na <sub>2</sub> O	0.34
TiO <sub>2</sub>	0.41
SO <sub>3</sub>	4.72
LG	2.67
Specific weight	3.10
Fineness, cm <sup>2</sup> /g	3307
Ignition loss	1.54
28-day compressive strength, MPa	49.3

Table 1. Content	t and components of the	cement.
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## 2.1.2. Crushed sand

The largest grain size of the crushed sand was measured as 4 mm. The specific gravity of the crushed sand was determined as 2.66. The sieve analysis results of the crushed sand are given in Table 2.

Table 2. Sieve analysis of the crushed sand (Hocaoğlu, 2022).			
	Passing siev	e (%)	
Sieve no	According to ASTM C778-13	Experimental results	
16 (1.18 mm)	100	100	
30 (600 µm)	96-100	99	
40 (425 µm)	65-75	69	
50 (300 µm)	20-30	28	
100 (150 µm)	0-4	4	

## 2.1.3. Mullein plant

The MP, in its natural state, exhibits hydrophobic property. In previous studies, total ash, acid-insoluble, and water-soluble ash in the mullein leaves were found as 14.32%, 4.75% and 8.30%, respectively (Mahek et al., 2011). On the other hand, the granulated MP has high water absorption capacity. The MP used in the experiments was taken directly from nature. First, the MP was dried in an oven at almost 105 °C for one day. Afterward, the MP was granulated using a grinder under laboratory conditions. The average particle size of ground MP was measured as 1-2 mm. The water absorption rate of the MP was carried out in the laboratory and determined as between 20-22%. The water absorption test of MP was conducted using the ASTM D570 (2022). Figure 2 shows the natural appearance of the MP, as well as its appearance before and after grinding and views taken from SEM.

In the previous studies, chemical analysis of MP was conducted, and according to the results of chemical analysis, it was observed that there was Al, Na, K, Mg, Sulfate, chloride, silicate, Cr, Ni, Zn and Pb elements (Riaz et al., 2013; Kifayatullah et al., 2001; Shah et al., 2004).



Figure 2. The appearance of MP (a) natural state, (b) drying, (c) grinding, (d) microstructure.

## 2.2. Method

## 2.2.1 Preparation of the mortars

The green mortars were prepared at dosages of 300 and in accordance with ASTM C305-20 (2020). In all series, the w/c ratio was designed as 1.00. There are two reasons for using a high water-to-cement ratio (w/c) of 1.00 in mortar mixes. The first reason is that the MP has high water absorption capacity. The second reason is that the application of electric current during mixing can increase the hydration temperature of the mortar, causing some of the hydration water to evaporate. Mortars were prepared by adding 0, 1.5, 3, 4.5 and 6% MP by weight instead of cement. While calculating the mixture, the water-saturated and surface dry weight of the crushed sand were used. While mixing the mortar, a dry mixture (cement and crushed sand) was made first. Then water was added in three stages. Finally, MP was added and mixed. The mixing ratio of one cubic meter mortar is shown in Table 3. The mixture process followed in this study is shown in Figure 3.

#### Table 3. Mixing ratio of the mortars.

MP (%)	Cement (kg/m <sup>3</sup> )	Sand (kg/m <sup>3</sup> )	MP (kg/m <sup>3</sup> )	Water (lt/m <sup>3</sup> )
0	300.00	1551.20	-	300.00
1.50	295.50	1551.20	4.50	295.50
3.00	291.00	1551.20	9.00	291.00
4.50	286.50	1551.20	13.50	286.50
6.00	282.00	1551.20	18.00	282.00



Figure 3. The mixing process of the study.

2.2.2. DC application on mortars, measuring the hydration temperature and moisture ratios

When mortars (with and without MP) were fresh, stress intensity of 27.5V was applied through a DC power supply for almost one day. For comparison, reference mortars were produced for each mixing ratio. The internal temperature values of the specimens were measured (by placing a temperature sensor in the center of the mortars) for every minute. The data was recorded in the data logger. The moisture (surface) content was also measured at every minute. The application of DC to the mortars and the measurement of hydration temperatures-moisture ratios are shown in Figure 4.



Figure 4. DC application, measuring and recording internal temperature and moisture values.

2.2.3. Curing of the samples, physical and mechanical experiments

The mortars were removed from the molds after one day. Then they were put in saturated lime water for 7 and 28-day according to the BS EN 12390-2 (2019). Afterward, the samples were kept at room condition until the test day (Figure 5).



Figure 5. Laboratory conditions in which the experiments were carried out.

After the mortars were taken from the curing pool, the saturated surface dry weights, oven dry weights (after the mortars were exposed to 110 °C for one day), and their weights in water were measured. The water absorptions of the mortars were calculated using Equation 1 and Equation 2 according to TS EN 12390 (2010).

$$Porosity = \frac{(W2 - W0)}{(W2 - W1)} X100$$
(1)

$$Water \ absorption = \frac{(W2 - W0)}{(W0)} X100 \tag{2}$$

In the equations,  $W_2$  is the weight in saturated air (g),  $W_1$  is the weight in water (g) and  $W_0$  is the oven dry weight (g). Afterward, the samples were subjected to 3-point tensile and compressive strength tests in accordance with ASTM C348 (2014).

#### 2.2.4. Durability experiments on mortars

The sulfate resistance experiment was carried out on 40x40x160 mm mortar specimens, where the electric current was not applied, according to ASTM C1012 (2004). The mortars underwent a 28-day curing process in lime water, followed by immersion in a 10% Na<sub>2</sub>SO<sub>4</sub> solution for 90 days. The Na<sub>2</sub>SO<sub>4</sub> solution was refreshed every 15 days to maintain a consistent concentration level. After 180 days, tensile and compressive strength tests were conducted on the mortars subjected to sulfate attack.

#### 2.2.5. Micro examination

EDX and SEM analyses were performed on some selected mortars. Firstly, 5-7 mm pieces were taken from the samples subjected to mechanical experiments. Then the surfaces of these samples were coated with carbon. Afterward, the mortars were put through SEM process. The microanalysis was performed by zooming 10000 times.

## 3. Results and discussion

#### 3.1. The effect of mullein plant on hydration temperature

Figure 6 compares the hydration temperatures of cementitious mortars with different proportions of MP (0, 1.5, 3, 4.5 and 6%). The results show that the internal temperature decreased as the amount of MP substituted with cement (Figure 6). Decreasing hydration temperature in cement-based materials means that the hydration reaction rate slows. It was seen that the hydration temperature decreased significantly in the mortars, when MP was added at high rates (4.5 and 6%). This decrease is attributed to the high-water absorption capacity of MP, which slows down the hydration reaction rate. It was thought that the most efficient results could be obtained when 1.5% MP by weight was added instead of cement since it had the closest internal temperature values compared to the control mortar. When 1.5% and 6% MP by weight were added instead of cement, approximately 0.50 °C and 2.75 °C decrease was observed in the internal temperatures of the mortars compared to the initial hydration (Figure 6).



Figure 6. Comparison of hydration temperatures of mortars with different MP ratios.

It is also undesirable for hydration reactions to excessively slow down in cement-based composite materials during the early stages of hydration. To eliminate the adverse impact of MP, it is aimed to increase the hydration temperatures of fresh MP-added mortars by applying 27.5V DC for approximately one day. It was observed that the hydration temperatures of the mortars with MP additives at different rates (0, 1.5, 3, 4.5 and 6%) and with DC application approximately increased by 4.27 °C, 4.00 °C, 2.64 °C, 1.84 °C and 1.48 °C. Previous studies have indicated that the final setting time for cementitious materials occurs when the internal temperature approaches the highest (first peak) values (Kosmatka et al., 2016; Hocaoğlu, 2021). The final setting times of the DC-applied mortars were approximately determined at 330, 211, 209, 99 and 94 minutes, respectively when 0, 1.5, 3, 4.5 and 6% MP were added (Figure 7).



Figure 7. Comparison of internal temperature values and setting (final) time in mortars.

The shortening of the final setting time can be explained in two ways as the MP ratio increases in DC-applied mortars. The first explanation is that the increase in the ratio of MP in the mortar causes more hydration water absorption. The second reason is the evaporation of some of the hydration water due to the increase in the internal temperature of the mortars with the DC application. Since there was less hydration water in the mortars containing a high MP ratio, it can be interpreted that the hardening occurs in a shorter time (Fabre et al., 2011).

#### 3.2. Effect of MP on mortar surface moisture during hydration

Figure 8 was prepared to compare the surface moisture ratios of mortars with different MP (0, 1.5, 3, 4.5 and 6%) additives from the initial stage of hydration. It was observed that the mortars with 1.5% and 3% MP in the first stages of hydration had lower moisture rates than the mortars with MP at other rates. However, it was seen that the moisture ratio increased in the following stages of hydration. In particular, significant changes in the moisture ratio of the mortars containing 4.5% and 6% MP were not observed.



Figure 8. Mechanism of formation of CSH gels by moisture measurement in mortars.

This situation can be explained as follows: due to the high-water absorption capacity of the MP, the hydration water was absorbed at a high rate during the initial stage of hydration in the mortars with 4.5% and 6% MP. It was thought that the

mortars with 1.5% and 3% MP absorbed less water at the beginning of hydration than mortars with other MP contents. It has been interpreted that after the setting (final) time, the water trapped in the MP leaves some of it, causing an increase in moisture. When Figure 8 was examined, it was concluded that the moisture ratios decreased and rose rapidly in the following minutes of hydration. These peaks mean that CSH gels formed (Hocaoğlu, 2022).

## 3.3. Comparison of the initial and final setting times

Previous studies have determined that initial and final setting times in cementitious materials can be predicted by measuring surface moisture (Hocaoğlu, 2022; Hocaoğlu, 2023). In cement-based materials, the initial setting time occurs when the surface moisture content reaches its first lowest value (Hocaoğlu, 2023). The final setting time occurs when the surface moisture content reaches the second lowest value (Hocaoğlu, 2022; Hocaoğlu, 2024). Figure 9 illustrates the estimation of the initial setting times of the mortars which MP was added to at different rates. When Figure 9 was examined, it was observed that the initial setting times of the mortars were realized at approximately 29, 35, 43, 45 and 62 minutes by adding MP with the ratios of 0, 1.5, 3, 4.5 and 6%.



Figure 9. Effect of the MP on initial setting time.

Figure 10 was drawn to estimate the final setting times of the mortars which MP was added to at different ratios. The final setting time of the mortars containing 0, 1.5, 3, 4.5 and 6% MP was determined as 262, 325, 337, 341 and 423 mins, respectively.



Figure 10. Effect of the MP on final setting time.

As the amount of MP in the mortars increased, the setting (initial and final) times were delayed. It is critical to slow down the hydration reaction in structures in hot weather conditions. In this study, it was concluded that the MP could be used as a set retarder.

#### 3.4. Gaining self-curing property by adding MP to cement-based mortars

Figure 11 compares the moisture content of 1, 7 and 28-day cured mortars with different MP ratios. The 1-day data in the Figures represent the fresh moisture content of the mortars (for approximately one day from the beginning of the hydration). When Figure 11 was examined, it was observed that, in general, moisture rates of the mortars cured for 28-day approach the moisture rates of the 1-day samples. However, it was observed that it became much closer with increasing MP in the mortar content. If the moisture ratio approaches the moisture level of the first day, it indicates that there is free water inside the mortar and on its surface. In general, it was observed that the moisture content of the mortars cured for 7 and 28-day increased between 0-500 minutes. It was thought that this situation emerged due to measuring the sample surface moisture (Figure 11 a-e). The free water in the mortar came to the surface of the mortar and caused an increase in the moisture rate. It was also observed that the low humidity at 7 days compared to 28 days for all mortars with different proportions of MP. It can be explained that rapid hydration in the first 7 days may reduce the moisture content.





Figure 11. Self-curing of mortars with mullein plant additives in different ratios (a-0%, b-1.5%, c-3%, d-4.5%, e-6%).

While the moisture content of the samples cured for 7 and 28-day after the 500<sup>th</sup> minute in the control mortar was almost the same, differences were observed in the mortars containing the MP. This result demonstrates that mortar with a self-curing can be produced. Previous studies have applied various methods to achieve self-curing properties in cementitious materials, such as superabsorbent polymers, lightweight aggregates, shrinkage-reducing chemical additives and wood dust addition have been applied (Lokeshwari et al., 2021; ACI (308-213) R-13, 2022; Kamal et al., 2018). However, these materials are usually high cost. In this study, a natural plant, which is economical and environmentally friendly, was used to provide self-curing properties to cementitious materials.

### 3.5. Flexural strength of the mortars

Figure 12 compares the flexural strength of mortars with different MP ratios. Figure 12a shows the 7-day (cured) flexural strength of the mortars, and Figure 12b represents the 28-day (cured) flexural strength. When the 7-day flexural strengths of the mortars were compared, it was seen that the flexural strengths of the DC-applied mortars were higher than those of the non-DC-applied samples. Conversely, the opposite result was obtained in samples cured for 28-days. When electrical current was applied on 7-day cured mortars with MP (0, 1.5, 3 and 4.5%) additives, the flexural strengths of the mortars increased by 14.26%, 7.24%, 5.73% and 2.98%, respectively compared to reference (DC was not applied) mortars. When the stress intensity of 27.5V was applied to 28-days cured mortars with 0, 1.5, 3, 4.5 and 6% MP additives, the flexural strengths of the mortars decreased by 10.23%, 5.50%, 8.26%, 5.16% and 12.67% respectively, compared to reference mortars.



Figure 12. Flexural strengths of the mortars with different MP (a- 7-day cured, b- 28-day cured).

## 3.6. Compressive strength of the mortars

Figure 13 compares the compressive strength of mortars with different MP ratios and cured for 7-day. It was seen that as the MP ratio in the mortar increased, the compressive strength decreased. When 1.5, 3, 4.5 and 6% MP were added to the 7-day cured mortars, the compressive strengths of the mortars were 12.98%, 22.89%, 54.15% and 64.67% lower, respectively than the control mortar (without MP). It was concluded that the compressive strengths of the mortars which were 1-day electrically cured and then cured in lime water for the remaining 6-day were higher than those that not electrically cured (Figure 13).



Figure 13. Compressive strengths of different ratios of MP-added and 7-day cured mortars.

When 1.5, 3, 4.5 and 6% MP were added to the 28-day cured mortars, the compressive strengths of the mortars were 6.24%, 26.19%, 44.88% and 62.33% lower, respectively than the reference (0% MP added and DC was not applied) mortar. The excessive absorption of MP to the hydration water can explain the low compressive strength values of mortars containing high MP. This confirms the results obtained in the porosity tests. In previous research, self-curing concrete has shown 10% less compressive strength than concrete cured in normal water (Vyawahare et al., 2014; Bashandy, 2015). It was observed

that the optimum ratio of MP to be placed in cement-based materials was 1.5%. It was also concluded that the compressive strengths of the electrical cured mortars which were lower than those that not electrically cured (Figure 14).



Figure 14. Compressive strengths of different ratios of MP-added and 28-day cured mortars.

### 3.7. The physical and durability experiment results

Figure 15 compares the porosity and water absorption rates of mortars containing different ratios of MP that were cured for 28-day. It was observed that increasing the MP content in the mortar led to a rise in porosity. For instance, adding 4.5% MP resulted in a 37.67% increase in porosity compared to the control mortar. A higher void ratio in cement-based composite materials generally indicates reduced mechanical strength and durability. In this study, the results obtained in physical experiments are compatible with those obtained in mechanical experiments. Additionally, the water absorption rate also increased with a higher MP content in the mortars (Figure 15). The increase in the water absorption rate could be explained by the high water absorption capacity of the MP.



Figure 15. Porosities and water absorptions of mortars depending on MP.

Figure 16 compares the durability of mortars with varying MP ratios. With the effect of sulfate attacks, cracks occur in the internal structure of the products formed in cementitious systems and cause an increase in porosity. Previous research has concluded that a 1% entrapped air gap can reduce compressive strength by approximately 5-7% (The Concrete Society, 1976).



Figure 16. Durability experiment results of mortars depending on MP.

When Figure 16 was examined, decreases were observed in flexural and compressive strength as the MP ratio in the mortar increased. In terms of durability, the optimum MP ratio for cement-based mortars was found to be 1.5%. The results showing the loss in flexural and compressive strength of mortar samples exposed to Na<sub>2</sub>SO<sub>4</sub> solution are presented in Table 4. Continuous loss of flexural and compressive strength is known to occur during immersion in sulfate solutions. However, the lowest decrease in strengths was observed in the control sample due to the sulfate attack. This reduced durability in mortars with higher MP content can be attributed to increased porosity. Increasing the porosity in mortars allows harmful ions to diffuse into the mortar easier.

Table 4. Flexural and compressive strength reduction rate depending on durability experiment.

Strongth reduction rotes 0/			MP, %		
Strength reduction rates, %	0	1.5	3	4.5	6
Flexural strength	36.65	39.20	39.39	39.80	40.28
Compressive strength	32.99	36.41	38.14	38.28	39.32

## 3.8. Micro examination

#### 3.8.1. SEM analysis results

When the mixing water becomes supersaturated with dissolved calcium ions, new hydration products begin to form, resulting in an increase in heat output. This situation is called the onset of hardening. C-H gels and C-S-H gels are crucial for the cement-based material to gain strength (Jaya et al., 2018). C-S-H gels reduce the free Ca(OH)<sub>2</sub> and water content in the cementitious systems (Salem, 2002). In the following stages of hydration, the C-S-H products are formed more tightly (Figure 17).



Figure 17. Variation of hydration reactions over time (Kosmatka and Voigt, 2016).

Figure 18a represents the mortar (28-day cured) without MP addition. When Figure 18a was examined, it was observed that CSH gels and needle-shaped ettringites formed. Figure 18b represents the reference mortar exposed to DC for 1-day and then cured in lime water for 27-day. A significant deterioration in the microstructure of the mortar was observed when an electric current was applied for one day, followed by curing in lime water for 27 days. Additionally, pore structure formation was noted in the DC-applied sample. The formation of pores in the microstructure of the mortar caused a decrease in compressive strength. SEM analysis of the mortar containing 1.5% MP is shown in Figure 18c. As a result of replacing the cement with MP at a ratio of 1.5%, it was seen that the C-S-H gels formed less than the reference (without MP) mortar (Figure 18c). Figure 18d shows the mortar with 3% MP substituted with cement exposed to DC for 1-day and then cured in lime water for 27-daya. It was seen that fewer C-S-H gels formed in the electrically cured (during one day) mortars (Figs. 18d-f). The microstructure deterioration occurred with the increase in the amount of MP in the mortar (Figure 18e). This is because the MP does not show pozzolanic reaction. The increase in MP ratio led to higher porosity, which, in turn, decreased the durability and compressive strength of the mortars.





Figure 18. Microstructure views of the mortars (a) 0V-0% MP, (b) 27.5V- 0% MP, (c) 0V- 1.5% MP, (d) 27.5V- 1.5% MP, (e) 0V- 4.5% MP, (f) 27.5V- 4.5% MP.

#### 3.8.2. EDS (Energy dispersive spectroscopy) analysis results

EDS can provide information about which elements are formed in cementitious systems (Kumar et al., 2020). EDS analysis results of the mortars depending on MP addition and DC application, which cured for 28-days, are shown in Figure 19. Silicon (Si) and Calcium (Ca) are effective in the formation of CH and C-S-H gels (Hocaoğlu, 2022; Hu, 2014). Figure 19a represents the mortar cured for 28 days without MP addition. Figure 19b represents the reference mortar exposed to DC for 1-day and then cured in lime water for 27-days. Figure 19c represents the 28-days cured mortar with 1.5% MP. Figure 19d shows the mortar with 1.5% MP, with DC applied for 1-day, and cured in lime water for 27-days. Figure 19f shows the mortar with 4.5% MP, with DC applied for 1-day, and cured in lime water for 27-day, and cured in lime water for 27-days. It was seen that Ca, the most influential element in forming C-S-H gels (Hocaoğlu, 2022), formed more without DC application (Figure 19). It was also concluded that the Ca formation decreased with the increase of the MP ratio in the mortar.





Figure 19. EDS analysis results of mortars depending on MP and DC (a) 0V-0% MP, (b) 27.5V-0% MP, (c) 0V-1.5% MP, (d) 27.5V-1.5% MP, (e) 0V-4.5% MP, (f) 27.5V-4.5% MP.

It has been seen in the literature that some studies have been conducted to determine the CaO/SiO<sub>2</sub> ratios in cementitious materials and to estimate the formation rates of CSH gels (Hu, 2014). Kunther et al. (2017) concluded that more hydration products formed when cement-based materials had a low CaO/SiO<sub>2</sub> ratio (Kunther et al., 2017). More hydration products mean higher mechanical strength. Table 5 compares the CaO/SiO<sub>2</sub> ratios of the mortars. CaO and SiO<sub>2</sub> in the sample were measured from the parts where the most compact (CSH gels occur) formed. It was concluded that the Ca/SiO<sub>2</sub> ratio increased with increasing MP ratio in mortar. It was also seen that the Ca/SiO<sub>2</sub> ratio increased with the DC application.

MP% - DC	CO <sub>2</sub> , kg	fc, MPa	Cf (CO <sub>2</sub> /fc)
0% MP - 0V	20.73	23.55	0.88
0% MP – 27.5V	34.74	22.14	1.56
1.5% MP – 0V	45.45	22.08	2.05
1.5% MP – 27.5V	36.14	19.48	1.85
4.5% MP – 0V	59.31	12.98	4.56
4.5% MP – 27.5V	38.78	11.98	3.23

Table 5. Comparison of CaO/SiO <sub>2</sub> rat	tios in 28-day cured mortars
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#### 3.9. Sustainability analysis

In particular, the negative environmental impact of cement production (Hossain et al., 2017) and high anthropogenic greenhouse gas (GHG) emissions cause worldwide concern. An environmentally friendly system can create embedded carbon (eCO<sub>2</sub>) in cementitious composite materials (Purnell and Black, 2012). The reactions shown in Equation 3 and Equation 4 occur during the process of cement hydration.

$$CaO + H_2O \rightarrow Ca(OH)_2 + heat$$
 (3)

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \tag{4}$$

Carbonation of portlandite  $(Ca(OH)_2)$  is critical for cementitious materials in terms of CO<sub>2</sub> storage (Kutchko et al., 2007; Scherer and Huet, 2009; Duguid and Scherer, 2010). Many scientists have associated eCO<sub>2</sub> values for concrete with compressive strength (Xiao et al., 2020). Hammond and Jones (2008) concluded that there is a monotonic relationship between characteristic compressive strength (8-50 MPa) and eCO<sub>2</sub> (0.061-0.188) for OPC. The CO<sub>2</sub> amounts obtained from the EDS analysis of different MP-added mortars are shown in Table 6. Equation 5 was used to measure the sustainability (Cf) (Topçu and Sofuoğlu, 2021) of the MP-added mortars.

$$Cf = \frac{emboided \ CO_2}{fc} \tag{5}$$

Where, fc is the 28-day compressive strength (MPa) of mortar, and Cf ( $CO_2/kg$ . MPa) is the embodied  $CO_2$  parameter (Topçu and Sofuoğlu, 2021). The Cf values of MP-added mortars were observed to have lower values than the control mortar (without MP). Previous studies found that when Cf had lower values, it had better sustainability (Xiao et al., 2020). It was concluded that OPC can be a more sustainable than MP (Table 6). In addition, it was concluded that sustainability also decreased as the amount of MP in the mortar increased. The study also found that electric current generally had a negative impact on sustainability.

Table 6. Sustainability analysis of mortars	depending on MP ad	dition and DC application.
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MP% - DC	$CO_2$	CaO	SiO <sub>2</sub>	CaO/SiO <sub>2</sub>
0% MP – 0V	20.73	58.64	13.23	4.43
0% MP – 27.5V	34.74	38.72	7.94	4.87
1.5% MP-0V	45.45	51.44	10.92	4.71
1.5% MP – 27.5V	36.14	48.10	9.79	4.91
4.5% MP $- 0V$	59.31	32.42	7.05	4.59
4.5% MP – 27.5V	38.78	53.28	7.84	6.79

## 4. Conclusions and comments

This study demonstrates both ecological and economic benefits, as it utilizes a natural plant to provide self-curing properties to cementitious materials. Furthermore, the positive impact of the results presented in this research is of critical importance in conserving limited water resources. The results obtained as a result of the study are summarized below:

- 1. When 1.5% and 6% MP by weight were added instead of cement, approximately 0.50 °C and 2.75 °C decrease was observed in the hydration temperature values of the mortars compared to the initial stage of hydration. It was concluded that the MP could be used as a setting retarder.
- It was concluded that the internal temperature values of the mortars with MP additives at different ratios (0%, 1.5%, 3%, 4.5%, and 6%) with DC application approximately increased by 4.27 °C, 4.00 °C, 2.64 °C, 1.84 °C and, 1.48 °C.
- 3. The final setting times of the DC-applied mortars were approximately determined at 330, 211, 209, 99 and 94 minutes, respectively when 0%, 1.5%, 3%, 4.5%, and 6% MP were added.
- 4. When the 7-day flexural strengths of the mortars were compared, it was observed that the flexural strengths of the DC-applied samples were higher than those of the non-DC-applied samples. When DC was applied on 7-day cured mortars with MP (0%, 1.5%, 3%, and 4.5%) additive, the flexural strengths of the mortars increased by 14.26%, 7.24%, 5.73%, and 2.98%, respectively compared to the reference mortars.
- 5. When 1.5%, 3%, 4.5%, and 6% MP were added to the 7-day cured mortars, the compressive strengths of the mortars were 12.98%, 22.89%, 54.15%, and 64.67% lower, respectively than the reference mortar.

- 6. When 1.5%, 3%, 4.5%, and 6% MP were added to the 28-day cured mortars, the compressive strengths of the mortars were 6.24%, 26.19%, 44.88%, and 62.33% lower, respectively than the reference mortar. As a result, it was determined that the optimum MP ratio placed in cement-based materials was 1.5.
- 7. It was observed that the increase in the amount of MP in the mortar caused a rise in its porosity. It was concluded that the porosities of the mortars increased by approximately 37.67 % with the addition of 4.5% MP instead of cement.
- 8. It has been concluded that in terms of durability, the optimum MP ratio was 1.5% in cement-based mortars.
- 9. A significant deterioration in the microstructure of the mortar was observed by applying an electric current to the mortar for 1-day and then curing it in lime water for 27-day.
- 10. MP may be a more sustainable alternative than OPC.
- 11. In this study, water savings can be achieved due to the development of a new self-curing method.

This study could be further improved by exploring the effects of incorporating MP into concrete at different ratios, as well as investigating its impact on lower w/c ratios. Additionally, improved mechanical strength may be achieved with better grinding of MP and increasing its surface area. It has been observed that the mechanical strength is low in MP-added mortars. It is thought that higher mechanical strength can be achieved when MP is used with nanomaterials. It was also thought that the study could be improved by producing MP-added concrete by BS EN 196-3 (2016) standard. This study is considered critical for future research on cementitious composite systems, aiming to shorten the traditional curing period.

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## References

ACI (308-213) R-13 (2022). Report on Internally Cured Concrete using Pre-Wetted Absorptive Lightweight Aggregate. American Concrete Institute.

- ASTM C 1012-04, Standard test method for length change of hydraulic-cement mortars exposed to a sulfate solution, ASTM International, 2004. www.astm.org.
- ASTM C305-20, Standard practice for mechanical mixing of hydraulic cement pastes and mortars of plastic consistency, ASTM International, West Conshohocken, PA, 2020. www.astm.org.

ASTM C348. Standard Test Method for Flexural Strength of Hydraulic-Cement. ASTM Int West Conshohocken, PA. 2014. www.astm.org.

ASTM D570-98, Standard Test Method for Water Absorption, ASTM International, West Conshohocken, PA, 2022. www.astm.org.

Bashandy, A.A. (2015). Performance of self-curing concrete at elevated temperatures, Indian Journal of Engineering and Materials Science, 22, 93-104. http://nopr.niscpr.res.in/handle/123456789/31251.

Bashandy, A.A., Meleka, N.N., Hamad, M.M. (2017). Comparative study on the using of PEG and PAM as curing agents for self-curing concrete. Challenge Journal of Concrete Research Letters, 8(1), 1–10. https://doi.org/10.20528/cjcrl.2017.01.001.

Bentz, D.P., Lura, P. Roberts, J.W. (2005). Mixture proportioning for internal curing. Reprinted from the Concrete International, 27(2), 35-40.

- Bentz, D.P., Weiss, W.J. (2011). Internal curing: A 2010 state-of-the-art review, U.S. Department of Commerce, NISTIR 7765. https://doi.org/10.6028/NIST.IR.7765.
- Bilek, V., Kersner, Z., Schmid, P., Mosler, T. (2002). The possibility of self-curing concrete. Proceedings of the International Conference of Innovations and Developments in Concrete Materials and Construction, 51-60.

BS EN 12390-2, Testing hardened concrete making and curing specimens for strength tests, 2019.

BS EN 196-3, Methods of testing cement, 2016.

Chand, M.S.R., Giri, P.S.N.R., Kumar, P.R., Kumar, G.R., Raveena, C. (2016). Effect of self-curing chemicals in self-compacting mortars. Construction and Building Materials, 107, 356-364. https://doi.org/10.1016/j.conbuildmat.2016.01.018.

Concrete Core Testing for Strength, Technical Report No:11, The Concrete Society, London, 1976.

- Duguid, A., Scherer, G.W. (2010). Degradation of oil well cement due to exposure to carbonated brine. International Journal of Greenhouse Gas Control, 4, 546-560. https://doi.org/10.1016/j.ijggc.2009.11.001.
- El-Dieb, A. S., & El-Maaddawy, T. A. (2020). Performance of self-curing concrete as affected by different curing regimes. Advances in Concrete Construction, 9(1), 33-41. https://doi.org/10.12989/acc.2020.9.1.033.
- El-Dieb, A.S. (2007). Self-curing concrete: Water retention, hydration and moisture transport. Construction and Building Materials, 21, 1282-1287. https://doi.org/10.1016/j.conbuildmat.2006.02.007.
- Fabre, S., Lesaignoux, A., Olioso, A., Briotte, X. (2011). Influence of water content on spectral reflectance of leaves in the in the 3–15-µm Domain. IEEE Geoscience and Remote Sensing Letters, 8(1), 143-147. https://doi.org/10.1109/LGRS.2010.2053518.
- Ghiasvand, H., Bastami, M., Farokhzad, R. (2022). Enhancing the internal curing process of self-compacting concrete containing lightweight aggregate and chemical additives. European Journal of Environmental and Civil Engineering, 1-19. https://doi.org/10.1080/19648189.2022.2026824.
- Hammond, G.P., Jones, C.I. (2008). Embodied energy and carbon in construction materials. Proceedings of the Institution of Civil Engineers Energy, 161(2), 87–98. https://doi.org/10.1680/ener.2008.161.2.87.
- Hocaoglu, I. (2021). Self-heating mortars with using graphene oxide and increasing CSH gel formation with the direct current application. Revista De La Construcción. Journal of Construction, 20(3), 559–575. https://doi.org/10.7764/RDLC.20.3.559.
- Hocaoğlu, I. (2022). Investigation of the effect of current in zeolite-graphene oxide additives of mortar and development of a novel method for determining the setting time. Journal of Building Engineering, 46, 103803. https://doi.org/10.1016/j.jobe.2021.103803.
- Hocaoğlu, I. (2022). Self-cooling mortar production with zinc oxide nanoparticles additive and investigation of the DC application when early-age hydration. European Journal of Environmental and Civil Engineering. https://doi.org/10.1080/19648189.2022.2144464.
- Hocaoğlu, I. (2023). Investigation of the effects of dosage and direct current intensity in new generation mortars with graphene oxide additives. Journal of the Faculty of Engineering and Architecture of Gazi University, 38(1), 421-434. https://doi.org/10.17341/gazimmfd.940271.
- Hocaoğlu, I (2024). A novel cost-effective method for self-estimation of crack occur time by piezoelectric measurement of glass powder-modified cementitious mortar. Journal of Building Engineering, 86, 108967. https://doi.org/10.1016/j.jobe.2024.108967.
- Hossain, M.U., Poon, C.S., Lob, I.M.C., Cheng, J.C.P. (2017). Comparative LCA on using waste materials in the cement industry: a Hong Kong case study, Resources Conservation and Recycling, 120, 199-208. https://doi.org/10.1016/j.resconrec.2016.12.012.
- Hu, C. (2014). Microstructure and mechanical properties of fly ash blended cement pastes. Construction and Building Materials, 73, 618-625. https://doi.org/10.1016/j.conbuildmat.2014.10.009.
- Jaya, R.P., Hainin, M.R., Wan, I.M.H., Nazri, F.M., Arshad, M.F., Muthusamy, K. (2018). Physical and chemical properties of rice husk ash concrete under seawater, International Journal of Integrated Engineering, 10, 165-168. http://umpir.ump.edu.my/id/eprint/22519.
- Jieting, X., Xiao, Q., Zhenying, H., Yongkang, L., Ben, L., Zhengzhuan, X. (2022). Effect of superabsorbent polymer (SAP) internal curing agent on carbonation resistance and hydration performance of cement concrete. Advances in Materials Science and Engineering, 3485373, 1-13. https://doi.org/10.1155/2022/3485373.
- Kamal, M. M., Safan, M.A., Bashandy, A.A., Khalil, A.M. (2018). Experimental investigation on the behavior of normal strength and high strength selfcuring self-compacting concrete. Journal of Building Engineering, 16, 79-93, https://doi.org/10.1016/j.jobe.2017.12.012.
- Kifayatullah, Q., Shah, M.T., Arfan, M. (2001). Biogeochemical and environmental study of the chromite-rich ultramafic terrain of Malakand area Pakistan. Environmental Geology, 40, 1482-1487. https://doi.org/10.1007/s002540100374.
- Kosmatka, S.H., Voigt, G., Taylor, P. (2016). Integrated materials and construction practices for concrete pavement: a state-of-the-practice manual. Center for Translation Research and Education, Iowa State University, 69-104. https://intrans.iastate.edu/app/uploads/2019/05/IMCP\_manual.pdf.
- Kumar, M., Bansal, M., Garg, R. (2020). An overview of beneficiary aspects of zinc oxide nanoparticles on performance of cement composites. Materials today: Proceedings, 43, 892-898. https://doi.org/10.1016/j.matpr.2020.07.215.
- Kunther, W., Ferreiro, S., Skibsted, J. (2017). Influence of the Ca/Si ratio on the compressive strength of cementitious calcium-silicate-hydrate binders, Journal of Materials Chemistry A. 5(33), 401-412. https://doi.org/10.1039/C7TA06104H.
- Kutchko, B.G., Strazisar, B.R., Dzombak, D.A., Lowry, G.V., Thaulow, N. (2007). Degradation of Well Cement by CO<sub>2</sub> under Geologic Sequestration Conditions, Environmental Science and Technology, 41(13), 4787. https://doi.org/10.1021/es062828c.
- Lokeshwari, M., Pavan, B.R., Bandakli, S.R., Tarun, P., Sachin, Kumar, V. (2021). A review on self-curing concrete. Materials today: Proceedings, 43(2), 2259-2264. https://doi.org/10.1016/j.matpr.2020.12.859
- Mahek, A., Tanveer, N., Jayalakshmi, S. (2011). Pharmacognostical standardisation and physico-chemical valuations of leaves of Verbascum Thapsus Linn. International Journal of Drug Development and Research, 3(1), 334-340.

- Purnell, P., Black, L. (2012). Embodied carbon dioxide in concrete: variation with common mix design parameters. Cement and Concrete Research 42, 874-877. https://doi.org/10.1016/j.cemconres.2012.02.005.
- Ramalingam, V., Ramesh, K., Duraipandi, M.U.J., Kuppusamy, S. (2022). Water absorbing polymer balls as internal water curing agent in concrete to support hydration reaction. Revista De La Construcción. Journal of Construction, 21(1), 83–92. https://doi.org/10.7764/RDLC.21.1.83
- Ravikumar, M.S., Selvamony, C., Kannan, S.U., Basil, G.S. (2011). Self-compacted self-curing kiln ash concrete. International Journal on Design and Manufacturing Technologies. 5(1), 63.
- Riaza, M., Zia-Ul-Haq, M., Jaafar, H.Z.E. (2013). Common mullein, pharmacological and chemical aspects, Revista Brasileira Farmacogn, 23, 948-959, https://doi.org/10.1590/S0102-695X2013000600012.
- Salem, T.M. (2002). Electrical conductivity and rheological properties of ordinary portland cement-silica fume and calcium hydroxide-silica fume pastes. Cement and Concrete Research, 32, 1473-1481. https://doi.org/10.1016/S0008-8846(02)00809-8.
- Scherer, G.W., Huet, B. (2009). Carbonation of wellbore cement by CO<sub>2</sub> diffusion from caprock. International Journal of Greenhouse Gas Control, 3(40), 731-735. https://doi.org/10.1016/j.ijggc.2009.08.002.
- Seongwoo, G., Young, C.C., Myoungsu, S. (2022). Internal curing of cement composites using kenaf cellulose microfibers. Journal of Building Engineering 47, 103867. https://doi.org/10.1016/j.jobe.2021.103867.
- Shah, M.T., Kifayattullah, Q., Arfan, M. (2004). Pedo and biogeochemical study of zinc-lead deposits of the Besham area, northern Pakistan: its implication in mineral exploration and environmental degradation. Environmental Geology, 45, 544-549. https://doi.org/10.1007/s00254-003-0909-8.
- Tatli, I.I. Akdemir, Z.S. (2016). Traditional uses and biological activities of verbascum species. Fabad Journal of Pharmaceutical Sciences, 31, 85-96.
- Topçu, I.B., Sofuoğlu, T. (2021). Properties of geopolymers produced with sugar press filter waste and fly ash under certain curing conditions. Journal of Building Engineering, 44, 102938. https://doi.org/10.1016/j.jobe.2021.102938.
- TS EN 12390-7, Concrete-hardened concrete tests Part 7: Determination of hardened concrete density, 2010. Turkish Standards Institute, Ankara, Turkey.
- TS EN 197-1, Cement. Composition, specifications and conformity criteria for common cements, European Standard, 2011.
- Turker, A.U., Camper, N.D. (2002). Biological activity of common mullein, a medicinal plant. Journal of Ethnopharmacology, 82, 117-125. https://doi.org/10.1016/s0378-8741(02)00186-1.
- Turker, A.U., Gurel, E. (2005). Common mullein (Verbascum thapsus L.): recent advances in research. Phytother Research 19, 733-739. https://doi.org/10.1002/ptr.1653.
- Vyawahare, M. R., Naik, B. (2014). Comparative study on durability of self cured SCC and normally cured SCC, Materials Science, Engineering, 3(8), 1201-1208.
- Xiao, R., Ma, Y., Jiang, X., Zhang, M., Zhang, Y., Wang, Y., Huang, B., He, Q. (2020). Strength, microstructure, efflorescence behavior and environmental impacts of waste glass geopolymers cured at ambient temperature. Journal of Cleaner Production, 252, 119610. https://doi.org/10.1016/j.jclepro.2019.119610.



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