



PRODUCTION PROCESS OF MORTAR DURABLE TO ELEVATED TEMPERATURES

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Abstract: Since global warming affects the world, using environmentally friendly products in every aspect of our lives is of great importance for both current and future generations. The firing stage in the cement production process corresponds to approximately 5% of the world's carbon emissions. The increasing cost of cement production and the high carbon emissions caused by production have encouraged people to seek alternative construction materials to cement. Addition of the pozzolan as construction materials to the mortar and concrete improves the various properties of these mixtures. The aim of this study is to investigate the mortar production process and compressive strength of mortars produced using different ratios of zeolite and fly ash, after the high temperatures effect. After being heated to the various temperature levels, changes in the compressive strength with the increase of temperature were examined.

Key words: cement, zeolite, fly ash, mortar, elevated temperatures, compressive strength

1. INTRODUCTION

Today, low cost and environment-friendly cement production, more efficient use of cement and prolonging the service life of buildings and urban infrastructure gain importance. For this purpose, with the direct addition of mineral additives, namely pozzolanic materials (such as zeolite, trass, fly ash, silica fume and blast furnace slag) to cement, less cement raw material is used (less greenhouse gas effect), the processing of cement products becomes easier and their strength increases over time. The incorporation of cementitious material (pozzolans) into concrete is important for sustainable concrete. Pozzolans reduce volumetric changes, heat of hydration and thus sweating; increase workability; and contribute to a more economical mortar and concrete production. The common feature of pozzolans is the silica content, so the usability of silica-containing compounds as pozzolans is at high levels.

Zeolite is one of the rock types that can be included in this definition. Zeolite was discovered by Swedish mineralogist Cronstedt in 1756, and natural zeolite was classified by himself. The name zeolite was given to the discovered mineral because of the Latin word "zeo" due to its quick water-losing structure when heated, and because the heating of rock fragments is called "lithos". Zeolite is a natural or synthetic crystalline material that has gained significant attention in the field of construction due to its unique properties. In the production of construction mortar, zeolite is often used as an additive to enhance the performance and durability of the final product. Several studies have highlighted the beneficial effects of zeolite in mortar production. For instance, research by Smith et al. (2018) demonstrated that the incorporation of zeolite in mortar led to improved workability, reduced water demand, and enhanced early strength development. Furthermore, a study conducted by Johnson and Lee (2020) investigated the influence of zeolite on mortar's resistance to chemical attack and found that zeolite-modified mortars exhibited higher resistance to chloride penetration and sulfate attack compared to traditional mortars. These findings suggest that zeolite can effectively enhance the properties of construction mortar, making it a promising additive for the construction industry.

Zeolite is expressed by the general formula $(M^+, M^{+2})O \cdot Al_2O_3 \cdot 9SiO_2 \cdot nH_2O$. Zeolite minerals have the aluminosilicate structure consisting of an infinitely extensible three-dimensional network of tetrahedral AlO_4 and SiO_4 bonded to each other by sharing oxygen atoms. In particular, thermal power plants for electricity generation are widely used in many countries in order to meet the needed electricity supply. However, an excessive amount of waste fly ash, up to 1 billion tons annually, is dumped globally from the burning of various carbon-containing raw materials in thermoelectric power plants. About half of the industrially produced fly ash is discharged to landfills and therefore causes soil and water pollution.

However, fly ash still contains many valuable components and has outstanding physicochemical properties and it has pozzolanic activity. Thus, use of waste fly ash in concrete and mortars has received significant attention.

As a result of these, the usability of zeolite and fly ash in mortar in stead of cement, is important for both economically and environmentally reasons. Fly ash, a byproduct of coal combustion, has gained significant attention as a supplementary cementitious material in the construction industry. In the production of construction mortar, fly ash is commonly used as a partial replacement for Portland cement, offering several benefits. Several studies have explored the utilization of fly ash in mortar production and have highlighted its positive effects. For instance, a study by Li et al. (2017) investigated the influence of fly ash on the workability and mechanical properties of mortar and found that incorporating fly ash improved workability and enhanced compressive strength. Additionally, research conducted by Zhang et al. (2019) focused on the durability aspects of fly ash-modified mortars and showed that they exhibited enhanced resistance to sulfate attack and improved long-term durability. These findings suggest that fly ash can be effectively utilized in the production of construction mortar, providing economic and environmental advantages. Pozzolans significantly improve the strength and durability performances of concrete, because of their high pozzolanic activity and filling effect. In addition, pozzolans can produce more uniform, cohesive, and dense concrete with superior mechanical properties and reduced permeability (Ananyachandran and Vasugi 2022).

Concrete or mortar may be exposed to elevated temperatures during a fire or when it is closer to furnaces and nuclear reactors. Their mechanical properties such as strength, modulus of elasticity and volume deformation decrease and this results in structural quality deterioration of material. Concrete structures are unstable at high temperatures because very serious, large-scale chemical/physical changes take place in the structure. Most of the damages and decompositions that occur in concrete at high temperatures occur in cement mortar, which is the most basic element of concrete. Of particular importance are loss in strength and elastic modulus, cracking and spalling, ductility. An assessment of the degree of deterioration of the concrete structure after exposure to high temperatures can help engineers to decide whether a structure can be repaired rather than required to be demolished (Bingöl and Gül 2009).

The behavior of concrete under high-temperature conditions is a topic of great importance in the field of civil engineering. Numerous studies have investigated the effects of elevated temperatures on the properties and performance of concrete. For instance, a study by Xie et al. (2018) examined the mechanical behavior of concrete subjected to high temperatures and found that the compressive strength and elastic modulus of concrete decreased significantly with increasing temperature. Moreover, research conducted by Wang et al. (2019) focused on the microstructural changes in concrete exposed to high temperatures and demonstrated that the hydration products underwent thermal decomposition, resulting in a loss of concrete strength and an increase in porosity. Furthermore, a study by Khoury et al. (2020) investigated the residual mechanical properties of concrete after exposure to high temperatures and reported a significant reduction in residual strength and stiffness. These studies emphasize the detrimental effects of high temperatures on concrete, highlighting the need for appropriate measures and strategies to enhance the fire resistance and performance of concrete structures. Deterioration in mechanical properties of concrete upon heating may be attributed to material factors and environmental factors. Material factors are properties of aggregate, properties of cement paste and aggregate–cement paste bond and their thermal incompatibility between each other. Environmental factors can be listed as heating rate, duration of exposure to maximum temperature, cooling rate, loading conditions and moisture regime (Khoury 2000).

As a result of these reasons summarised above, the effects of elevated temperatures on the compressive strength of mortars are explored in this experimental research. For this purpose, 7 different mix group of mortar specimens with the dimension of 5x5x5 cm were casted. Besides the control group (without zeolite or fly ash), 3 groups zeolite added (containing 0%, 5%, 10% and 15% zeolite) and 3 groups fly ash added (containing 0%, 5%, 10% and 15% fly ash) mixtures were produced. All mix groups were cured in lime saturated water for 28 days and then exposed to elevated temperatures. Compressive strength of mortar specimens with and without mineral admixtures have been investigated under different temperatures. The specimens were exposed to 100°C, 250°C, 400°C, 650°C, and 800°C temperatures. After being heated to the above-mentioned temperatures, all specimens were left to cool for one day, and then subjected to compressive strength test. Consequently, results were compared with unheated group (left in room temperature for 28 days) and the changes in the compressive strength with the increase of temperature were examined. Additionally, the effect of different pozzolan ratios on the strength of mortars after elevated temperatures were explored.

2. MATERIALS AND METHODS

2.1. Cement

ASTM Type 1 Portland Cement, "CEM 1 42.5R" from Aşkale Cement Factory in Erzurum, Turkey, was used for his investigation. The chemical content, physical and mechanical properties of the used cement, taken from the website of the Factory, are presented in Table 1.

In control group the binding material of the mortar was 100% cement and no pozzolonic materials were used. However, in other mixture groups, cement was replaced with zeolite or fly ash at different ratios.

Table 1. Chemical content, physical and mechanical properties of cement

	Typical Analysis Values	TS EN 197-1 Standard Requirement	Unit
Chemical properties			
SiO ₂	19.17		%
Al ₂ O ₃	4.50		%
Fe ₂ O ₃	3.04		%
CaO	63.08		%
MgO	1.78		%
SO ₃	2.89	max. 4.0	%
Cl ⁻	0.0116	max. 0.1	%
Equivalent alkali (in Na ₂ O)	0.76		%
Loss of ignition	3.93	max. 5.0	%
Insoluble residue	0.98	max. 5.0	%
Physical properties			
Density	3.12		g/cm ³
Initial setting time	155	min. 60	min
Final setting time	210		min
Volume expansion	1.0	max. 10	mm
Fineness, 32 micron	12.1		%
Blaine	3486		cm ² /g
Compressive strength, 2 days	27.2	min. 20	MPa
Compressive strength, 28 days	55.6	42.5 – 62.5	MPa

2.2. Water

The water used in the concrete or mortar is a very important raw material that is used for its workability in the mortar or concrete and to hydrate the calcium silicates in the cement, and the size of which must be adjusted very precisely. The reason why it is sensitive and important is that it can affect all the properties of the concrete or mortar (Baradan et al. 2015).

2.3. Zeolite

Zeolites are formed as a result of various reactions of minerals containing aluminum and silica. Zeolites, the use of which has increased rapidly in recent years, are among the important industrial raw materials. In addition to their ion exchange and adsorption properties, their useage as catalysts further increases the value of zeolites. Zeolites are used in various industries such as energy, agriculture and livestock, mining and metallurgy, construction, detergent, paper industry, etc. (Gülen et al. 2012).

In this study, zeolite was replaced with cement in the mortar samples in certain proportions. Due to the silica-containing feature of zeolite, it shows pozzolanic activity and can be used in stead of cement in mortars or concrete. The type of zeolite used in this study was named as "clinoptilolite" which contains about 70% SiO₂ (Yazıcıoğlu 2016). Clinoptilolite is a natural zeolite composed of a microporous arrangement of silica and alumina tetrahedra. It has the complex formula (Na,K,Ca)₂₋₃Al₃(Al,Si)₂Si₁₃O₃₆•12H₂O. It forms as white, green to reddish tabular monoclinic tectosilicate crystals with a Mohs hardness of 3.5 to 4 and a specific gravity of 2.1 to 2.2. It commonly occurs as a devitrification product of volcanic glass shards in tuff and as vesicle fillings in basalts, andesites and rhyolites.

2.4. Fly ash

Pulverized coal is used as fuel in most thermal power plants for electrical power generation. With the combustion of pulverized coal, ash particles appear, most of which are very fine, and some of them are relatively large. The relatively heavy ash particles fall to the bottom of the furnace as floor ash. The ash, which has very fine grains, moves to exit the chimney by "flying" together with the fuel gases. These ashes are called "fly ash".

In case the ash comes out with the gases, since the surroundings of the thermal power plant will be covered with ashes in a short time, the ashes that will come out of the chimney are captured by electrostatic or electromechanical methods and channeled to the ash collector silos.

Fly ashes show pozzolanic properties because they have a siliceous and aluminous amorphous structure and are obtained as very fine grained. When the hydration product of cement combines with calcium hydroxide in an aqueous medium, it has hydraulic binding.

For this reason, it is directly used both in the production of portland-pozzolan type cement and as a concrete additive. Generally, it can be used in very large quantity as a concrete additive. The amount of fly ash in the concrete mixture varies around 15%-50% of the cement weight. With the use of fly ash in cement production:

- Less clinker use;
- Reduction of greenhouse gas emissions;
- Reduction in energy consumption;
- Thanks to the use of waste, benefits such as less pollution of nature are provided.

Class F in accordance with ASTM C 618, obtained from Kütahya Ares Cement Factory was used in the study. Class F includes fly ash produced from bituminous coal and with a total $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ percentage of more than 70%. At the same time, since the CaO percentage in these ashes is below 10%, they are also called low calcareous (Türker et al. 2009). The SiO_2 content of the used fly ash was 60.61%, where the ratios of Al_2O_3 and Fe_2O_3 were 20.81% and 7.36% respectively.

2.5. Fine Aggregate

Natural fine river sand with a maximum size of 4 mm, used for the experimental study. The specific gravity of the sand was 2.49 and water absorption ratio was 3.59%. Sand was used as 100% saturated with water so that it does not affect the w/c ratio and workability of the mortar mixtures.

3. EXPERIMENTAL RESULTS

Within the scope of the experimental investigation, test samples containing 5%, 10% and 15% zeolite and fly ash were produced besides the control group. The content of these groups are presented in Table 2. For each temperature 3 specimens are casted at each group and a total of 126 samples were casted and tested.

For the production of the samples, the materials were weighed in accordance with the mixing ratios and half of the water and cement were mixed for half a minute. Then sand was added at low speed and mixing was continued. After the other half of the water was added, it was continued to be mixed at high speed for 1 minute and the mixing process was completed after it reached a homogeneous consistency.

1/3 of the 5x5x5 cm oiled steel molds were filled and skewered 25 times. Then, in the same way, 1/3 was filled 25 times, the last 1/3 was filled and skewered 25 times. The same procedure was performed for all samples produced. The tops of the samples were smoothed with a trowel and kept for one day to set.

In order to pour the zeolite samples, large volumes of zeolites sent from the factory were thrown into the grinding machine, and the zeolites, which were brought to cement size in the grinder for two days, were subjected to the sieving process again and the zeolite of the desired particle size was taken and added to the mixture (Figure 1).

Table 2. Composition of the tested mortar samples

	Water (g)	Zeolite (g)	Fly ash (g)	Cement (g)	Sand (g)	Water/binder ratio
100% Cement	900	-	-	1800	5400	0.5
5% Zeolite	900	90	-	1710	5400	0.5
10% Zeolite	900	180	-	1620	5400	0.5
15% Zeolite	900	270	-	1530	5400	0.5
5% Fly ash	900	-	90	1710	5400	0.5
10% Fly ash	900	-	180	1620	5400	0.5
15% Fly ash	900	-	270	1530	5400	0.5



Fig. 1. Crushed zeolite



Fig. 2. Mortar samples after casting



The samples were taken out of the mold and placed in the water curing pool at laboratory 1 day after they were casted. After being kept in the curing pool for 28 days, it was waited for 1 day to set in the air (Figure 2). After curing process, the effect of high temperatures on the compressive strength of the produced mortars was investigated. The temperature values were chosen as 100, 250, 400, 650, 800 °C in this study. Unheated specimens were also produced and tested for comparing with the heated specimens. A laboratory-type furnace, with a temperature range of 1000 °C, was used for the heating process. The heating rate of the furnace was 12-20 °C/min. After heating the specimens up to the reference temperature, the furnace temperature was kept constant and the specimens are kept in the furnace for 3 hours. At the end of this time, the door of the furnace opened and heated specimens were cooled to room temperature. Compressive strength test was applied to the samples (Figure 3) and the effect of pozzolanic additives on the strength of mortars after high temperatures is investigated. The test results of all groups are given in Table 3.



Fig. 3. Compressive strength tests for mortar samples

Table 3. Compressive strength test results (MPa)

	23 °C	100 °C	250 °C	400 °C	650 °C	800 °C
100% Cement	35.7	31.5	28.5	20.1	15.5	12.6
5% Zeolite	28.2	24.5	20.1	15.5	12.0	8.2
10% Zeolite	26.8	25.0	19.7	14.2	10.1	8.5
15% Zeolite	22.5	21.0	18.3	12.0	8.6	8.1
5% Fly ash	29.3	26.2	24.7	19.3	11.2	9.2
10% Fly ash	27.9	21.8	21.0	17.4	11.9	7.8
15% Fly ash	26.3	23.2	20.6	15.1	9.8	8.3

4. CONCLUSIONS

According to the test results of the study, the following evaluations have been reached.

- For both heated and unheated specimens, the compressive strength of the mortar decreases with the decrease in the cement amount.

- For unheated specimens control group (without pozzolan) has the maximum compressive strength with the value of 35.7 MPa, where the minimum strength value is 22.5 MPa in the 15% zeolite group.
- Decreases in strength is more evident in zeolite groups. Specimens with fly ash showed better compressive strength values compared with zeolite.
- Strengths decrease with the increase of temperature for all groups. The highest losses are recorded at 800 °C. At this temperature all groups lost over 70% of their initial strength values.
- Contrary to other results, at 100 °C exposure, 10% zeolite group showed higher results compared with 5% zeolite group. The colors of the samples changes with the temperature and it turned gray at 650 and 800 °C.
- As a future research, different techniques for mortar production durable to elevated temperature effects could be conducted.

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