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Journal of Science and Technology

E-ISSN 2146-7706

Pozzolanic activity of pumice under different curing temperatures and durations

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A R T I C L E I N F O

Article history:
Received 4 November 2022
Received in revised form 6 December2022
Accepted 27 December 2022

Keywords:
Pumice
Pozzolans
Pozzolanic activity
Sustainability

A B S T R A C T

Objective of the work: This study focused on the investigations of the pozzolanic activity of the pumice under different curing temperatures and durations.

Materials and Method: Pumice powder, standard sand, and slaked lime were used as materials in this study. Within the scope of the study, one mixture design was used following TS 25. In total, 9 different curing regimes such as three different heat cures at 55˚C, 75˚C, and 90˚C and three different curing durations as 7, 14, and 28 days for each curing temperature. Some experimental analyses including compressive strength, flexural strength, and ultrasonic pulse velocity test were conducted to investigate the effect of the curing regimes on the pozzolanic activity of pumice.

Results: The pozzolanic activity of pumice increased to a certain extent with increasing curing temperature and duration. The optimum curing regime obtained for the pozzolanic activity of pumice in this study was curing at 75 ˚C for 28 days, which the highest strength values were obtained with.

1. Introduction

Sustainability is the main concern in societies due to contributing to the persistence of the environment through the preservation of natural sources. Sustainable practices support ecological, human, and economic health by arising environmental consciousness in modern construction works. The enormous amounts of raw materials and energy consumption for manufacturing construction materials are significant factors that are related to sustainability (Elizondo-Martínez et al., 2020; Chandrappa and Biligiri, 2016, URL-1; Alaskar et al., 2021; Karasin et al., 2022; İpek, 2022). These manufacturing processes are also responsible for a significant amount of greenhouse gases released in nature. Concrete is the most used construction material with water owing to its versatility and availability all over the world. Being such a crucial material for the construction industry, the sustainability of concrete has vital importance for societies and sector representatives. The sustainability of concrete is maintained with its constituents, especially with cement which is the foremost ingredient of concrete. However, the production of one ton of Portland cement releases approximately one ton of carbon dioxide (CO_2) and other greenhouse gases. Since cement production consumes a large amount of energy and releases carbon dioxide (CO_2), a greenhouse gas type, into nature in significant amounts, societies and researchers are looking for alternative materials that can be substituted for cement to minimize its usage in concrete, although it is an inevitable ingredient of concrete. Therefore, replacing the cement in the concrete with a more secondary binding material (mineral additive) than general use is one of the most applied and easiest methods to produce more innovative and sustainable concrete (Güneyisi et al., 2014; Justnes and Martius-Hammer, 2015; Naik, 2008; Pereira et al., 2013). Today, pozzolans are used as a substitute material for cement in concrete production. Pozzolans are natural or by-product materials that do not naturally have

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cementitious properties. However, when finely ground, they are siliceous or siliceous and aluminous materials that react with calcium hydroxide, also known as portlandite, in an aqueous medium and exhibit cementitious properties. And, the ability of the active silica in pozzolan to react with calcium hydroxide and water is defined as pozzolanic activity (Massazza, 1993). In addition to silica and aluminum which are the main oxides of pozzolans, they can contain iron oxide, calcium oxide, alkaline, and carbon. The amount of oxides depends on the source of pozzolans. Pozzolans were first used about two thousand years ago during the time of the ancient Romans. In the Italian town of Pozzuoli, volcanic ash was first found by the Romans to be a binding material when mixed with water and slaked lime. They are generally divided into two groups natural and by-product or artificial products. Natural pozzolans are usually obtained with the method of break or ground. Volcanic glasses, volcanic tuffs, trasses, and some clays are types of natural pozzolans. The artificial pozzolans are acquired as by-products from some activities of industry sectors. Silica fume, fly ash and large furnace slag are the most well-known artificial pozzolans. In addition to the utilization of pozzolan as cement substitute material in concrete, they are used to improve workability, minimize segregation, reduce the hydration heat, prevent the detrimental effects of alkali-silica reactions, cut down the permeability, enhance the ultimate strength and increase the durability against sulfate in concrete. When pozzolans and Portland cement mixture are reacting, the free lime (calcium oxide or portlandite) in the cementitious mixture decrease due to the pozzolanic reaction. After a while, there are more calcium-silicate-hydrate (C-S-H) products in mixtures with pozzolans compared to the Portland cement mixtures. Containing more C-S-H gels that have the higher binding characteristic of pozzolanic concretes results in increasing the strength and durability of concrete against environmental effects. How well the reaction between the pozzolan in the concrete mixture and the hydrated lime is determined by the pozzolanic activity. For pozzolans to be used in Portland cement concrete, they must have sufficient pozzolanic activity (Bozkurt and Karaca, 2019; Erdoğan, 2016; Güner and Süme, 2000).

In general, four types of methods are used to determine the pozzolanic activity of a pozzolan. These are:

- Chemical methods
- Physical methods
- Mechanic methods
- Micro-structural methods.

The chemical method is one of the methods for directly determining the pozzolanic activity. The method is used to determine the chemical content of the material. This method is based on determining the change in calcium hydroxide that interacts with active minerals in pozzolans by using chemical titration techniques while the reaction is going on between pozzolan and the cementitious mixture (Bulut and Tanaçan, 2009; Kurugöl, 2017).

In the physical method, the pozzolanic activity is determined by physical changes that occurred in the structure of the pozzolan. The physical changes are determined by measuring the changing in electrical conductivity at various times. Another method of determining the pozzolanic activity with the physical method is to measure the increase in the specific surface of cement in the mixture. And, the mechanic method is based on determining the compressive strength of the mixture.

In micro-structural methods, some methods such as petrographic analysis, electron microscope, differential thermal analysis (DTA), thermogravimetric analysis (TG), X-ray powder diffraction (XRD), X-ray Fluorescence (XRF) are utilized to determine the pozzolanic activity. The petrographic analysis is used to determine the morphology that are resulting from reactions. An electron microscope presents a chance to study cement hydration products in detail. DTA analysis is based on a comparison of the heat given (exothermic) or taken (endothermic) heat when a substance undergoes physical and chemical changes. The endothermic peaks seen in the DTA graph represent the water loss or decomposition, and the exothermic reaction peaks are indicative of representing the formation of a new composition. TG analysis shows the weight loss of material under the effect of heat. Thus, DTA and TG provide to determine the quantity of C-S-H gels and consumed calcium hydroxide. XRD shows the graph of crystalline minerals in the mixture to detect the hydration products. XRF allows to determine the pozzolanic activity by stating the SiO_2, Al_2O_3, Fe_2O_3 oxides as percentage in pozzolan (Bulut and Tanaçan, 2009; Chen, 1984; Joshi, 1970; Kurugöl, 2017).

Pumice is a lightweight aggregate of volcanic origin formed during the solidification of molten lava. The porous nature of pumice is due to the formation of tiny air spaces when the gases in the molten lava are trapped as it cools (Kabay et al., 2015; Yücel et al., 2020). Pumice has been used as an aggregate and/or mineral admixture in the concrete industry (Öz, 2018). The usage of pumice powder (PP) as a natural pozzolan in the cement industry is quite promising for sustainable development (Gencel, 2015). When reviewing the literature, there have been some studies that focused on the pozzolanic activity of pumice under different circumstances. Mboya et al. (2017) examined the pozzolanic activity of cement mixtures with pumice additives and stated that the pozzolanic activity effect of pumice emerged after 28 days of curing and caused an increase in the strength of cement mixtures. Sarıdemir et al. (2016) indicated that the addition of ground pumice in the ratio of 5% increased the compressive strength of high-strength concrete, which was attributed to the cementitious properties of pumice. In their study, Kılıç and Sertabipoğlu (2015) stated that replacing the cement with natural and heated pumice separately up to 15% had a positive effect on the compressive strength of the cement mortar, which was also attributed to the cementitious properties of the pumice. In addition, the authors reported that the heat treatment (1000˚C) applied to pumice significantly affected the pozzolanic activity of pumice, resulting in a much greater increase in the strength of the mortar compared to that of natural pumice. Yu et al. (2020) proved the pozzolanic characteristics of pumice by microstructural and mechanic methods and stated that the pumice satisfies the pozzolanic activity index criteria specified in Chinese Standard JG/T 315. Kabay et al. (2015) stated that replacing cement with pumice powder caused lower mechanical strength at early ages, but increased the strength of pumice at later ages compared to reference concrete, which they attributed to the pozzolanic effect of pumice. This study presented here investigated the pozzolanic activity of pumice under different curing temperatures and durations by mechanic test method following TS 25 (2008) which is a Turkish standard about natural pozzolan (trass) for use in cement and concrete definitions, requirements, and conformity criteria. The X-ray fluorescence (XRF) and X-ray powder diffraction (XRD) were used in the context of the microstructural investigations of the pumice. The compressive strength of specimens was defined for each curing condition to investigate the pozzolanic activity following the TS 25 (2008) standard. However, flexural strength and ultrasonic pulse velocity (UPV) tests were also performed on the test specimens to make comparisons.

2. Experimental Study

2.1. Materials

In this study, pumice obtained from the Bitlis region with a specific gravity of 2.35 was used to produce mortar specimens. The specific surface area of the pumice was 4250 cm^2/g. The pumice was ground by the grinder and drought in a drying oven at 105 ± 5 ˚C until obtaining constant weight. It was prepared in such a way that the amount remaining on the sieve with a 200-micron mesh size would be at most 0.6% by mass, and the amount on the sieve with a 90-micron mesh size would be at the most 8% by mass. The chemical analysis of pumice was determined with the X-ray fluorescence (XRF) method and the mineral phase analysis was conducted with the X-ray powder diffraction (XRD) method. The specific gravity of slaked lime used in this study was 2.40. The slaked lime contained a minimum of 95% CaO and a maximum of 5% MgO. The proportion of slaked slake remained over 45μ was at most 5%. Standard CEN sand with a specific gravity of 2.56 was obtained from Limak Cement Factory to prepare the mortar specimens following the TS EN 196-1 (2016). The grain size distribution of sand is presented in Table 1. Bitlis city mains water, which does not contain harmful organic matter and mineral salts, was used in the production of the mortar samples.

Table 1. Grain size distribution of standard sand.

Aperture size (mm)	Cumulative Remaining %
2,00	0
1,60	7 ± 5
1,00	33 ± 5
0,50	67 ± 5
0,16	87 ± 5
0,08	99 ± 1
Moisture	0.20 %

2.2 Mixture proportioning and curing

The pozzolanic activity test was performed following the standard of TS EN 196-1 (2016) and TS 25 (2018). The required amount of material to prepare three mortar test specimens is given in Table 2. The molds were covered with a 6 mm thick glass plate measuring 210 mm x 185 mm to prevent evaporation after pouring. They were kept at room temperature (23 ± 2) °C for 24 hours. After 24 hours, without removing the molds, they were kept in a drying oven at (55 ± 2) °C for another 6 days. The mortar samples are taken out of the oven and allowed to cool down to room temperature for being ready to test the procedure. At the end of this period of at least 4 hours, the samples are subjected to strength tests. In addition to keeping specimens at 55 ± 2 °C for 6 days referred to as reference specimens in TS 25 (2008), the effect of different curing temperatures and duration was investigated throughout the study. The different curing regimes and Mix IDs are explained in Table 3. For example, P-75-14 means keeping the specimen at room temperature (23 ± 2) ˚C for 24 hours and after in a drying oven at (75 ± 2) ˚C for 13 days.

Table 2. Pozzolanic activity test mixture proportions.

Materials	TS 25 (g)	Proportions (g)
Slaked Lime [$Ca(OH)_2$]	150	150
Pumice	2×150×(S.G* of Pumice/S.G* of Slaked Lime)	294
Standard Sand	1350	1350
Water	0.5×(150+Pumice)	222

**S.G: Specific gravity*

2.3. Test specimens and methods

Prismatic specimens of 40 × 40 × 160 mm dimensions were produced for testing flexural strength and ultrasonic pulse velocity tests. The compressive strength test was conducted on half specimens obtained after the flexural strength test. The strength tests were conducted by a fully automatic computer-controlled cement compression and flexural test press that is capable of applying loads up to 10 kN and 200 kN for flexural and compressive strength tests respectively following TS EN 196-1 (2016). Each experimental parameter was determined by averaging the results of three samples. Ultrasonic pulse velocity (UPV) tests were conducted on specimens before the flexural strength test was performed. Strength properties without damaging the test specimen using an ultrasonic pulse velocity device can be obtained. The time it takes for the ultrasonic sound waves sent into the sample to reach the other side of the sample is measured and the wave velocity is calculated. With this wave velocity, the strength properties of the sample can be estimated. After determining the time taken for the sound waves sent from one end of the sample to reach the other end during the experiment, the wave velocity is calculated with the following formula (Cosmes-López et al., 2017; Demirhan et al., 2019; Massazza, 1993):

$$V = \frac{S}{t} \times 10^3 \tag{1}$$

Where;

V= Ultrasonic pulse velocity (m/s)

S= The Distance between the two ends of the sample (m)

t= Time taken by the pulse to go through (microseconds).

The microstructural analyses including XRF and XRD were conducted on the ground pumice specimen. The XRF analysis was performed at Kastamonu University Central Research Laboratory by Spectro-Xepos II device. The XRF spectroscopy provides the opportunity to determine elemental composition (URL-2). The XRD analysis was made on the specimen to investigate the crystal structure and phase analysis of pumice at Bingöl University Central Laboratory Application and Research Center by Rigaku Ultima IV device (URL-3).

Table 3. The curing regimes and Mixture ID.

Temperature (°C)	Mixture Proportions	Curing duration (days)	Mix ID
55 °C	Mixture proportions given in Table 2	7	P-55-7
55 °C		14	P-55-14
55 °C		28	P-55-28
75 °C		7	P-75-7
75 °C		14	P-75-14
75 °C		28	P-75-28
90 °C		7	P-90-7
90 °C		14	P-90-14
90 °C		28	P-90-28

3. Results and Discussion

3.1. Microstructural analysis

The chemical analysis of pumice was submitted in Table 4. TS 25 (2008) states that the total amount of (SiO_2 + Al_2O_3 + Fe_2O_3) of a natural pozzolan must be at least 70% by mass. And, the amount of SO_3 and Cl must be at most 3% and 0.1% respectively. When Table 5 is analyzed, it is seen that pumice meets these criteria. The pumice sample used in this study obtained from the Bitlis region has a similar chemical composition to the samples taken from other pumice deposits in Turkey presented in the literature such as Nevşehir (Tolğay et al., 2004), Isparta Gelincik (Kalay, 2010), Manisa Kula (Coşan, 2016), Kayseri (Gündüz and Yılmaz, 1998) and İzmir Menderes (Aksay, 2005) as summarized in Table 5. When analyzing Table 5, it was seen that in general pumice specimens have a total percentage of SiO_2+Al_2O_3+ Fe_2O_3 oxides between 80% and 90% except that of Manisa Kula. Therefore, the pumice obtained from Manisa Kula can show higher pozzolanic activity.

Table 4. Chemical compositions of Pumice**.**

Chemical Analysis, (%)	TS 25	Pumice
Na_2O	-	7.87
MgO	-	0.03
SiO_2	≥70	72.06
Al_2O_3		11.83
Fe_2O_3		3.91
P_2O_5	-	0.01
SO_3	≤ 3.0	0.01
Cl	≤ 0.1	0.06
K_2O	-	3.70
CaO	-	0.44
MnO	-	0.08

Table 5. The (SiO_2+Al_2O_3+Fe_2O_3) oxides comparison of pumice specimens from different regions in Turkey.

Pumice Origin	(SiO_2+Al_2O_3+Fe_2O_3) (%)
Bitlis	87.80
Nevşehir (Tolğay et al., 2004)	88.32
Isparta Gelincik (Kalay, 2010)	81.20
Manisa Kula (Coşan, 2016)	95.17
Kayseri (Gündüz and Yılmaz, 1998)	86.50
İzmir Menderes (Aksay, 2005)	87.14

The XRD pattern of the pumice is presented in Fig. 1. The pumice was found that it has a very broad reflection (peak) between 20° and 30° confirming the presence of the amorphous nature of this material. In addition, the main crystalline mineral phases were identified as albite-calcian, anorthoclase, and quartz.

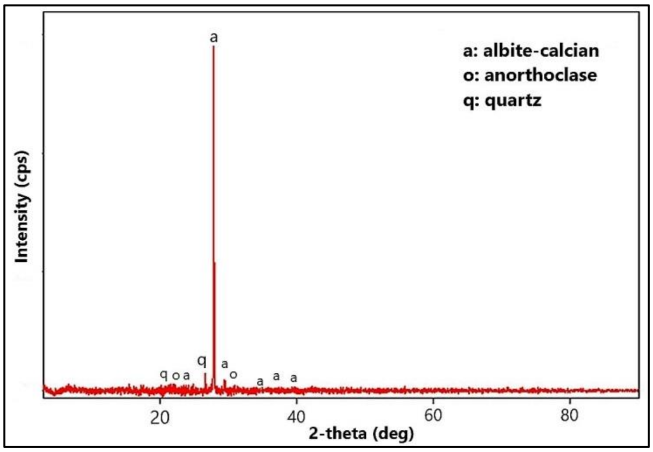

Figure 1. The X-ray diffraction pattern of pumice.

3.2. Strength tests and UPV analyses

The strength tests and UPV analysis results are presented in Table 6. The TS 25 (2008) standard states that the mixture prepared with natural pozzolan and lime must have a compressive strength of at least 4 MPa to meet the pozzolanic activity criteria after curing at room temperature (23 ± 2) °C for 24 hours and at 55 ± 2 ˚C for 6 days. In the TS 25 standard published in 1975 and repealed, the flexural test criterion was also specified among the pozzolanic activity tests, as well as the compression test, and the flexural strength of the sample was requested to be at least 1 MPa. However, in the current standard of TS 25 (2008), this criterion is no longer required. Although it is not specified in the standard, to make a comparison, the flexural strength tests were performed in the context of this study. On the other hand, the UPV values of specimens were conducted to support the strength test results. When Table 5 is analyzed, it is seen that the maximum compressive and flexural strength was obtained with P-75-28 ID specimen as 8.90 MPa and 2.13 MPa, respectively. Also, the highest UPV value was obtained with this specimen as 3799 m/s. The lowest compressive and flexural strength results were obtained with a specimen of P-55-7 ID as 6.48 MPa and 1.77 MPa, respectively. In addition, the lowest UPV value was obtained for the same specimen as 2450 m/s. It is concluded from Table 5 that, increasing both curing temperature and duration increases the strength of the specimen up to a level. This was attributed to the increase of the pozzolanic reactions with temperature and exposure time. Increasing pozzolanic reactions means producing more C-S-H gel by consuming calcium hydroxide. However, an excessive increase in curing temperature can result in a decrease in strength as seen in curing at 90 ˚C in Table 5, which has been attributed to lower UPV values, i.e. damage to the microstructure, as seen in the table, meaning an increase in void formation. The pozzolanic activities of pumice under different curing durations and temperatures based on compressive strength are presented in Fig. 2. The change in flexural strength and UPV values of mixtures under different curing durations and temperatures are demonstrated in Fig. 3 and Fig. 4 respectively.

In a similar study conducted to reveal the pozzolanic activity of perlite, it was stated that as the curing duration increased, the pozzolanic activity increased, however, it was affected adversely by the increase of curing temperature from 55°C to 65°C. When comparing the pozzolanic activity of perlite and pumice based on compressive strength specimens cured at 55 °C for 7 days, it was revealed that perlite had compressive strength approximately 62.04% higher than that of pumice (Bulut and Tanaçan, 2009). In another study, Baki et al. (2019) investigated the pozzolanic activity of rhyolite, fly ash, trachyte, and blast furnace slag for 7 days at 55 °C and found approximately 7, 7.2, 8.2, and 8.6 MPa, respectively. This means each of them shows higher pozzolanic activity than pumice.

Table 6. The strength tests and UPV analyses results.

Mixture ID	Compressive Strength (MPa)	Flexural Strength (MPa)	UPV(m/s)
P-55-7	6.48	1.77	3450
P-55-14	7.27	1.83	3573
P-55-28	8.39	1.98	3628
P-75-7	6.97	1.89	3475
P-75-14	7.83	2.01	3684
P-75-28	8.90	2.13	3799
P-90-7	6.27	1.76	3387
P-90-14	6.12	1.73	3361
P-90-28	6.01	1.68	3302

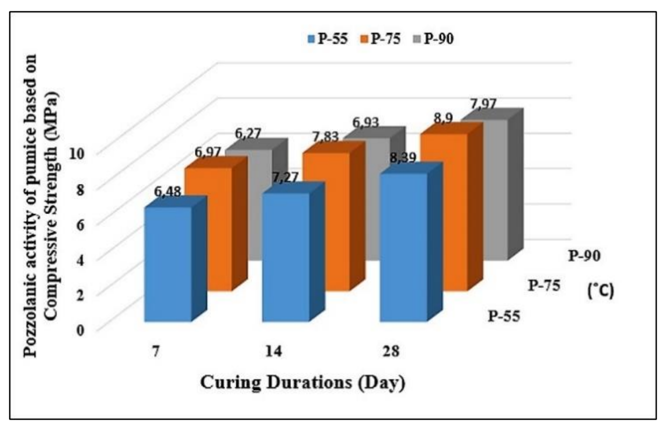

Figure 2. Change in pozzolanic activity of pumice under different curing durations and temperatures based on compressive strength.

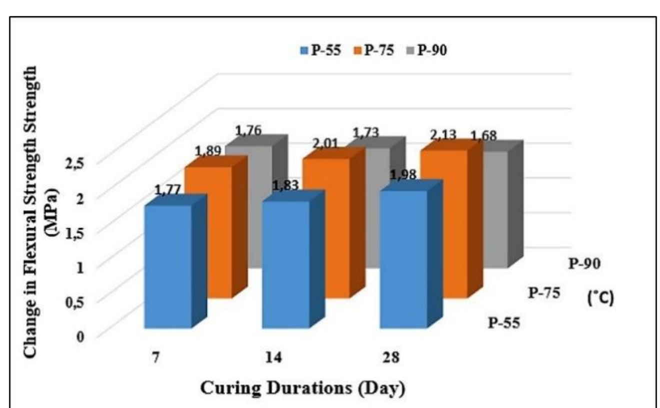

Figure 3. Change in flexural strength of mixtures under different curing durations and temperatures.

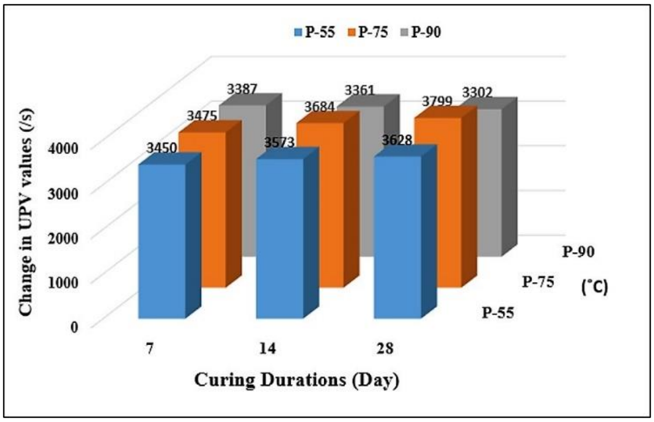

Figure 4. Change in UPV values of mixtures under different curing durations and temperatures.

4. Conclusions

The pozzolanic activity of pumice was affected by both curing temperature and duration. Increasing the curing temperature from 55°C to 75°C caused an increase in the strength of the mortar prepared with pumice powder. With the prolongation of the curing duration, the strength properties of the mortars have also improved. However, increasing the curing temperature up to 90°C resulted in a decrease in the strength properties of mortars. In addition, the results also showed that the strength of mortars decreased with increasing curing duration at 90°C. These results were attributed to the increase in the pozzolanic activity of pumice up to a certain curing temperature and duration, which means producing more C-S-H gels with consuming calcium hydroxide. Moreover, increasing the temperature after a certain level caused a reduction in the strength properties, which was attributed to the damage of the excessive temperature to the microstructure. Based on the findings, it will be useful to determine the possibilities of developing pumice as a building material by using its pozzolanic characteristics at different temperatures and using it together with binders such as cement and lime.

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