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RESEARCH ARTICLE

EVALUATION OF CAPPADOCIA PERLITES AS A BUILDING MATERIAL WITH NATURAL STONE RESIDUES

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ABSTRACT

The superior properties of perlite (thermal insulation, sound insulation, fire resistance, lightness) have led to the investigation of the possibilities of perlite usage in many areas. Although perlite has pozzolanic properties and does not have reserve problems in our country, it has not been a preferred material by cement manufacturers. The use of marble travertine residues in cement production is not very common in our country, although some cement manufacturers support it. In this study, the availability of cement samples produced by the use of natural perlite and marble travertine residues belonging to Nevşehir Acıgöl region in pozzolanic and doped Cements was investigated. In this study, the availability of cement samples produced by the use of natural perlite and marble travertine residues belonging to Nevşehir Acıgöl region in pozzolanic and doped Cements was investigated. Portland cement 5%, 10%, 15% and 20% marble travertine residues and perlite were replaced by pozzolanic and doped Cements and their properties were tested and their suitability investigated. In the experimental studies, 4 types of perlite-doped pozzolanic cement, 4 types of marble travertine-doped cement and composite Cements were produced. In the study, density, cement fineness, setting Times, water content, volume expansion, compressive strength tests were performed on the produced cements under laboratory conditions. The physical and chemical properties of the blended cements produced were compared with Portland cement. When the results of the experiment were examined, the increase in contribution rates decreased the pressure resistances of the concretes. The setting times of cement mortars increased and also contributed positively to the workability of concrete. There was no adverse effect on volume expansion. The pozzolanic and doped cements produced with perlite and marble travertine residues are within the standards according to TS en 197-1 and in cement production of both materials separately to 20 % and together up to 40% pozzolanic cement production up to 35% Portland Composite cement production can be used.

Keywords: *Marble and travertine residues, Perlite, Pozzolanic Cement, Physical Properties, Mechanical Properties*

1. INTRODUCTION

The United States is one of the world's largest producers and consumers of crude perlite and extended perlite, followed by China, Greece, Italy, the Philippines, Mexico and Turkey, respectively. Although 21% of the world's reserves are found in Turkey, 97% of perlite production is done by other countries.

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Although most of the countries that have active role in the world cement sector have perlite reserves, there are very few studies on this subject [1]. Many of the academic studies have focused on the use and development of perlite as aggregates in the production of concrete rather than evaluating it as an additive material in the production of cement. It is known that pozzolanic materials are used as additive materials in cement production [2,3]. However, natural perlites also have pozzolanic properties and unfortunately the use of perlite in cement production is not very common. For this reason, researchers have great duties for the use of natural perlite in cement production [4].

Research shows that the use of mineral additives is widespread in cement production in order to reduce cement production costs. The use of Mineral additives has positive effects on concrete strength in parallel to the reduction of production costs and contributes to an environmental production by reducing CO_2 emissions [5,6]. Pozzolanic cements are of strategic importance in the concrete industry as they are cheaper than Portland cements and can be used for up to 50% of cement weight in the concrete mix. Moreover, the increasing workability of concrete, reducing the speed and amount of hydration temperature, increasing the final pressure resistance, providing affordability and finally reducing the alkali-aggregate reaction has greatly increased the preferability of pozzolanic cements in recent years [6]. In order to realize all these developments, pozzolanic materials such as volcanic ash, volcanic tuff, diatomite soil and baked clay, fly ash, granulated blast furnace slag, silica smoke, and rice husk ash are used. It is possible to achieve all positive developments shown and proven in both cement and concrete with natural perlite. The reason for this can be explained by the natural perlite has a pozzolanic property and sufficient reactive silica. In addition, fly ash, granulated blast furnace slag, silica fume and rice husk ash are artificial pozzolans while perlite has natural pozzolanic properties. Its features may vary depending on the region and the region it is located. The results from a natural pozzolan taken from a particular region are very difficult to predict. Therefore, it would be useful to determine the behaviour of natural pozzolans with limited variability and high reserve [7- 9].

International academic studies have focused on its use in concrete for natural perlite. Some of those; Erdoğan (2013), Early-age activation of cement pastes and mortars containing ground perlite as a pozzolan; Liu (2014), Thermal properties of lightweight dry-mix shotcrete containing expanded perlite aggregate; Yu (2003), Investigation on pozzolanic effect of perlite powder in concrete; Patchta (2019), Development and testing of grouts based on perlite by-products and lime are titled studies. Their common features are on improving concrete properties by using natural perlite or expanded perlite as the main product or by-product [9-11].

Erdem et al. [1] Use of perlite as a pozzolanic addition in producing blended cements, published a study called. The pozzolanic property of perlite, and grindability have investigated. For this purpose, natural perlites taken from two different regions of Turkey (Izmir and Erzincan) for the pozzolanic materials standards have been tested. Cement samples were produced with the addition of 20% or 30% perlite. Turkish perlites were found to have amorphous structure and conform to ASTM C 618. Therefore, it was concluded that perlite samples from Izmir and Erzincan region have sufficient pozzolanic properties for use in cement and concrete industry. The study concluded that grinding perlite was easier than Portland cement clinkers, and that less energy would be needed to produce blended cement by combining perlite and clinker. Cement standards allow the use of pozzolanic materials up to 55%. The study could be tried for higher contribution rates in this commitment. Moreover, due to its low cost, especially in Turkey, Cem IV 32,5/ R-B pozzolanic cements, which are cheap and can reach high strength in the long term, are preferred in many countries.

42% of the world's marble reserves are located in Turkey. Many academic studies have been carried out in recent years to evaluate the residues in marble quarries and processing plants. It is possible to use marble residues in almost all industrial areas. As with perlite, marble waste is predominantly considered as a concrete aggregate. However, the standards allow the use of limestone for some types of cement. Due to its similar chemical structure, marble and travertine wastes are the best competitors to limestone. The use of marble and travertine residues in cement production will help to protect the environment by reducing the use of limited natural raw material resources, preventing the destruction of nature and eliminating the problems associated with the deterioration of the natural balance that will occur as a result of accumulation of marble and travertine residues. In this way, it will be contributed to the green production approach, which has become a slogan recently [12-14].

For all these purposes, industrial raw materials from two different regions of Turkey (Cappadocia perlites; opium marble travertine mix residues) were tested in order to determine the suitability of their availability in cement production. Natural perlite and marble travertine mix residues were ground separately and together and cement samples were prepared. The performance of natural perlite as pozzolan in blended Cements has been investigated. Different amounts of perlite and marble travertine residual mixture (5%, 10%, 15% and 20% perlite by weight, marble travertine residual mixture, perlite + marble travertine residual mixture) cement mixtures were produced in different proportions. The following tests were conducted to determine the performance of the Cements: particle size distribution with grinding, normal consistency, robustness, setting time and compressive strength.

2. MATERIAL AND METHOD

2.1 Materials Used in Experimental Studies

Portland cement clinker, gypsum (Yıldızeli), marble travertine mix residues (Afyon), natural perlite (Nevşehir), standard sand and water were used in the preparation of cement mixtures. The sand used in the study is standard Rilem sand. The substance used was dilithium tetraborate (merck), lithium iodide (merck), ethylene glycol (merck), HCl (merck) Ca $(OH)_2$ and $CaCO_3$. Both the chemical test results and the mechanical strength prove that the material conforms to the limits specified in the relevant standard (TS EN 196-1:2009).

2.1.1 Natural perlite

The pozzolan tested was natural tuff taken from Cappadocia region that is generally used for brick production. These chemical analyses are observed by İstanbul University Central Laboratory. Before grinding, pozzolans are oven dried at 100°C.The chemical and physical analyzes of the natural perlite used in the experiments were carried out and their properties were determined. The chemical and physical analysis of the natural perlite sample is given in Table 1. According to ASTM C 311, to be called as pozzolan, a material should satisfy strength activity index parameters, so, the first test applied to material was strength activity. At the end of 7 and 28 days, the compressive strength of mortars those have 20% natural tuff in them, are tested and compared to the control cubes, without any pozzolan in it, and the results are given in Table 1. Its chemical composition is shown in Table 1. The chemical analysis result of the perlite sample is given in Table 4.

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Table 1. Strength activity index of natural pozzolan.

Blaine Fineness (cm^2/gr)	Flow (%)	Strength Activity Index (%)	
		7 days	28 days
4125	104	80	81

According to ASTM, strength activity after 7 and 28 days, should be at least 75%. Also, according to Turkish and European Standards, the ratio is 70%. The pozzolan, as shown in Table 1. satisfied the requirements of both standards, and received the name of pozzolan after strength activity test. Also, it was observed that the ratio increased by the time passes.

2.1.2 Portland cement clinker

The clinker is obtained from Çimsa Afyon Cement Factory. XRF; (Atomica technical panalytical axios model) method was used for chemical analysis and this clinker was used in experiments. The specific mass of clinker used was 3.17 g / cm^3. The chemical analysis result of the clinker sample is given in Table 4.

2.1.3 Gypsum

The gypsum was obtained from the Tokat Adoçim Cement factory and the chemical analysis was performed by XRF and the amount of crystal water was determined. The chemical analysis result of the gypsum sample is given in Table 4.

2.1.4 Marble travertine mix residues

The marble travertine mix residues were obtained from the waste fields of Afyon natural stone factories. The marble travertine mix residues used in the experiments were blended in the laboratory and made composites. Chemical and physical analyzes of the prepared samples were tested and their properties were determined (Table 4).

2.1.5. Cements and Codes

Using natural stone residues and perlite, 12 types of cement were produced. In order to define cement types, codes of each cement were determined. The codes of the cement types are shown in Table 2.

Table 2. The mixtures properties used in the tests and codes.

Portland Cement	PC
% 5 Natural Stone Residue	NSR-5
% 10 Natural Stone Residue	NSR-10
% 15 Natural Stone Residue	NSR-15
% 20 Natural Stone Residue	NSR-20
% 5 Natural Perlite	NP-5
% 10 Natural Perlite	NP-10
% 15 Natural Perlite	NP-15
% 20 Natural Perlite	NP-20
% 5 Natural Stone Residue, % 5 Natural Perlite	NSRNP-5

% 10 Natural Stone Residue, % 10 Natural Perlite	NSRNP-10
% 15 Natural Stone Residue, % 15 Natural Perlite	NSRNP-15
% 20 Natural Stone Residue, % 20 Natural Perlite	NSRNP-20

2.2. Method
Experimental program of the study was divided into three main parts:

i) Chemical analysis of the three types of main cements;
Using XRF method, chemical ingredients of clinker, gypsum, and natural perlite were chemically analyzed.

ii) Physical analysis of the three types of cements;
Fineness (Both Blaine and 45µ wet), specific gravity, particle size distribution, normal consistency, setting time, loss on ıgnition, heat of hydration, ınitial and final setting times

iii) Mechanical tests of the three cements;
2, 7, 28 days compressive strengths of the cement mortars (Table 3.).

Table 3. Tests performed on Cements.

Test	Test Type
Chemical Composition	X-Ray Fluorescence
Fineness by Blaine Apparatus	ASTM C204
Fineness by 45µ Sieve	ASTM C430
Normal Consistency	ASTM C187
Setting Time	ASTM C191
Compressive Strength	ASTM C109
Strength Activity	ASTM C311

3. TEST RESULTS AND DISCUSSIONS
In this study, natural perlite and marble travertine mixture residues were blended separately and together and cements were produced using additives up to 40% in total and their physical and mechanical properties were investigated. The following results were obtained in the continuation of the studies for this article. The results were primarily compared with tables and graphs and the results were discussed.

3.1. Raw Material Mineralogical Analysis
SEM micrograph of perlite and its magnified view (Fig. 1a, 1b and 1c) revealed the irregular morphology of perlite particles with broken or ragged edges. Similar pattern was observed in other reported micrographs of perlite.

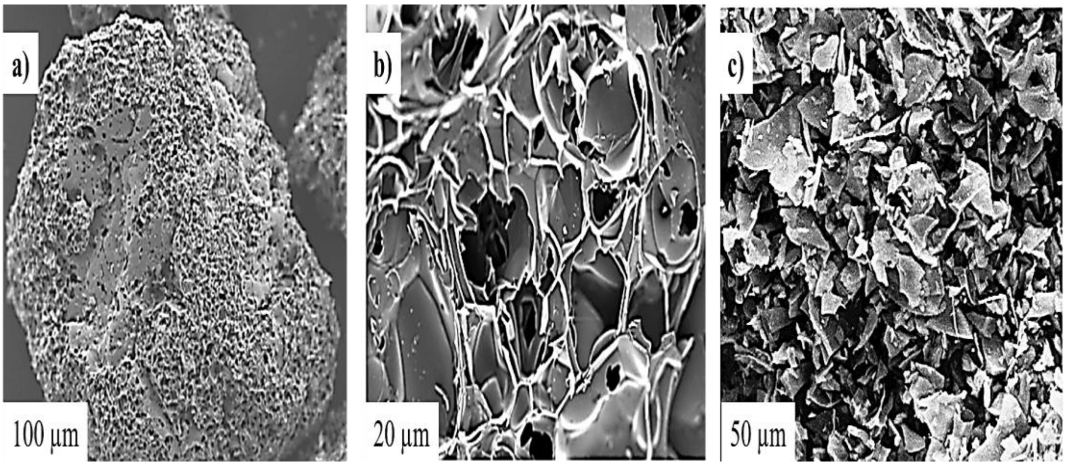

Figure 1. Figure 1a. SEM image of the outer surface of raw perlite; Fig. 1b. SEM image of the inner structure of raw perlite [15]; Fig. 1c. SEM image of expanded perlite.

The low density of perlite raw material samples is due to the wide porosity between 20 µm and 100 µm, as can be seen in the SEM analysis images in Figure 1. Moreover, expanded perlite, which is used in many fields industrially, has a considerably high porosity. Crystal-like, high-porous and glassy structures are clearly visible in all three SEM images.

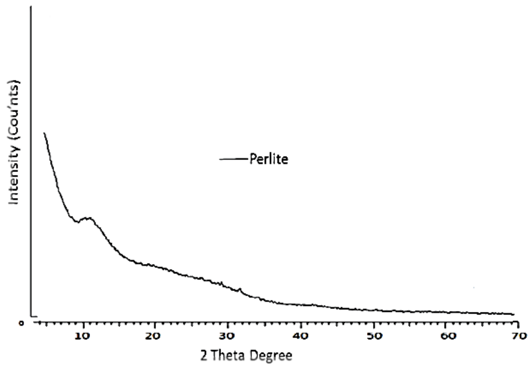

Figure 2. XRD profile of the natural perlite.

Silicate glass constitutes the main structure of the perlite sample. Perlite material can be characterized as rhyolite glass consisting of biotite, quartz and feldspar minerals. The most abundant element in its structure is Si (75% SiO_2 by mass) followed by Al_2O_3 and then alkaline metal oxides. More than 90% of its mineral composition is amorphous. It is seen in apatite, zircon, hornblende and ilmenite even though it is minor in its structure (Fig 2).

A large part (90-97%) of the volume of Perlite is glass and also contains crystallizing minerals such as feldspar and biotite. Some magnetite and apatite can be seen. The paste made of volcanic glass contains microlites and phenocrystals. Despite the glassy structure of perlite, it is thin from its onion peel-looking cracks somewhat dispersed zeolite crystallization, i.e. morderite formation, has been identified. Perlite has an average refractive index value of 1,497. Perlite glass has an isotropic appearance when viewed in polarized light, but sometimes a light pair it may indicate fracture. X-ray analysis (XRD) showed that perlite contains up to 4% free silica [16].

Table 4. Chemical and physical analysis result of raw material samples.

Component	Clinker	Gypsum	Perlite	Marble travertine residues
	%	%	%	%
SiO_2	20,83	0,7	70,96	10,91
Al_2O_3	5,24	0,32	12,61	1,8
Fe_2O_3	3,77	0,13	3,72	1,05
CaO	66,83	33,71	1,8	47,31
MgO	1,2	0,78	0,25	1,04
Na_2O	0,39	0	1,22	0,18
K_2O	0,84	0,03	3,77	6,2
SO_3	0,61	41,58	0,25	0,6
LOI	0,28	22,39	4,61	30,64
Total	99,99	99,64	99,19	99,73

3.2. Specific Gravity, Fineness, and Particle Size Distribution of Cements

It is known that the specific gravity of the pozzolanic cements with perlite added decreases the specific gravity of the cement as the amount of perlite increases. The biggest reason for this is that perlite has a very porous structure and thus its specific weight is low. It is stable as a result of previous studies that the density of the pozzolanic cements with perlite added produced will decrease, and the density will increase with the addition of limestone. Therefore, no density tests have been performed.

Fineness is an important criterion for increasing the reaction speed and capability of the cement. However, increasing the fineness of the cement is a costly task to increase the reaction rate, so it is always necessary to work with optimum fineness in order not to increase the production costs and energy consumption. The cement samples with additives prepared in line with the industrial experience obtained from previous years were grinded in a laboratory type ball mill for 45 minutes. Fineness and blaine tests were performed for each cement sample with additives. 45µ, 90 µm and 200 µm fineness values were examined for each sample. grinding characteristics emerge better.

Travertine and marble residues are from the limestone and calcite family and they are very similar to each other mineralogically. They have revealed that harder, calcite and marble are hard to grind as a result of both hard grove and bond tests [17]. Perlite, on the other hand, resembles shaving due to its high silica content and glassy structure. Scientific studies conducted again revealed that the shade is easily grinded compared to clinker and limestone [18]. Therefore, it can be easily interpreted that perlite-added cements will reduce the grinding energy and decrease the production costs as the contribution rate increases. This study supports this.

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Namely,

To compare the fineness of cement samples in the same time frame;
cements produced with marble and travertine residues;
NSR20>NSR15>NSR10>NSR5
cements produced with natural perlite;
NP20>NP15>NP10>NP5
cements produced with marble and travertine residues and natural perlite mix
NSRNP20>NSRNP15>NSRNP10>NSRNP5
Final; NSR>NSRNP>NP screen sizes can be listed from high to low.

For the fineness values are also given in Table 5.

Table 5. Physical analysis result table of cement samples.

Cement Type	Fineness %			Blaine Fineness (cm^2/gr)
	45 µm	90 µm	200 µm	
NSR-5	9,2	0,8	0	3436
NSR-10	11,9	0,9	0,1	3579
NSR-15	13,4	1,2	0,2	3723
NSR-20	14,1	1,6	0,2	3864
NP-5	5,7	0,4	0	4174
NP-10	6,0	0,4	0	4437
NP-15	7,4	0,5	0,1	4383
NP-20	8,8	0,7	0,1	4520
NSR-5, NP-5	5,9	0,4	0	4061
NSR-10, NP-10	6,8	0,6	0,1	4165
NSR-15, NP-15	8,7	0,7	0,1	4388
NSR-20, NP -20	9,8	0,8	0,2	4498

As can be followed from Table 5, while pozzolan added with increasing percentages by mass to the cements, the weight of the cements decreased. The aim in this study was increasing the surface area by the clinker decreases, so, the cements tested had 500 cm^2/gr more surface area for every 25% increase in natural perlite mass.

3.4 Normal Consistency, Setting Time, Autoclave Expansion, and Heat of Hydration of Cement Pastes

Pozzolanic materials such as zeolite, pumice, trass used in place of cement increase the amount of binder paste since they have a smaller grain size. Because they are very fine grained, they prevent sweating by holding the water in fresh concrete. They also make a positive contribution to the workability of concrete. As they are very fine grained, they cause the gaps in the concrete to decrease and increase the water impermeability. It is preferred for pouring concrete in hot weather. Its workability is better than Portland cement, it has long-lasting strength gains thanks to the pozzolanic materials it contains, and it has high durability. Pozzolanic materials reduce the need for water formed in concrete. The fine filling materials it contains tightens the micro pores in the concrete and increases its impermeability. It increases the quantity obtained thanks to its low density in the construction chemical production and contributes to the processability in ready-mixed concrete manufacturing. It provides long-term durability with its pozzolanic feature. It is possible to achieve all these properties because the natural perlite material also has pozzolanic properties.

So far, scientific studies have shown that the water needs of pozzolanic cements increase with the increase in additive rates compared to Portland Cements, and the lengthening of the setting times. Erdem et al. [2] found that with the increase in the amount of contributions, the need for water and setting time increased. In addition, it has been proven that perlite species are not effective in improving cement properties, especially in setting times and water requirements. Moreover, in parallel with the values found in the study by Erdem et al. [1], it was observed that the plug-in times and water requirement increased with the increase in the contribution rate and thus had a positive effect on the workability.

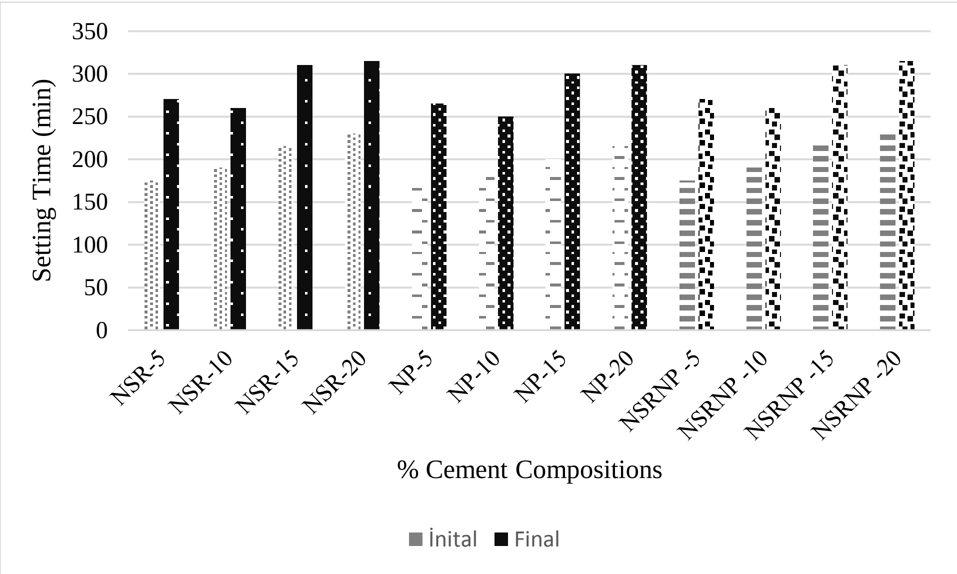

Figure 3. Setting time of the cement samples.

According to TS-EN 196-3 in the standards for pozzolanic cements, the starting time of the setting time must be > 75 minutes. Furthermore, according to TSE EN 196-3, the volume expansion amount of concrete must be < 10 mm. Looking at Fig.3, it is seen that the cement samples obtained from the mixture of perlite, marble travertine residues and both materials (perlite and marble travertine residues) are below the standard values and meet the requirement. Especially, the setting times and workability of perlite-added cements are more prolonged compared to marble travertine-added cements and mixture cements setting time.

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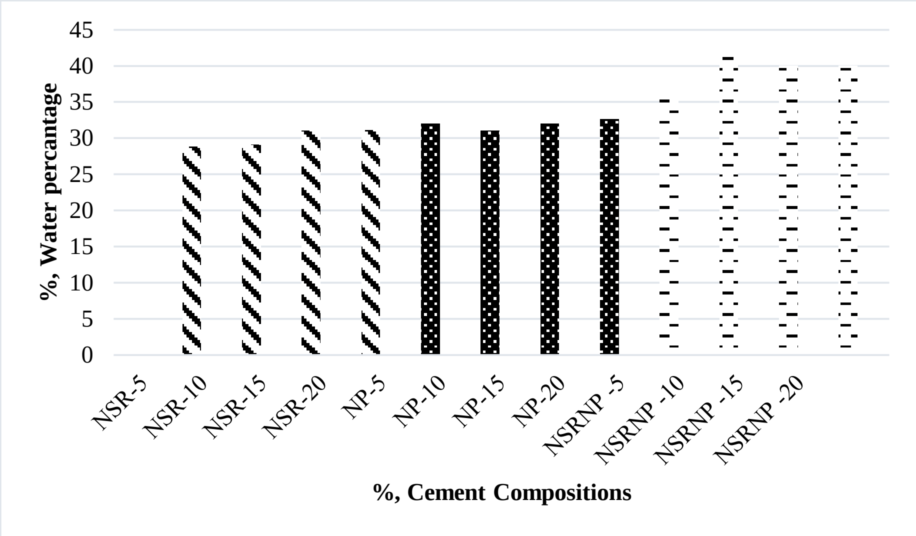

Figure 4. Water normal consistency of the cement samples.

Darweesh and Samhan [19] reported the longer initial and final outlet time of pastes by partially substituting the cement with perlite powder (PP) (63 um) at levels of 5%, 15% and 25% by weight. As the PP content increased, the setting time increased. Initial and final setting time increased by about 0.93% and 4.46%, including 15% and 25% PP, respectively. Virtue et al. [20, 21], Partly applied to 20% and 30%, EP (370 m by weight / kg) is reported in the long first and last setting of the paste by substituting cement with blaine surface area. With the increased EP content, the plug-in time was extended. Fig. 3-4. summarizes the effect of perlite on the workability and setting time of mortar and concrete.

3.5. Compressive Strength of Cement Mortars

According to TS EN 196-1, two-day compressive strength should be between> 20 MPa and 28-day compressive strength> 42.5 MPa and <62.5 MPa in Portland cement, especially for Cem 1 42.5 R cement. Cement producers prefer Cem IV 32,5 R / B cement in order to reduce the cost and increase the durability in the long term by using more additives. According to TS EN 196-1, up to 55% pozzolanic additive material can be used to produce Cem IV 32,5 R-B pozzolanic cement. However, as the pozzolanic additive rate of the material used increases, Cem I 42,5 R decreases the strength compared to Portland cement. For this reason, according to TS EN 196-1, Cem IV 32,5 R-B pozzolanic cement has a two-day compressive strength> 10 MPa and a twenty-eight-day compressive strength value between> 32.5 MPa and <52.5 MPa.

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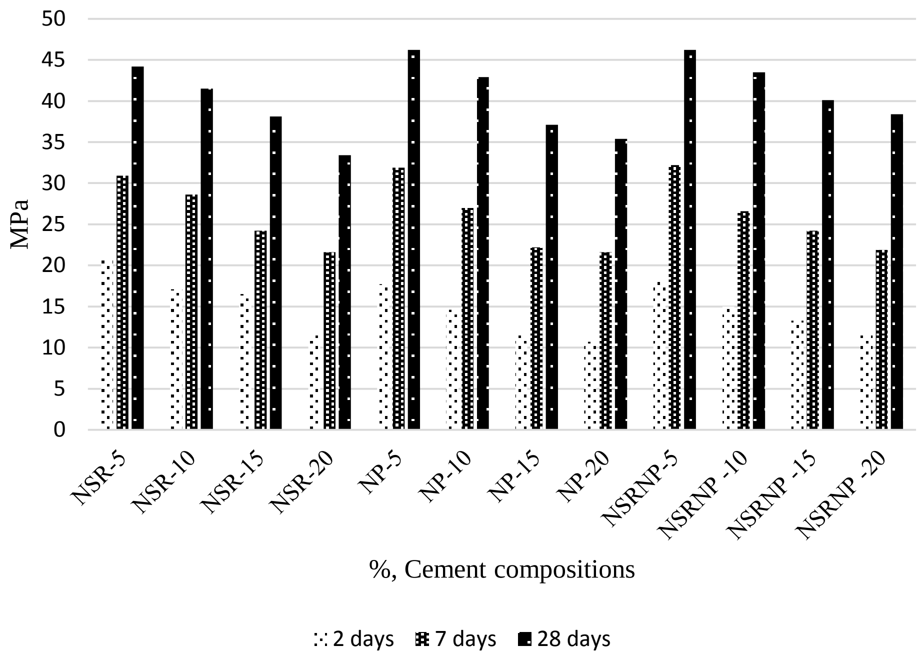

Figure 5. compressive strengths of the cement samples.

When the compressive strength values are examined in Fig. 5, it is observed that the perlite contribution rate increases and the pressure resistance decreases. Then the compressive strength values of the cement produced with mixture cement and then marble travertine residues are followed. When cement samples are compared with TSE EN 197-1 standard, it is seen from Fig. 5. that the strength requirements for Cements are met by all blended cements produced.

In the study conducted by Erdem et al. [1] it is observed that 20% perlite use produces higher strength than 30% perlite use when the strength of mortars containing Cements with the same fineness, same perlite and same grinding method is compared. The low strength of the Cements containing 30% perlite can be explained by the lower PC content (especially when pozzolanic reactions are not effective for early ages) and the more charged water cement ratio. The effect of finer particle size distribution has also been observed on the strengths of mortars containing different types of perlite. The strength values of the more finely ground and more homogeneous perlite-doped Cements were higher. However, due to the ongoing puzolanic reactions of perlite-doped cements, the differences will become smaller in later ages.

Topçu et al. [22] reported a decrease in compressive strength and bending strength of mortars including EP (size 0-2 mm). Lanzón and García-Ruiz [23] reported a reduction in the compressive strength and bending strength of mortars, including the entire composition EP (weight 0.08-1 mm). 28-day reduction in pressure strength, % 0.59, % 1.18, % 1.77, % 3.54 and including 7.08% EP respectively% 29.22, % 38.1, % 49.87, % 68.63 and is 80.97%. daily bending strength respectively% 32.51, % 45.95, % 53.15, % 71.83 and it was 85.5%. Fiat et al. [24] they reported that the inclusion of 5%, 7% and 8.5% EP (size 0-1 mm) in mortars led to a decrease in mechanical strength.

4. CONCLUSIONS

Although there have been very few studies on the use of both natural perlite and expanded perlite in cement production in Turkey, it is known that perlite produced in Turkey has pozzolanic properties as a result of these studies. Turkish perlites generally have amorphous structure and have properties suitable for ASTM C 618.The Nevşehir-Acıgöl perlites used in this study also have amorphous structure and contain pozzolanic properties suitable for evaluation in cement production.

The use of perlite and marble travertine residues in cement production both separately and together increased the starting and finishing time of the cement outlet and the workability of the concrete. However, all these changes remained below the limit values according to TS EN 196-3 and met the requirements.

The strength values of the cement samples produced are low compared to Portland cement. Moreover, this is an expected result according to the literature. However, according to TS EN 197-1, cement samples meet the requirements of pozzolanic cement and composite cement concrete strength classes. That is to say;

Portland calcareous cement		Pozzolanic cement		Composite cement
Cem II/A-L	Cem II/A-LL	Cem IV/A	Cem IV/B	Cem V/A
NSR-5	NSR-5	NP-5	NSR20, NP20	NP-5
NSR-10	NSR-10	NP-10		NP-10
NSR-15	NSR-15	NP-15		NP-15
NSR-20	NSR-20	NP-20		NP-20
		NSR5, NP5		
		NSR10, NP10		
		NSR15, NP15		

As perlite is more easily milled than trass clinker and limestone, it reduces cement production costs and saves energy. It also provides thermal insulation thanks to its superior properties, it is resistant against fire, it can be used as a light building element due to its low density. Because of these reasons, more studies should be done about perlite.

The use of natural stone residues in addition to prolonging the life of Natural Resources and preventing the environmental pollution created by giant natural stone residues are ensured. In addition, since Portland cement will be substituted, cement production costs will be reduced again.

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