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CHEMICAL CHARACTERIZATION OF PATNOS SCORIA (AĞRI, TURKEY) AND ITS USABILITY FOR PRODUCTION OF BLENDED CEMENT

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Abstract. This paper reports results of investigations on suitability of scoria (PTS), collected from Patnos (Ağrı) in Turkey, for blended cement production. Scoria (basic pumice) was chosen as cement replacement materials due to its availability and cost in Ağrı in Turkey. The portland cement was replaced by scoria within the range of 0, 5, 10, 20, 30, 40 and 50%. Characterization of scoria was subjected by the X-ray fluorescence (XRF), X-ray diffraction (XRD), Fourier Transform Infrared (FTIR) spectroscopy, Scanning Electron Microscopy (SEM), BET surface area and porosity, zeta potential (ζ) and thin sections. The standard tests were conducted for the obtained fresh and hardened states of scoria blended cement paste. Furthermore, the obtained cements were characterized by the XRF. According to experimental results, scoria up to 20% ratio could be added into clinker and it has a good potential of manufacturing blended scoria cement.

keywords: scoria, blended cement, Patnos

1. Introduction

Cement industry utilizes natural pozzolans as substitutes for portland cement due to environmental (CO_2 emission reduction), economical (cost reduction) and chemical (reduction of alkali aggregate reaction and increased chemical resistance) advantageous (Fu et al., 2003; Sersale 1987; Saraswathy et al., 2003;Vuk et al., 2002; Binici and Aksoğan 2006; Yilmaz, 2009).

Pumice is one of the natural pozzolan and is a volcanic origin rock formed during explosive eruptions. It has a highly porous structure which is formed by dissolved gases precipitated during cooling as lava hurtles through the air. Generally, it has not a crystalline structure and SiO_2, Al_2O_3 and Fe_2O_3 constitute major contents of pumice (Gündüz et al., 1998; Lura et al., 2004; Ersoy, 2010).

Turkey has important potential pumices reserves (68% of the reserve of the world). Although pumice has been used in the world industry for a long time, it has been evaluated and its importance has been recognized by Turkish industry for the last 30 years. Fifty six per cent of pumice reserves (acidic pumice and scoria) in Turkey occupies a large surface area in the East Anatolia Region depending on the recent volcanic activities (T.R. Report, 2001). Therefore, it is important to assess pumice reserve in Eastern Turkey for its use in the cement industry.

There are two aims of the current research. One is to characterize scoria from Patnos (Ağrı, Turkey). A literature survey showed that no academic studies have investigated this scoria for its use in the construction industry. Therefore, the second purpose of this study is to determine the potential use of scoria from Patnos in the cement industry as a replacement material.

2. Materials and methods

2.1. Sample preparation

More than 50 kg of scoria sample was collected from scoria deposit in Patnos (Turkey). In order to reduce the amount of sample, sampling was performed using the cone and quartering method and riffles, since sampling must have mineralogical, physical and chemical homogeneity. The sample was crushed and ground using a laboratory dodge jaw crusher, rod mill and ball mill to reduce their size to 200 mesh (74 μm) for mineralogical and chemical analyses. Five hand samples were prepared for thin section studies. Cutting, polishing and thinning processes were performed using an oil system. Glue that hardened under UV light was used.

2.2. Characterization of scoria samples

Chemical analysis of the scoria sample was carried out using the X-ray fluorescence (XRF Spectro IQ) technique.

The composition of the scoria sample was checked by the X-ray Powder Diffraction. By comparing positions of diffraction peaks against those of the ICDD cards, the target material could be identified. The XRD data were collected using the Rigaku X-ray Diffractometer (Model RadB-DMAX II) with Cu K_α (30 kV, 15 mA, λ=0.154051 nm) radiation at room temperature. Scanning was done between $5^o<2\theta<70^o$. The measurements were made with 0.01 and 0.05 degree steps and 1 degree/minute rate. The divergence slit was variable. The scattering and receiving slit were 4.2 degree and 0.3 mm.

As a further characterization method, the FTIR analysis was carried out in order to investigate functional groups of scoria. The IR spectrum of material was measured in the range of 400 to 4000 cm^{-1} by the KBr pellet method using the Perkin Elmer Spectrum One device. IR pellet was prepared using spectroscopic grade KBr with a sample (KBr-to-sample ratio of 100 mg : 3 mg). KBr was dried at 180°C for 12 hours before the preparation of pellet.

Surface area and porosity values of the scoria sample were determined using a Tri Star 3000 (Micromeritics Instrument Co. USA) surface analyzer which was used to measure nitrogen adsorption isotherm at 77 K in the range of relative pressures from 10^{-6} to 1. Before measurement, the sample was degassed at 400ºC for 2 h.

The zeta potential of the scoria sample was measured by a Zeta Meter 3.0 (Malvern Instruments Ltd.) equipped with a microprocessor unit. The zeta potential was calculated automatically using the Smoluchowski equation and as a function of pH of the solution according to the electrophoresis method with high sensitivity. A sample of 0.5 g was taken from each pumice sample and then transferred into a glass beaker and 100 cm^3 of aqueous solution was added. The mixture was stirred using a magnetic shaker and the pH of the test solution was adjusted to the desired value by drop-wise addition of diluted NaOH (0.5%) or HCl (0.1 N). After stirring the solution, the suspension was stored to let larger particles to settle.

In order to investigate the morphology of the pumice sample, the Leo EVO 40 scanning electron microscope, which does not need palletizing, was used. SEM images were obtained from the scoria samples in powder form.

To investigate petrographical characteristics of the scoria sample, thin sections of 5 rock samples were prepared and determined using the LEICA Polorizan Microscope.

In order to measure the true densities of the scoria sample and cement, pycnometer was used.

2.3 Preparation of test specimens

A reference cement (ordinary portland cement, OPC) was produced by mixing portland cement clinker, 96% in weight, and gypsum, 4% in weight. This mixture was then ground for 40 minutes in a laboratory-type ball mill. Scoria blended cement samples (PBC) were obtained using 5%, 10%, 20%, 30%, 40% and 50% (by weight of clinker) pumice replacement by mixing and inter-grinding. The gypsum content was kept constant in all cements as 4%. Before grinding operation, portland cement clinker, scoria and gypsum were crushed, and sieved through a 9.5 mm sieve. The purpose of sieving was to keep the uniformity between each specimen through using the same feed sizes. Gypsum was dried at 40°C prior to crushing whereas the natural pozzolans were dried at 110°C.

2.4. Tests conducted on the scoria blended cements

The chemical compositions of the control specimen and scoria blended cements were performed by the X-ray spectrometer (XRF). Physical analyses were performed in accordance with TS EN 196-6. Fineness of the scoria blended cement samples was determined by measuring the Blaine fineness and amount of material retained on 45, 90, and 200 µm sieves after vacuum sieving.

Following tests were carried out on the scoria blended and control cements: fineness, specific surface area by Blaine instrument, normal consistency, setting time, soundness by the Le Chatelier method and compressive strength. The amount of water necessary for the cements to have normal consistency was determined according to TS EN 196-3. Then, the pastes having normal consistency was used to determine the setting time and soundness through conducting tests as described in this standard. Compressive strength and flow values of the mortars were determined according to TS EN 196-1. Preparation of cement mortar mixtures was completed according to TS EN 196-1. In these tests, 450 ±2 g of cement and 1350 ±5 g of standard sand were used. PBC mortars were prepared with 225 cm^3 of water whereas the water content of the blended cement mortars were adjusted to have a w/c ratio of 0.5 as stated in the standard. The prepared mortars were poured into rectangular-prism-shaped three-part mortar molds 40x40x160 mm and compressive strength tests were performed by an automated strength testing instrument in accordance with TS EN 196-1. The compressive strength of the mortars was determined after 1, 2, 7 and 28 days. Three cube specimens were tested for each day.

3. Results and discussion

3.1. Mineralogical and chemical characterization of scoria

3.1.1. X-ray fluorescence (XRF) and geochemistry

The result of chemical analysis of the scoria sample is given in Table 1. Chemical analysis indicates that SiO_2, Al_2O_3, Fe_2O_3 and CaO constitute major contents of the pumice samples. As it is known, pumice is an amorphous porous volcanic rock, which is composed mainly of SiO_2. Pumices show acidic and basic properties and are named acidic pumice and scoria (basic pumice), depending on the SiO_2 content. According to the XRF results, the pumice sample used in the present study can be classified as scoria (basic pumice) due to the relatively low SiO_2 and high Fe_2O_3 contents (Gündüz et al., 1998).

According to TS 25, natural pozzolans should have specific chemical properties to be used in the cement industry. These requirements and comparison with scoria (PTS) are given in Table 2. The results in Table 2 indicate that the scoria samples collected from Ağrı (Turkey) satisfies the TS 25 requirements and therefore can be used in cement industry as a cement additive.

Table 1. Chemical composition of scoria sample

	PTS
SiO_2	54.92
TiO_2	2.55
Al_2O_3	16.92
Fe_2O_3	10.31
MgO	2.01
CaO	6.47
Na_2O	2.17
K_2O	1.87
P_2O_5	0.39
SO_3	0.29
LOI	0.86
Reactive silica	31.52
Puzzolonic activity (kg/cm^2)	100

Table 2. Comparative study of chemical properties according to TS 25 standard

Chemical Properties	TS 25 Standard	PTS
	wt. %	wt. %
$SiO_2 + Al_2O_3 + Fe_2O_3$	> 70.0	81.73
MgO	< 5.0	2.12
SO_3	< 3.0	0.29
Reactive silica	> 25	31.52
Cl	< 0.1	0.009

3.1.2. X-ray diffraction (XRD)

The XRD pattern of scoria is given in Fig. 1. As stated before, pumice is an amorphous volcanic rock. X-ray diffraction data (Fig. 1) indicates that scoria has mainly amorphous structure (Arrigo et al., 2007; Ersoy et al., 2010), but some little crystalline mineral phases, anorthite (JCPDS Card File No. 73-1435), hornblend (JCPDS Card File No. 71-1062) and crystalline quartz (JCPDS Card File No. 76-0823) are also observed in the XRD pattern.

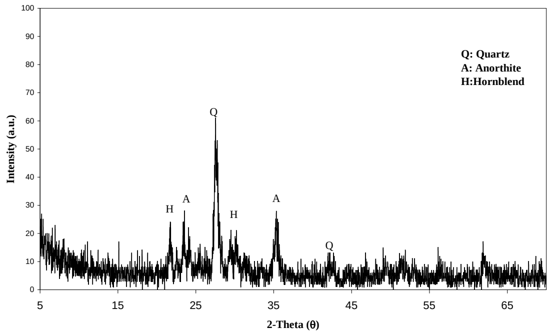


Fig. 1. XRD patterns of scoria

3.1.3. FTIR spectra

The infrared spectrum of scoria is given in Fig. 2. In the IR spectrum, the strong bands observed in the frequency range of 1000–1040 cm^{-1} is characteristic of silica. As seen, this peak is very broad, confirming the highest silica content in the scoria sample. On the other hand, the bands belonging to other metal oxides could be observed in the mentioned range as oxide or Si-O-M form. The broadening of the peak may be attributed to this. This explanation is also compatible with the literature data. In the literature, symmetric vibrations at 1049 cm^{-1} was attributed to Si-O-Al bonds (Perraki and Orfanoundaki, 2004; Blanco et al., 2006). Beside, the band in the 780–790 cm^{-1} region is attributed to Si–O bending strength vibrations of amorphous quartz. The band observed around 3000 cm^{-1} is characteristic for water (OH stretching vibrations). According to the FTIR bands, it could be seen that the main structure of the scoria samples is amorphous silica confirmed by the XRF and XRD results. Amorphous silica reacts with $Ca(OH)_2$ and forms cementitious materials, which are important for concrete in terms of durability and the rate of gaining strength (Hossain, 2005).

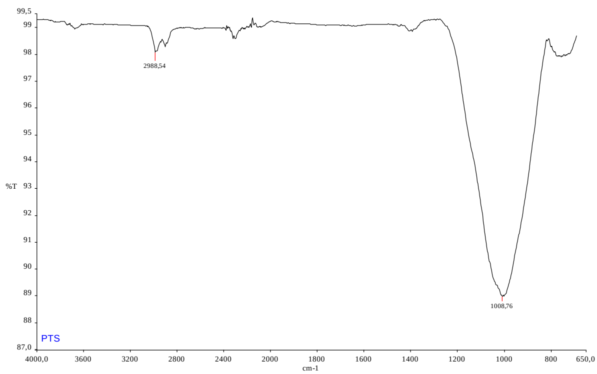


Fig. 2. FTIR spectrum of scoria

3.1.4. Electro-kinetic property

ζ potential variation of scoria is given in Fig. 3. The zeta potential increases in the negative direction with increasing the pH. The isoelectrical point (IEP), which represents no net electrical charge of surface at the specific pH, is not observed for scoria. A literature survey also shows that the same result was observed for pumices by Tunc and Duman (2009) and Ersoy et al. (2010).

According to the XRF, XRD and FTIR results, the scoria sample is composed mainly of SiO_2. Therefore, the zeta potential of quartz was also measured and given in Figure 3. The IEP, like literature values (Huang and Fuerstenau, 2001; Prasanphan and Nuntiya, 2006) for quartz was at pH 2. For scoria and quartz, the negative zeta potential can be attributed to surface charge on a solid which may originate from ion adsorption, surface dissociation and isomorphic replacement of ions of the solid phase by others of a different charge.

Yılmaz (2009) mentioned that clinker has positive zeta potential values depending on the Ca^{2+} ions in crystal structure. Figure 3 indicates that the zeta potential of scoria is negative. As is known, electrically different charges attract each other. Thus, PTS and clinker pull each other and their particles easily come together.

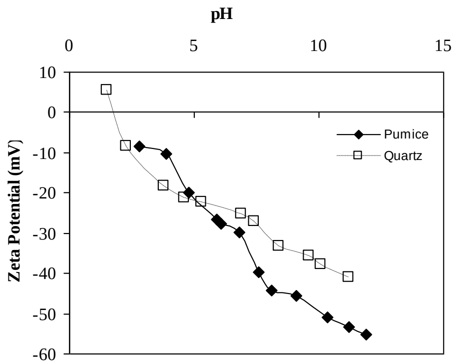


Fig. 3. Zeta potential vs. pH of the scoria sample

3.1.5. BET surface area and pores volume and size of tested pumice samples

Surface areas and porosity of pumice arer given in Table 3. It can be seen that the basic pumice sample (> 200 mesh) has not a big surface area and micro- and mezzo-pore structure.

Table 3. Surface area and porosity value

Pumice Samples	S_{BET} (m^2/g)	S_{ext} (m^2/g)	S_{mic} (m^2/g)	S_{mezo} (m^2/g)	V_t (cm^3/g)	V_{mic} (cm^3/g)	V_{meso} (cm^3/g)	D_p (nm)
PTS	0.67	0.22	0.45	0.22	0.003	0.0003	0.0027	15.41

D_p:4V/A by BET, S_{ext}=S_{meso} + S_{macro}

3.1.6. SEM images

The SEM images of scoria samples are given in Fig. 4. They indicate that the scoria sample have irregular morphology, non uniform plate shape and glassy form (amorphous structure).

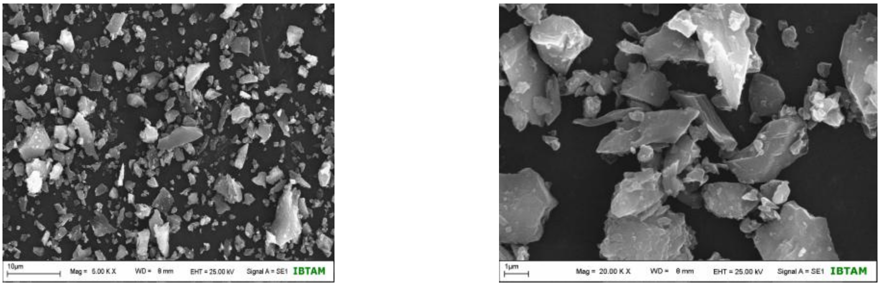
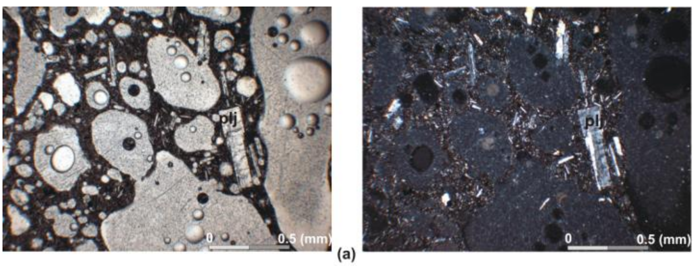


Fig. 4. SEM images of the scoria samples at different magnifications

3.1.7. Thin section

The thin sections, which were randomly selected from the images, are given in Fig. 5. The thin sections studies reveal that the scoria sample has mainly hyalopilitic-porphyritic texture, and from place to place, vitrophyric-porphyritic texture is also seen. Scoria is mainly composed of volcanic glass and there are phenocrystals of plagioclase, pyroxene and rarely hornblende in the matrix. Plagioclases in the matrix show polysynthetic twins and the minerals are classified as a long rod-shaped (Figs. 5 a–c). Pyroxene is easily distinguished with live color tone (Fig. 5 b) and lots of gas gaps are observed in the matrix (Fig. 5).

A literature survey shows that pozzolan can be considered as a good component for the cement industry, if it contains high amounts of zeolite minerals and volcanic glass (Planungs Report, 1984). The thin section studies show that scoria is mainly composed of volcanic glass, so the Patnos scoria pumice is a good pozzolan for the cement industry.



(a)

Fig. 5. Thin sections of the scoria sample: (a) plagioclase minerals and gas gaps in volcanic glass matrix. (b) pyroxene minerals seen in volcanic glass and plagioclase, (c) phenocrysts of plagioclase, pyroxene and gas gaps

(continued)

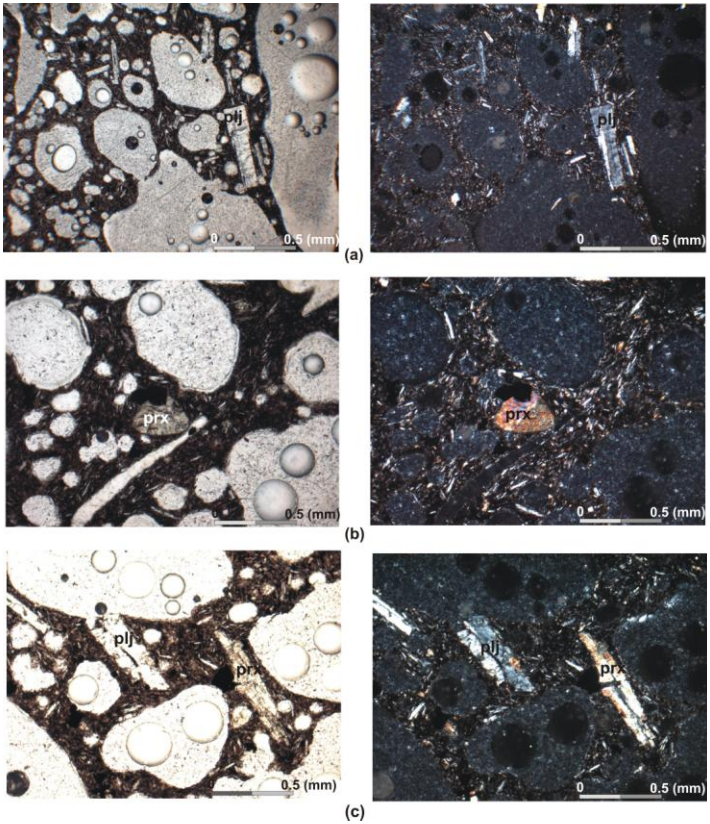


Fig. 5. Continued

3.2. Chemical and physical properties of scoria blended cement

Chemical analyses of the scoria blended cements and control specimen are given in Table 4. The chemical composition of the portland cement indicates that the CaO/SiO_2 ratio is greater than 2 and the percent of MgO content is smaller than 5%. These values satisfy the main composition requirements imposed by the TS EN 197– 1 standard. Depending on the scoria content, SiO_2, Al_2O_3 and Fe_2O_3 contents increase and CaO content decreases with increasing the pumice ratio in the cement mixture.

Physical and mechanical properties of control (OPC) and the scoria pumice blended cements are given in Table 5.

The scoria blended cement mortars to be used for compressive strength testing were prepared to have a w/sbc of 0.50 as stated in TS EN 196-1. Table 5 shows the compressive strength values after 1, 2, 7, and 28 days. The trends, observed as variation of comparative strength with the percentage of scoria, indicate a decrease with the increase of scoria content in cement as expected. The literature survey shows that the comparative strength decreases, depending on the pumice content due to the reduction of clinker content in the cement mixture (Yazıcıoğlu and Demirel 2006; Mehta, 1981; Hossain, 2003; TCMB research 2003). In TS EN 197 – 1, early strength (2 days) values should be greater than 10 MPa and standard strength (28 days) values should be between 42.5 MPa and 62.5 MPa. When the obtained results are compared to the standard, it can be seen that the strength requirements were satisfied by the blended cements up to 20 % scoria content.

Table 4. Chemical composition of control (OPC) and scoria blended cements

Oxides	PTS						
	OPC	5%	10%	20%	30%	40%	50%
SiO_2	21.31	22.76	24.42	27.75	29.07	34.41	37.73
Al_2O_3	5.37	5.88	6.45	7.59	8.01	9.87	11.01
Fe_2O_3	3.74	4.39	4.75	5.46	5.50	6.88	7.59
MgO	2.54	2.49	2.46	2.41	2.13	2.30	2.25
CaO	60.52	58.11	55.39	49.96	46.92	39.09	33.66
Na_2O	0.56	0.65	0.74	0.90	0.85	1.23	1.39
K_2O	0.82	0.85	0.90	1.00	1.07	1.20	1.30
SO_3	2.08	1.98	1.89	1.71	1.89	1.36	1.18
HM	1.99	1.74	1.54	1.21	1.10	0.76	0.59
SM	2.34	2.27	2.24	2.18	2.15	2.11	2.08
AM	1.43	1.45	1.47	1.51	1.46	1.56	1.57
LSF	1.99	77.16	68.38	54.07	49.69	33.94	26.59

Comparing the setting time of the portland and blended cements, the portland cement and cements including 5 % and 10 % PTS have close setting time and setting time increases with increasing the amount of PTS. This result is very compatible with literature. In the literature, it is mentioned that the setting time considerably increases by the addition of greater natural pozzolan amounts (30–70% in mass) (Öner et al., 2003; Yetgin and Çavdar 2006; TS 25; Targan et al., 2003). The variation of setting time can be explain by reduction of the cement content in the mixture and a low specific surface area. As seen in Table 4, the values for blended cements are smaller than for the control cement. The low specific surface area and clinker content may decrease the rate of the hydration process which increases the setting time. The result is in a good agreement with the literature (Aydin and Gül, 2007).

Table 5. Physical and mechanical properties of control and the scoria blended cements

		PTS						
		5%	10%	20%	30%	40%	50%	OPC
Compressive Strength (MPa)	1 day	10.58	9.21	8.33	7.74	6.66	5.19	13.13
	2 days	18.62	17.64	15.48	13.13	13.72	11.17	26.46
	7 days	40.67	40.18	39.00	26.66	24.30	19.60	45.57
	28 days	50.18	49.00	43.71	36.26	33.32	27.44	55.66
Range Dimension	200 µm	0.5	0.6	0.4	0.6	0.7	0.2	0.2
	90 µm	1.5	1.7	1.1	1.8	1.6	1	3.2
	45 µm	12.4	13.1	13	14.6	12.7	11.4	12.6
Blain, cm^2/gr		3897	3793	3740	3525	3620	3694	4011
Setting Time (min.)	Initial	115	120	150	170	165	230	100
	Final	160	160	200	220	190	285	140
Water demand, %		26	27.4	27	27.4	27.4	27.9	26.3
Density, g/cm^3		3.08	3.06	3.02	2.98	2.97	2.94	3.13
Soundness, mm		3	3	3	3	2	2	4

The OPC and PTS blended cements (for all scoria content) have nearly the same water demand ratio. A literature survey shows that the chemical structure, porosity and specific surface area of cement mixtures affect water demand (Lea, 1976). In the present study, a big difference between water demand ratios was not observed. This may be explained by the BET results. The BET data show that scoria (>200 mesh) has not a big surface area and big porosity values. Because of this, the water content does not change considerably.

As seen in Table 5, soundness of the portland and blended cements are different. Scoria addition reduces the volume expansion since the total CaO (see XRF results of blended cement) and free CaO amounts depend on amount of pozzolan. Çavdar and Yetkin (2009) mentioned that, as a result of CaO, the reaction temperature and rate decrease and the material crystallizes perfectly. Therefore, reduction of the volume expansion of scoria blended cements can be explained by reduction of CaO amount in the scoria blended cement structure.

4. Conclusion

In the light of the experimental results, the following conclusions were obtained.

1. The scoria sample (PTS) is mainly composed of amorphous silica, Al_2O_3 and Fe_2O_3.
2. PTS has mainly hyalopilitic-porphyritic texture and from place to place, vitrophyric-porphyritic texture. Scoria is mainly composed of volcanic glass and there are phenocrysts of plagioclase, pyroxene and rarely hornblende in the matrix.
3. PTS possess sufficient pozzolanic characteristics to be used as an additive during cement production, since it satisfies the standard requirements.
4. Tests conducted on the scoria blended cements suggest the manufacture of the blended scoria cement with a maximum replacement of 20%.
5. Finally, it can be said that use of scoria found in Patnos (Ağrı, Turkey) can be beneficially used for cement production and this will help to reduce clinker consumption.

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