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Use of tuffs from central Turkey as admixture in pozzolanic cements Assessment of their petrographical properties

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In memory of Prof. Dr. Ayla Tankut

Abstract

Tuffs from Galatean Volcanic Province were studied for their use as admixtures in pozzolanic cements. The effects of petrographical properties on the pozzolanic activity of mortar specimens were investigated by optical microscopy, X-ray powder diffraction (XRD), scanning electron microscope equipped with an energy dispersive X-ray system (SEM–EDX), and chemical analysis. The chemical compositions of tuffs conform well to the requirements of ASTM C 618 and the Turkish Standard TS 25, and $SiO_2 + Al_2O_3$ + total Fe_2O_3 exceeds 70%. Pozzolanic activities were determined according to their 7th day flexural and compressive strengths and vary between 1.7 and 3.0 MPa and 7.4 and 16.0 MPa, respectively. The mechanical strength of mortars is affected by alteration of tuffs used in the mixture. Clay minerals and zeolites form by the alteration of volcanic glass, which is the most reactive phase and has a reducing effect on mechanical strength. The alteration also causes the enrichment of tuffs with respect to $K_2O + Na_2O$. The methods used provided rapid evaluation of tuffs as potential admixtures in cements.

Keywords: Admixture; Pozzolan; Petrography; SEM; Mechanical properties

1. Introduction

Volcanic tuffs, which are a variety of pyroclastic rocks, are commonly used in the cement industry as an admixture for the production of pozzolanic cements. They are natural pozzolans rich in siliceous or siliceous and aluminous components that in itself possess little or no cementitious value. On the other hand, in finely divided form and in the presence of moisture, they chemically react with the calcium hydroxide released during the hydration of Portland cement at ordinary temperatures to form compounds possessing cementitious properties. A good pozzolan should possess a sufficient amount of reactivity that is expressed as "pozzolanic activity." The origin of pozzolanic activity lies in the high content of reactive silica in pozzolans [1]. The reactivity of silicate minerals in saturated calcium hydroxide solution having pH = 12.6 increases with the degree of disorder in their crystalline structure [2]. So the nature and amounts of siliceous and aluminous materials in tuffs have important effects on their pozzolanic activities. Tuffs can include combinations of various silicate minerals such as quartz, feldspar, mica, hornblende, pyroxene, zeolite, cristobalite, clay minerals, amorphous pumice, and glass shards. In general, a good pozzolanic tuff has low quantities of clay minerals, low quantities of alkali feldspar, high quantities of zeolite minerals, and volcanic glass. In addition, it should exhibit high porosity and specific surface area [3].

Tuffs, which are used as admixtures in cement, are very important economically. They are cheap raw materials and its utilization leads to considerable savings in the unit cost of concrete. Since the pozzolanic activity occurs at ordinary temperatures, today they are also used for saving energy by replacing a good proportion of clinker in Portland cements [4]. Turkey is rich in natural pozzolans, which are also called "trass" in the cement industry. Almost 155,000 km^2 of the country is covered by Tertiary- and Quaternary-age volcanic rocks, among which tuffs occupy important volumes. Although there are many geological investigations on these

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volcanics, their potential as natural pozzolans is not well established [5]. In Turkey, like in most Mediterranean countries, tuffs have been used in mixture with lime since historical times. Today, the cement industry in Turkey is one of the well-established and developed industry and it has a permanent interest of new supply sources of tuffs since almost about one-third of the total production in recent years was "trass cement," which is a Portland–pozzolan cement [6,7].

In this study, tuffs from various locations at central Turkey were investigated to understand the relations between their petrographical properties and the pozzolanic activity. It also aims to contribute to the permanent interest of the cement industry in the evaluation of natural pozzolans.

2. Regional geologic setting

Volcanic rocks of Tertiary and Quaternary age are widespread in Turkey and form various volcanic provinces (Fig. 1). A large and continuous Neogene volcanic terrain is situated at the northwestern central region, comprising the Galatean Volcanic Province including Ankara Volcanics. In this terrain, the volcanic activity is characterized by calc-alkaline intermediate to felsic lava and pyroclastic products, and younger alkaline volcanics, mainly of basaltic type, occur as small lava flows [8–10]. The field relations, petrography, and geochemistry of the calc-alkaline volcanics and associated pyroclastics have been described by various studies. Air-fall tuffs from Ankara area range in age from Miocene to Pliocene [11] and their widespread exposures occur in the close vicinity of the city of Ankara, Elmadağ, Sincan, and Çubuk locations (Fig. 1). The Kemeres tuff unit of the pyroclastic rocks from Karaşar area covers large areas [12]. The lacustrine deposits of the intervolcanic Pelitcik basin that is located in the south-central part of the Galatean Volcanic Province include widespread exposures of tuff layers in alternation with claystone and dolomicrite [13]. The easternmost exposures of the Galatean Volcanics occur

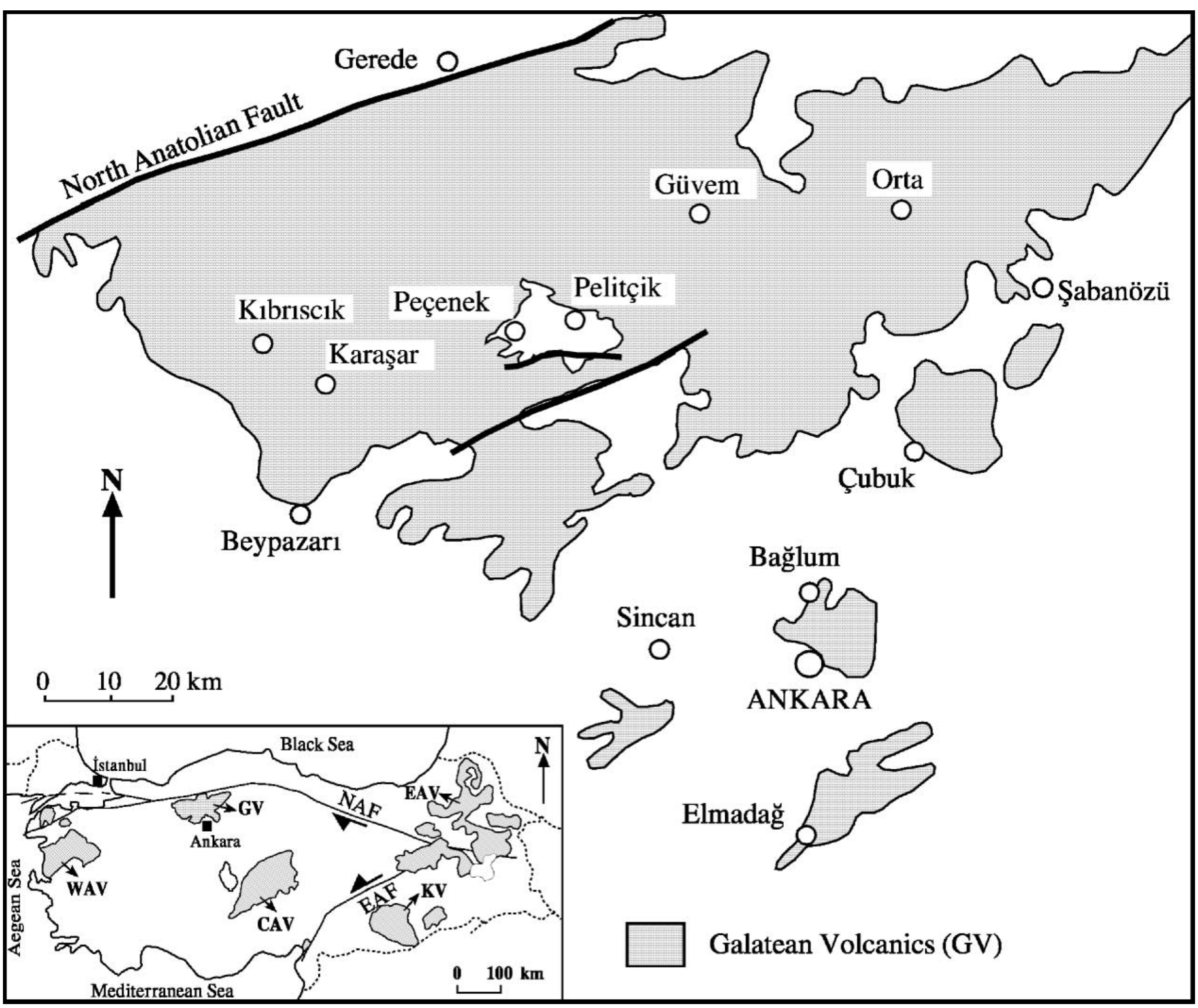

Fig. 1. Regional geological setting of the study area and sampling locations. GV = Galatean Volcanic Province; WAV = Western Anatolian Volcanic Province; CAV = Central Anatolian Volcanic Province; EAV = Eastern Anatolian Volcanic Province; KV = Karacadağ Volcanic Province; NAF = North Anatolian Fault; EAF = East Anatolian Fault.

Table 1
Sample descriptions

Target area	Sample location	Sample number
Elmadağ	Kadınebe Hill	EL1, EL2, EL3
	Çatalhöyük Hill	EL4
	Eşikonartan Hill	EL5
	Dabırga Hill	EL6, EL7
Bağlum	Sivri Hill	B2
	Kaşkaya Hill	B3, B4
	Bağlum Center	B7
Orta	Akçaören Village	OR1A, OR1B
	Bulduk Village	OR2A, OR2B
	Naltepe Hill	OR3B, OR4A, OR4C, OR4D
Peçenek	Sarıkavak Village	PE1, PE2, PE3, PE6
	Yoncatepe Village	PE8
Karaşar	Uşaklıgöl Village	BEY3
	Karaşar	BEY5

near Orta. Late Miocene-age Kepezinkaş formation consists of tuffs and agglomerates alternating with dolomitic limestone layers [14].

3. Methods of study

3.1. Field observations and sampling

Tuffs were sampled along the stratigraphic sections located within the target study areas at Elmadağ, Bağlum, Pelitçik, Karaşar, and Orta located in the Galatean Volcanic Province (Fig. 1). The first two locations represent the Ankara volcanics.

Elmadağ exposures are the products of eruptions from a set of volcanic centers aligned approximately in the northeast–southwest direction. Alternating layers of white- to pink-colored tuff and agglomerate comprise the major succession and form hills usually capped by andesitic and younger (Pliocene) basaltic lava. At some hills the thickness of pyroclastics reaches 250 m. The layers are structurally deformed by faulting and folding. The Neogene volcanics overlay, by an unconformity, the basement rocks of the Triassic Karakaya melange including Permian-age limestone blocks. Ankara volcanics at Bağlum area have similar stratigraphic properties. Tuff layers having white, gray, and pink colors reaching 90-m thickness are overlain by lava of andesitic and basaltic compositions. At the slopes of hillsides, syndepositional faults and folds and sedimentary structures such as grading indicate a source area towards north. The basement rock is the Cretaceous-age ophiolithic melange including the blocks of Jurassic limestone, graywacke, and phyllite.

At the Orta area, a thick succession of Neogene-age andesitic and basaltic lava, epiclastic rocks, and pyroclastics cover large areas. Samples were collected from the andesitic tuffs of Kepezinkeş formation that are well exposed at hills capped unconformably by basaltic lava of Pliocene age. The Kemeres tuff from Karaşar area has dirty white to yellow colors and is andesitic in composition. It is bounded, at top and bottom, by andesitic lava and laterally grades into volcanic breccia. The pyroclastic sequence attains thickness of more than 200 m. The Kemeres tuff is intercalated with thin marl layers. At Pelitçik basin, the Galatean Volcanics are overlain by lacustrine facies that also includes various tuff layers. At the north of Peçenek, the largest outcrops of tuffs are observed between Sarıkavak and Yoncatepe villages. In this area, a sequence of limestone/dolostone/claystone is disrupted with vitric tuff horizons with maximum thickness of approximately 9 m. Table 1 lists the target areas, sampling locations, and sample numbers referred in this study.

3.2. Experimental methods

Mineralogical and petrographical properties of the samples were identified under the polarizing microscope by using their thin sections. X-ray powder diffraction (XRD) analysis of the powdered bulk samples were carried out on a Phillips PW 340 X-ray diffractometer operating at 40 kV and 30 mA using Cu K_{α} radiation. Micromorphological features of tuffs were examined by means of a Leo 435 VP scanning electron microscope (SEM) equipped with an Oxford Model energy dispersive X-ray system (EDX).

Major element analysis of the samples comprising SiO_2, TiO_2, Al_2O_3, total Fe_2O_3, MgO, CaO, Na_2O, K_2O, and SO_3 were carried out by means of wet chemical analysis. SO_3 is determined by using Hitachi 100-60 Model UV–visible spectrometer and the other elements were analyzed by using UNICAM 939 AA spectrometer. Loss on ignition (LOI) is calculated by heating samples at 1000 °C for 1 h.

The pozzolanic activity, the behavior of pozzolan in mortar, was determined according to the Turkish TS 25 [15], TS 24 [16], and the British Standard BS EN 196-1 [17] standards. Pozzolanic activity test procedure is similar to the one described in ASTM C 311 [18] standard. According to TS 25, at least three mortar specimens were prepared by mixing slaked lime, standard sand, and pozzolan (tuff) having the weights shown in Table 2. Immediately after molding, the mortar specimens were covered to prevent evaporation and cured in a moist environment (at 23 ± 2 °C) for 24 h. Then, they were cured at 55 ± 2 °C, to increase the rate of reaction, in an oven for 6 days. After removing from the oven and cooling at room temperature for 4 h, the

Table 2
Proportions of the ingredients of mortar specimens according to TS 25

Material	Weight (in gram)
Slaked lime $Ca(OH)_2$	150
Pozzolan	$2 \times 150 \times N = T$
Standard sand	1350
Water	$0.5 \times (150 + T)$

N = factor obtained by dividing density of tuff by the density of lime.

specimens were tested for their 7th day flexural and compressive strengths according to the Turkish Standard TS 24 [16] by the "Rilem–Cembureau Method." This standard is the same as the British Standard BS EN 196-1 [17].

4. Results

4.1. Petrography

In the tuffs studied, three principle kinds of pyroclasts, namely juvenile fragments, crystals, and lithic fragments are present in varying quantities. The juvenile fragments are observed both as pumice fragments and glass shards that are disseminated in the matrix. Pumice fragments are highly vesicular (Fig. 2a) and shards commonly have cuspate shapes (Fig. 2b) in most of the samples. Blocky shards of brown glass with curviplanar surfaces are characteristic only for the tuffs from Karaşar area (Fig. 2c). The juvenile fragments are abundant in tuffs from Elmadağ, Bağlum, and Peçenek exposures up to 70 wt.%. Various types of mineral crystals are observed in the thin sections and can be detected in XRD patterns (Figs. 2b,d and 3). Plagioclase (Na–Ca feldspar) and quartz are the most common ones. Biotite, hornblende, and clinopyroxene are the other minerals of magmatic origin. The lithic fragments are composed of volcanic rock fragments of andesitic and trachytic compositions (Fig. 2d).

Tuffs exhibit low degree of alteration and clay minerals, cristobalite, zeolite, calcite, and iron oxide are the main authigenic products. Presence of smectite, illite, and kaolinite are detected in the XRD diffractograms (Fig. 3). Smectite is the most widespread clay mineral, whereas kaolinite and illite are present only in the tuffs from Orta and Bağlum areas, respectively. SEM–EDX analysis showed that the smectite is an Fe–Al variety with a typical flaky morphology (Fig. 4a,b). Pumice fragments and glass shards are generally fresh and exhibit microfractures and dissolution pores (Fig. 4c). In their chemical composition, Si, Al, and O are the major elements while Na, Ca, and K are found in minor quantities (Fig. 4d). Authigenic growth of clay minerals and zeolite associated to volcanic glass are recognizable. Zeolite has a fibrous habit and compositionally consists of Si, O, Al, K, and Ca, indicating erionite

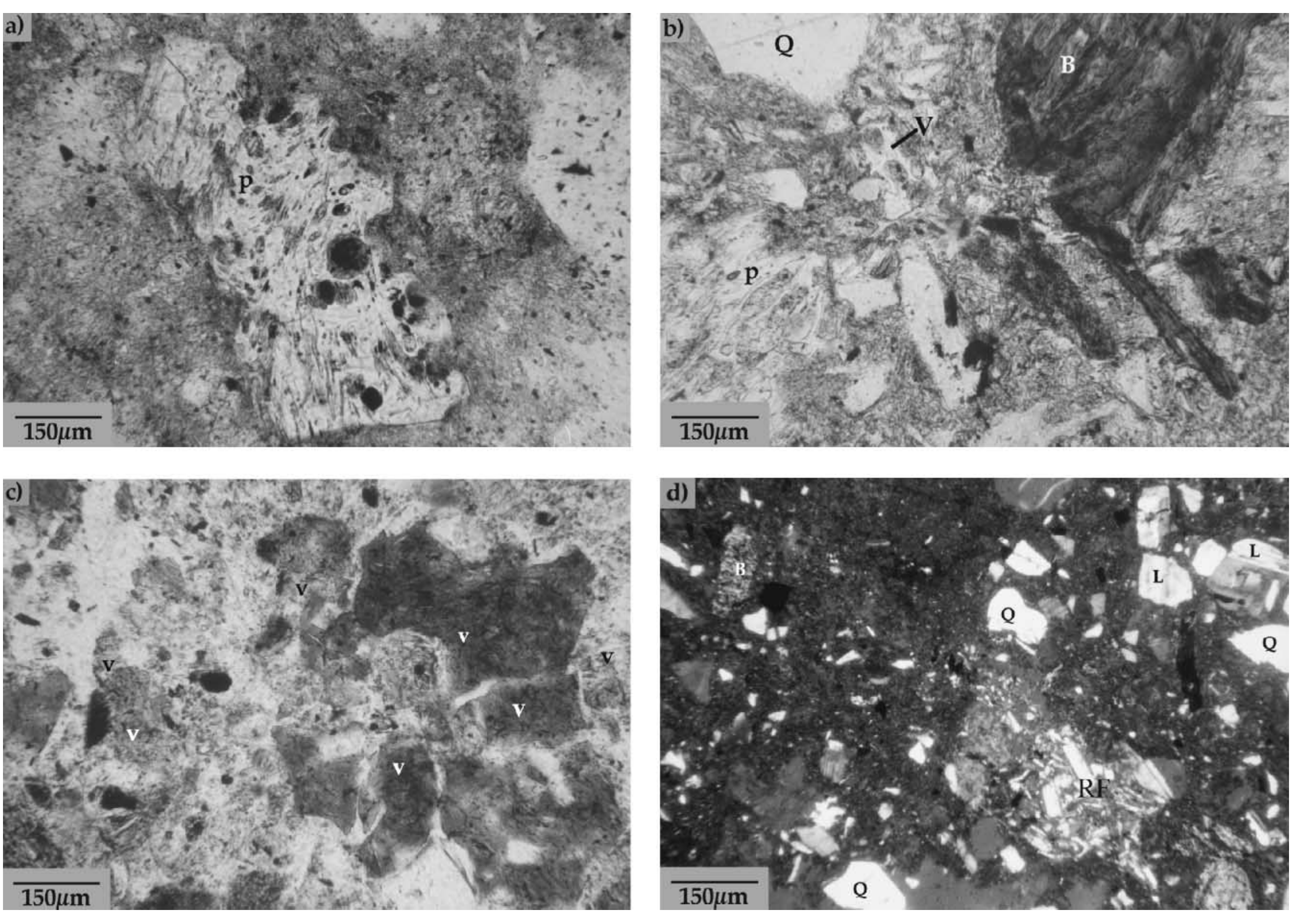

Fig. 2. Thin section photomicrographs of tuffs illustrating various pyroclasts and textures: (a) pumice grain (p) with abundant vesicles — Sample EL5, (b) abundant glass shards (V) in the matrix and biotite (B), quartz (Q), and pumice (P) — Sample B4, (c) brown glass shards (V) with curviplanar surfaces — Sample BEY3, (d) biotite (B), quartz (Q), plagioclase (L), and rock fragments (RF) — Sample B4. (a–c: ordinary illumination, d: X polars).

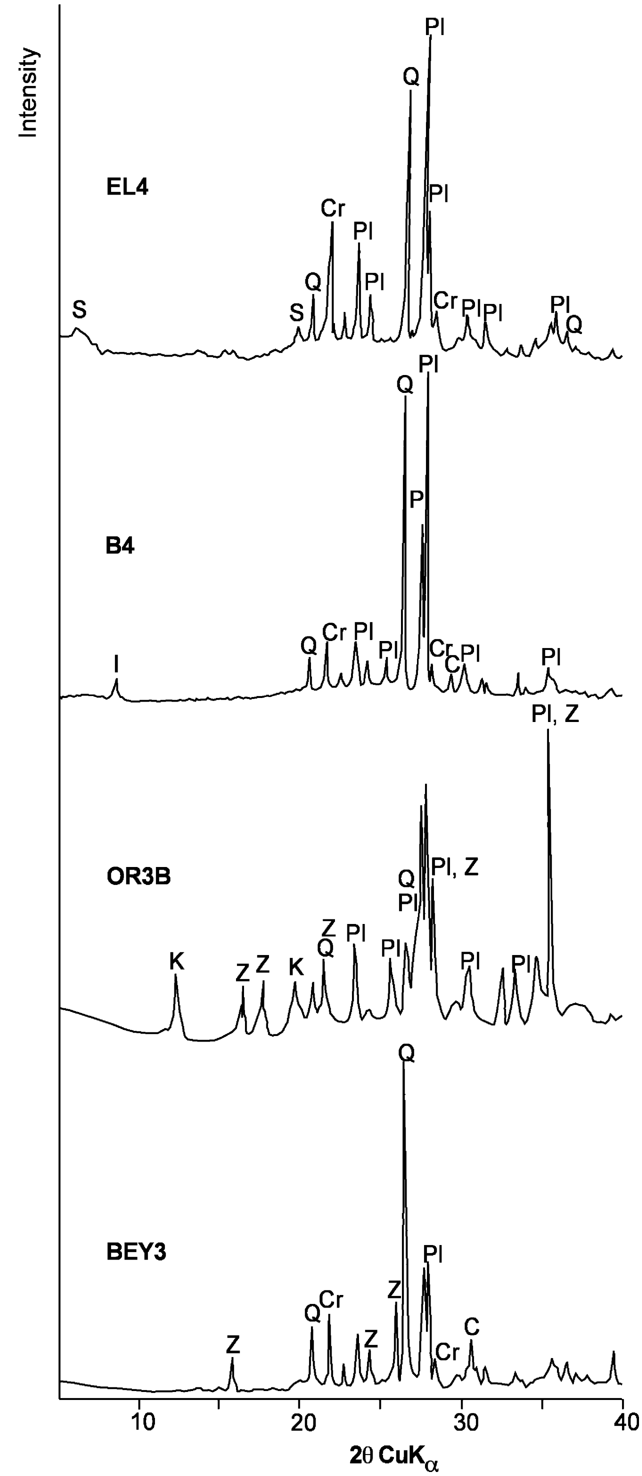

Fig. 3. X-ray diffractograms of whole rock samples. Pl = plagioclase; Q = quartz; Z = zeolite; C = calcite; K = kaolinite; I = illite; S = smectite; Cr = cristobalite.

(Fig. 4e,f). Although the volcanic glass and pumice fragments are generally fresh, clay mineral and opaline silica may form by their alteration. Opaline silica is common in the Bağlum tuffs and occurs as micron-sized spheres attached to volcanic glass shards (Fig. 4g,h).

4.2. Chemical composition

Major elemental oxide compositions of tuff samples are shown in Table 3 and reflect their mineralogical characteristics. Abnormally high CaO contents (10.69–16.04%) and LOI (11.22–13.94%) values of the Samples B2 and B3 are due to calcite enrichment, which is also observed in thin sections to be around 20%. Tuffs from the Orta area are distinguished by K_2O contents varying between 5.70% and 2.85%, which are relatively higher compared to the other samples. Although volcanic glass in tuffs contribute to their alkali content (Fig. 4d), higher K content of Orta tuffs is attributed to the presence of a zeolite mineral with K cation as also identified by XRD analysis (Fig. 3). In the studied tuffs, the main source of Na is plagioclase crystals. However, a slightly higher Na content in some samples, such as BEY3 and BEY5, is caused by a Na–zeolite (analcime) as also identified by XRD (Fig. 3).

The results of the chemical analysis (Table 3) are compared with the chemical requirements in ASTM C 618 [19] and TS 25 [14] as given in Table 4. The amounts of $SiO_2 + Al_2O_3$ + total Fe_2O_3 that play an important role in the occurrence of pozzolanic reactions are >70%, except for the Sample B3. All of the tuff samples investigated in this study also fulfill the chemical limitations for MgO, SO_3, and LOI. So, except for one sample, the studied tuffs fit well with the chemical requirements as to chemical composition prescribed in Table 4.

4.3. Pozzolanic activity

Pozzolanic activities of 13 test specimens are shown in Table 5. Their flexural strengths range between 1.7 and 3.0 MPa, while their compressive strengths are in the range of 7.4–16.0 MPa. These values fulfill the mechanical requirements of the standard as given in Table 4. All the mortar specimens that are prepared by mixing the tuff samples from the Elmadağ area are characterized by their high mechanical strengths.

The diagrams in Fig. 5 show the variations in the strength of mortars and in some of the oxide contents of tuffs. In the direction of decreasing strengths also occurs an increase in the K_2O and $Na_2O + K_2O$ contents. In addition, SiO_2 and Al_2O_3 values of tuffs show opposite relations with each other. The mortar specimens made of tuffs having high Al_2O_3 percentages (EL7, BEY5, OR4D, and BEY3) have relatively lower strengths.

It is known that the activity of a pozzolan depends on the $SiO_2 + Al_2O_3$ "reactive" content [20]. The solubility of silicate minerals are highly affected by factors like crystal structure, specific surface area, and pH [21,22], and the siliceous amorphous phase of raw materials is considered highly reactive. The studied tuffs from the Elmadağ and Bağlum areas have fresh volcanic glass, opaline silica, and free quartz as the most siliceous phases.

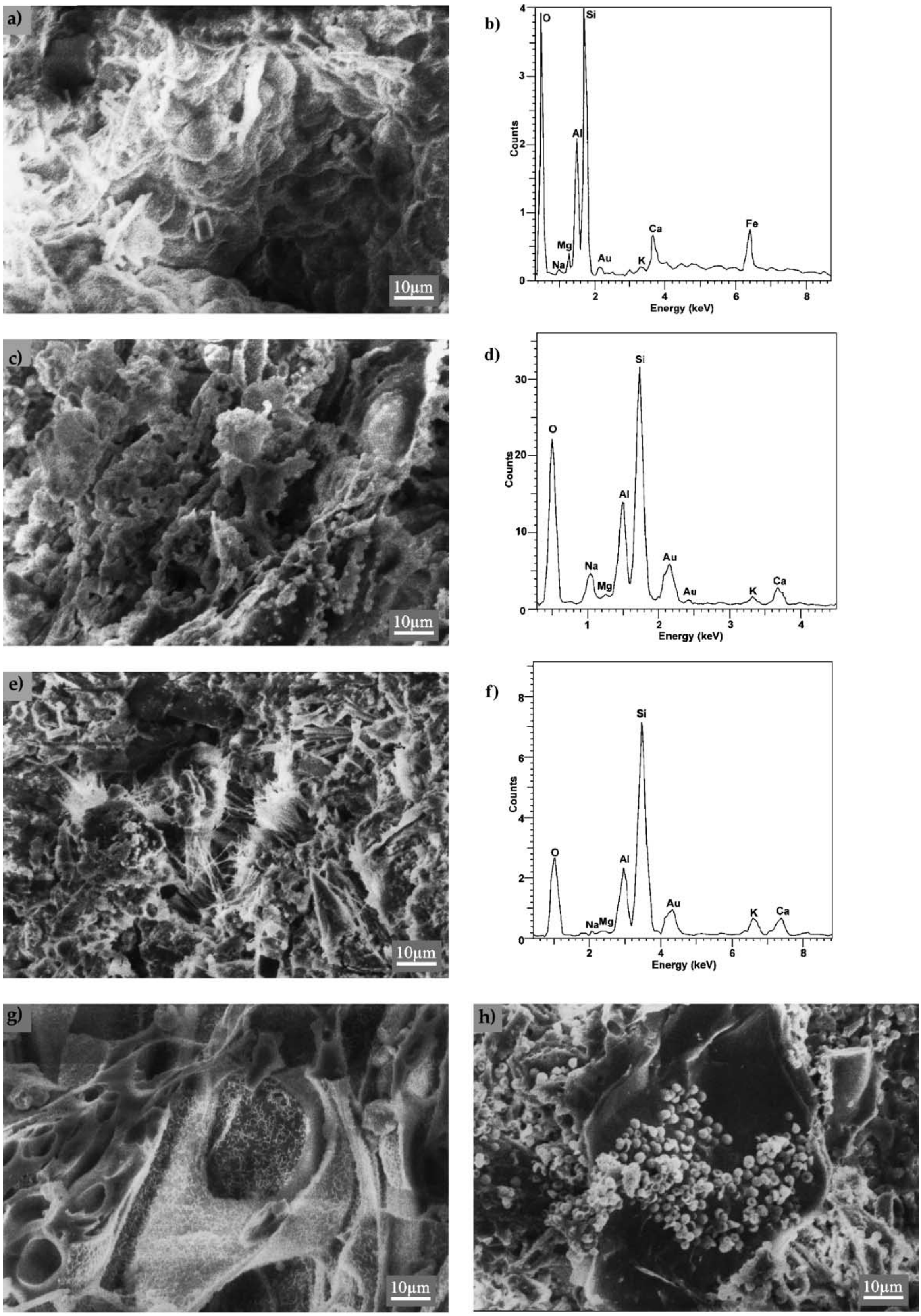

Fig. 4. SEM micrographs and EDX analysis. (a) Flaky morphology of smectite in the Sample BEY5 and (b) its EDX spectrum, (c) dissolution texture of volcanic glass in Sample EL1 and its (d) EDX spectrum, (e) fibrous zeolite forming clusters in the Sample BEY3 and (f) its EDX spectrum, (g) authigenic clay mineral attached to glass shard surface — Sample B7, and (h) opaline spheres on glass shard surface — Sample B4.

Table 3
Results of major element analysis of tuff samples (w/w%)

Sample	SiO_2	TiO_2	Al_2O_3	TFe_2O_3	MgO	CaO	Na_2O	K_2O	SO_3	LOI	Total	S + A + F
EL1	62.42	0.81	18.89	4.13	0.33	3.07	3.10	1.36	0.15	4.95	99.21	> 70
EL4	64.82	0.77	18.15	3.28	0.33	2.88	2.75	1.84	0.16	4.40	99.38	>70
EL5	64.86	0.49	18.08	3.16	0.16	2.63	2.75	2.38	0.21	4.55	99.27	>70
EL6	64.64	0.52	18.18	3.23	0.35	3.15	2.70	1.86	0.19	4.69	99.54	>70
EL7	62.53	0.52	20.58	3.24	0.35	2.87	2.85	1.79	0.14	4.54	99.41	>70
B2	55.77	0.69	13.83	2.90	0.53	10.69	2.06	1.87	0.21	11.22	99.77	>70
B3	48.69	0.24	13.54	2.20	0.37	16.04	2.55	1.95	0.08	13.94	99.60	< 70
B4	68.18	0.62	15.95	2.66	0.32	2.07	3.50	2.75	0.18	3.47	99.70	>70
B7	65.56	0.30	18.63	3.15	0.24	1.68	2.98	2.47	0.22	4.36	99.77	>70
OR1A	67.21	0.10	17.06	1.47	0.31	0.72	2.35	3.87	0.15	6.07	99.31	>70
OR1B	70.51	0.37	14.10	0.82	0.21	0.42	1.78	4.88	0.06	6.38	99.53	>70
OR2A	70.33	0.29	13.90	0.96	0.25	0.39	1.95	4.88	0.10	6.52	99.57	>70
OR2B	66.12	0.18	18.16	2.01	0.25	0.74	2.46	3.47	0.14	5.80	99.33	>70
OR3B	56.00	1.20	19.23	4.41	0.53	2.69	1.78	5.70	0.19	7.52	99.25	>70
OR4A	65.64	1.18	18.40	2.66	0.29	1.59	2.55	2.85	0.14	4.56	99.86	>70
OR4C	57.29	1.13	20.77	4.40	0.52	1.72	1.70	4.50	0.29	7.05	99.37	>70
OR4D	56.29	1.48	20.18	3.45	0.31	2.41	2.47	5.70	0.18	6.77	99.24	>70
PE1	63.15	1.05	18.20	3.65	0.16	2.64	2.75	2.52	0.15	5.12	99.39	>70
PE2	65.60	3.04	17.38	2.38	0.17	1.56	2.02	3.24	0.17	6.70	99.56	>70
PE3	68.00	0.81	15.87	1.66	0.12	1.55	2.42	3.45	0.11	5.68	99.69	>70
PE6	67.81	0.20	17.62	1.54	0.17	2.07	2.38	2.45	0.07	5.48	99.79	>70
PE8	66.33	0.32	17.40	1.09	0.14	1.39	1.60	3.02	0.09	7.98	99.36	>70
BEY3	61.78	1.48	21.14	2.19	0.12	3.72	3.58	2.11	0.07	3.14	99.33	>70
BEY5	65.55	0.71	20.86	1.31	0.17	1.83	3.58	3.00	0.10	2.12	99.23	>70

S + A + F = $SiO_2 + Al_2O_3 + TFe_2O_3$; TFe_2O_3 = total Fe_2O_3; LOI = loss on ignition.

They have higher pozzolanic activity (Fig. 5), most probably due to the abundance of amorphous phase. Comparatively, tuffs from the Orta and Karaşar areas are more altered and the formation of the secondary minerals like kaolinite and Na,K–zeolites from volcanic glass is detected by thin section and XRD analysis. Chemically, these samples have higher Al_2O_3 (>20%) and K_2O (>3%) contents. As can be seen in Fig. 5, these tuffs have lower values of mechanical strengths most probably due to negative effect of alteration.

5. Conclusions

Almost all of the studied tuffs from the Galatean Volcanic Province have major chemical components, $SiO_2 + Al_2O_3$ + total Fe_2O_3 exceeding 70%, conforming with the chemical requirements of ASTM and Turkish Standards. They also fulfill the mechanical requirements in terms of flexural and compressive strengths. In these respects, the studied region has important reserve potential for the cement industry.

Table 4
TS 25 standard requirements

$SiO_2 + Al_2O_3 + TFe_2O_3$ (%)	MgO (%)	SO_3 (%)	LOI (%)	7th day flexural strength (MPa)	7th day compressive strength (MPa)
>70	< 5	< 3	< 10	>1	>4

Although the SiO_2 and Al_2O_3 affect the pozzolanic activity of tuffs mainly, the abundance of highly vesicular pumice fragments and glass shards in comparison with quartz, plagioclase (Na–Ca feldspar), biotite, hornblende, clinopyroxene crystals, and lithic fragments are also important. Among the studied tuffs, those from Elmadağ and Bağlum areas are rich in glassy fragments that impart higher strength to the mortar. Obviously, the alteration and formation of the secondary mineral phases are also important. Authigenic clay minerals that are attached to their surfaces and zeolites form by dissolution of volcanic glass and reduce the reactivity of amorphous glass surfaces.

Chemical analysis reflects the mineralogical characteristics of tuffs. Higher values of Ca, K, Na, and Al are due to

Table 5
Results of the pozzolanic activity tests

Sample	7th day flexural strength (MPa)	7th day compressive strength (MPa)
EL1	2.4	12.2
EL4	3.0	16.0
EL5	2.7	14.4
EL6	2.8	13.6
EL7	2.4	10.5
B4	2.2	9.8
B7	2.7	12.4
OR4D	1.8	7.9
PE1	1.8	9.3
PE3	2.0	8.4
PE6	2.2	12.5
BEY3	1.7	7.4
BEY5	1.8	8.8

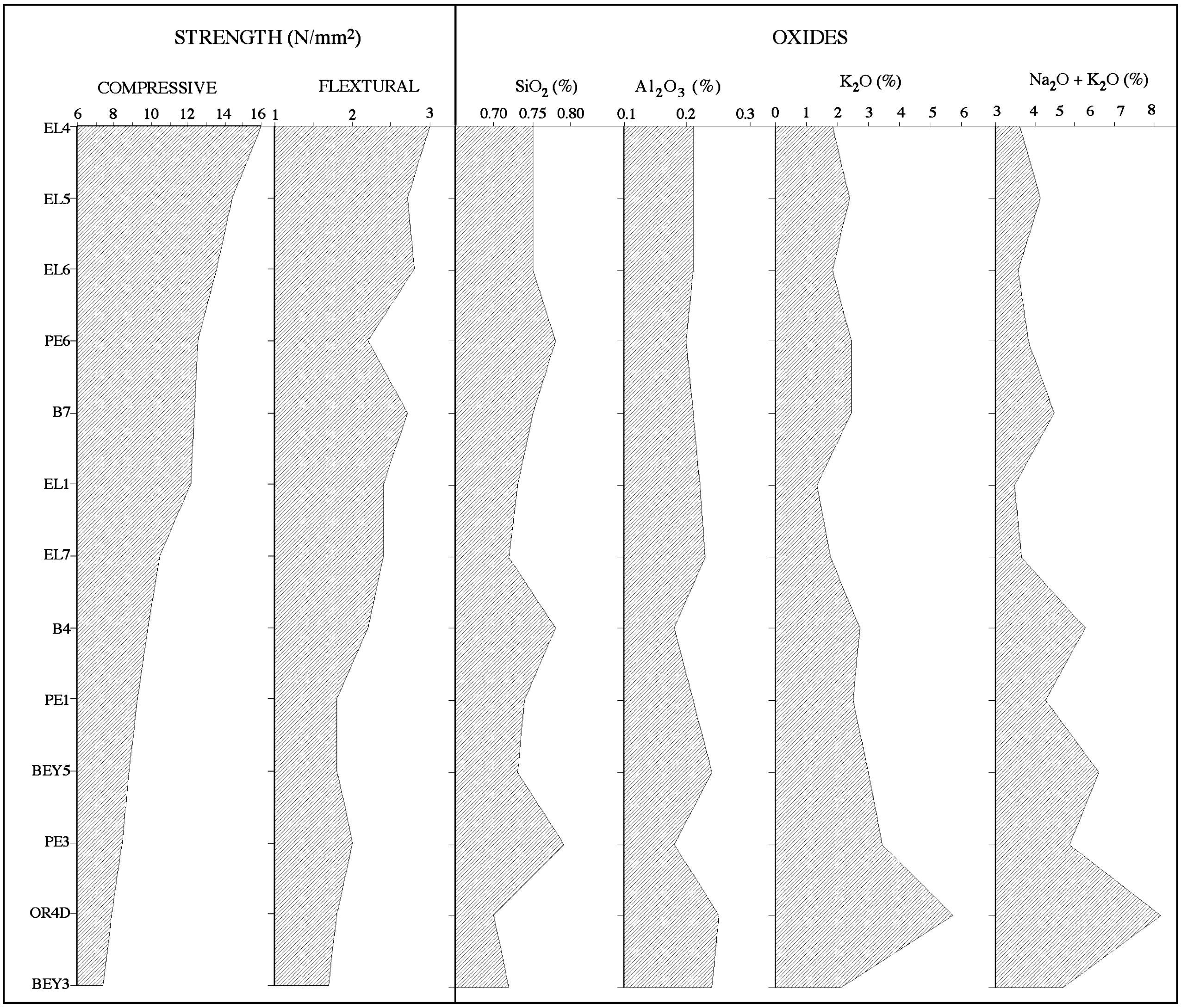

Fig. 5. Variations of mechanical properties and chemical composition of tuff samples.

increasing quantities of calcite, plagioclase, clay minerals, and zeolites. The chemical composition of tuffs shows a variation, especially with respect to K_2O and K_2O+Na_2O, which seems to relate inversely with decreasing strengths of the mortar specimens.

Petrographical analysis of tuffs by means of optical microscopy, XRD, SEM–EDX, and spectroscopic techniques are useful for the evaluation of the suitability of tuffs in pozzolanic cements. They are fast and easily accessible techniques to reveal the petrographical, mineralogical, and textural characteristics that may have important control on the technical behavior of pozzolanic cements.

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