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Improving the geopolymeric reactivity of Earth of Datça as a natural pozzolan in developing green binder

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ABSTRACT

The purpose of the current study is to investigate the geopolymeric reactivity of volcanic tuff of Datça Peninsula (Earth of Datça) in Turkey and to evaluate whether it can be used as a green binder. The effects of alkali activator type, alkali hydroxide concentration, activator ratio, and curing conditions on the physical and mechanical properties were investigated. A combination of sodium silicate solution (SS) and potassium hydroxide (KOH) (12.5 M) with an activator ratio of 2.5 is selected as optimum activator type. Sealed curing at 90 °C and 90 ± 5% RH for 24 h provides the highest physical and mechanical properties. Results reveal that "Earth of Datça" as a natural pozzolan is a suitable precursor for the production of a geopolymer material with the properties of 2.0 g/cm^3 bulk density, 5.4% water absorption ratio, 0.64 saturation coefficient, and 10.5 MPa compressive strength. The mortar produced in this research has promising characteristics to be used as a building material.

1. Introduction

Cement production is an energy intensive process, and one tonne of ordinary Portland cement (OPC) production emits approximately one tonne of CO_2 [1] mostly stemming from calcination of limestone and clay at 1400 °C and the grinding process of clinker. Recently, the quest for alternative binders with a low ecological footprint has been proceeding around the world. Geopolymers may be regarded as an environmentally benign alternative because of their production requires less energy and leads to less CO_2 emissions compared to that of OPC [2]. The production of geopolymer binder relies on a reaction of solid aluminosilicate source with aqueous alkali activators, moulding and curing at ambient or elevated temperatures. Any kind of aluminosilicate source which can be dissolved in alkaline medium such as slag, fly ash [3–7], bottom ash [8,9], boiler ash [10], calcined clay [11], copper mine tailing [12], ceramic dust waste [13], calcined clayey sediments and water potabilization sludge [14], can be used for geopolymer synthesis. Indeed, most of the studies in literature have focused on using of industrial solid wastes (artificial pozzolans) in the production of geopolymer binders. However, the availability of industrial solid wastes depends on other production technologies. The amount of slag can only increase as the demand for steel increases. Furthermore, its production is concentrated in a relatively small number of countries, which makes its availability limited. Besides, due to the environmental pressures more steel is being recycled. Availability of fly ash depends on the electricity production, which burns coal. Adverse environmental effects of coal burning is being questioned and its quality is very variable. Metakaolin when obtained as an industrial ceramic waste or by the production of calcined clay where no sophisticated equipment is necessary may be a good alternative [15]. On the other hand, natural pozzolans are available in huge amounts in certain geographical regions and are easily accessible, which ensures low cost and environmental impact [16].

Natural pozzolan, containing mostly amorphous silica (SiO_2) and alumina (Al_2O_3), has been proven to function as a geopolymer precursor [17,18]. In the geopolymerization, activator type and ratio along with the curing conditions are effective parameters. The compressive strength of the resulting material is commonly used as a quantitative indicator of reactivity of aluminosilicate source [19]. Taftan pozzolan-based geopolymer binder in Iran, activated with a mixture of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) and cured at ambient conditions, exhibits 28-day compressive strengths up to 63 MPa [20,21]. The effects of various curing conditions (hydrothermal treatment at 45–85 °C for 5–20 h and autoclave curing at 125–210 °C for 20–50 h) on the properties of the Taftan pozzolan-based geopolymer were also investigated. The highest compressive strengths obtained were 57.5 MPa after hydrothermal treatment at 85 °C for 20 h and 108.7 MPa after autoclave

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curing at 210 °C for 30 h [22]. Sirjan natural pozzolan-based geopolymer cements were activated by a proportioned mixture of NaOH and Na_2SiO_3, exhibiting 28-day compressive strength up to 56 MPa [23]. Volcanic ash powders from Etna (Italy) and Cameroon were used as aluminosilicate sources and activated with a combination of NaOH and Na_2SiO_3 and cured at 25-75-100-200 and 400 °C; 12 h for 400 °C and 48 h for the rest. The test results reveal that the values of compressive strength (>20 MPa) and water absorption (≤20%) comply with the ASTM C62 and C216 standards for building and facing bricks [24]. Jordanian natural pozzolan-based geopolymer mortar was produced and strength grades up to 24 MPa were obtained depending on the NaOH concentration (10–12 M), Na_2SiO_3/NaOH ratio (2–2.5) and alkaline activator-to-binder ratio (0.30–0.40) [25]. The compressive strength up to 6 MPa was obtained with natural Bigadic pozzolan-based geopolymer mortar in Turkey after the optimization of NaOH concentration (8–12 M) and Na_2SiO_3/NaOH ratio (1.0–3.0) [26]. Geopolymer mortar and concrete samples with a compressive strength up to 37 MPa were produced by using Nevşehir volcanic tuff of Turkey activated with NaOH (10–16 M) and a mixture of NaOH and Na_2SiO_3 with an alkaline activator-to-binder ratio (0.35–0.60) and curing temperature (90–150 °C) [27]. The Bafoundou tuff was activated with 4–12 M NaOH solution and cured at 40 and 80 °C for 28 days or at 120 °C for 12 h to improve the durability of tuff-based bricks. The specimens attained compressive strengths that range from 4 to more than 20 MPa, depending on the alkali concentration, temperature and length of curing [28]. Clay from the Niemenike deposit and pre-treated clay at 700 °C were used as aluminosilicate sources to manufacture bricks at relatively low temperatures (≤100 °C). Test results show that the strengths of natural and calcined clays increase with the increasing of NaOH concentration from 4 M to 12 M and the produced bricks are durable and relatively inexpensive [29].

Turkey is rich in natural pozzolans and about 155.000 km^2 of the country is covered by volcanic rocks. There are volcanic rocks at the antique site settled at the far west end of Datça Peninsula called Knidos and its eastern region called Çeşmeköy, Yazıköy and Cumalı [30] (Fig. 1). According to the researchers, they come to these regions through air from the strong volcanic eruptions of volcanic centers of Nysiros and Yelli Islands and deposited at the dip basin [31].

The purpose of this study is to investigate the geopolymeric reactivity of volcanic tuff of Datça Peninsula [Earth of Datça (ED)] in Turkey and to evaluate whether it can be used as a green binder. In the scope of the study, as extensively studied in the literature, key factors affecting the degree of geopolymerization of the material (alkali activator type; the concentration of alkali hydroxide; sodium silicate-to-alkali hydroxide ratio which is termed as activator ratio; and curing conditions) and their consequent results as physical and mechanical properties are investigated.

2. Materials and methods

2.1. Materials

Natural volcanic tuff of ED in Turkey was used as aluminosilicate source. Particle size distribution of ED is given in Fig. 2. The specific surface area of the ED was measured as 5467.75 cm^2/g according to the Blaine method [32]. The specific gravity is 2.52 g/cm^3.

Semi quantitative element (X-ray Fluoresant spectometer- XRF) analysis and Qualitative phase (XRD) analysis of the pozzolanic sample (ED) and hardened mortar produced for the pozzolanic activity (ED)T test were done in the previous research [30] by using Philips 71 PW-2404 model XRF (X-ray Fluoresant spectometer) equipment (Table 1) and a Shimadzu XRD-6000 equipment using Cu X-ray tube

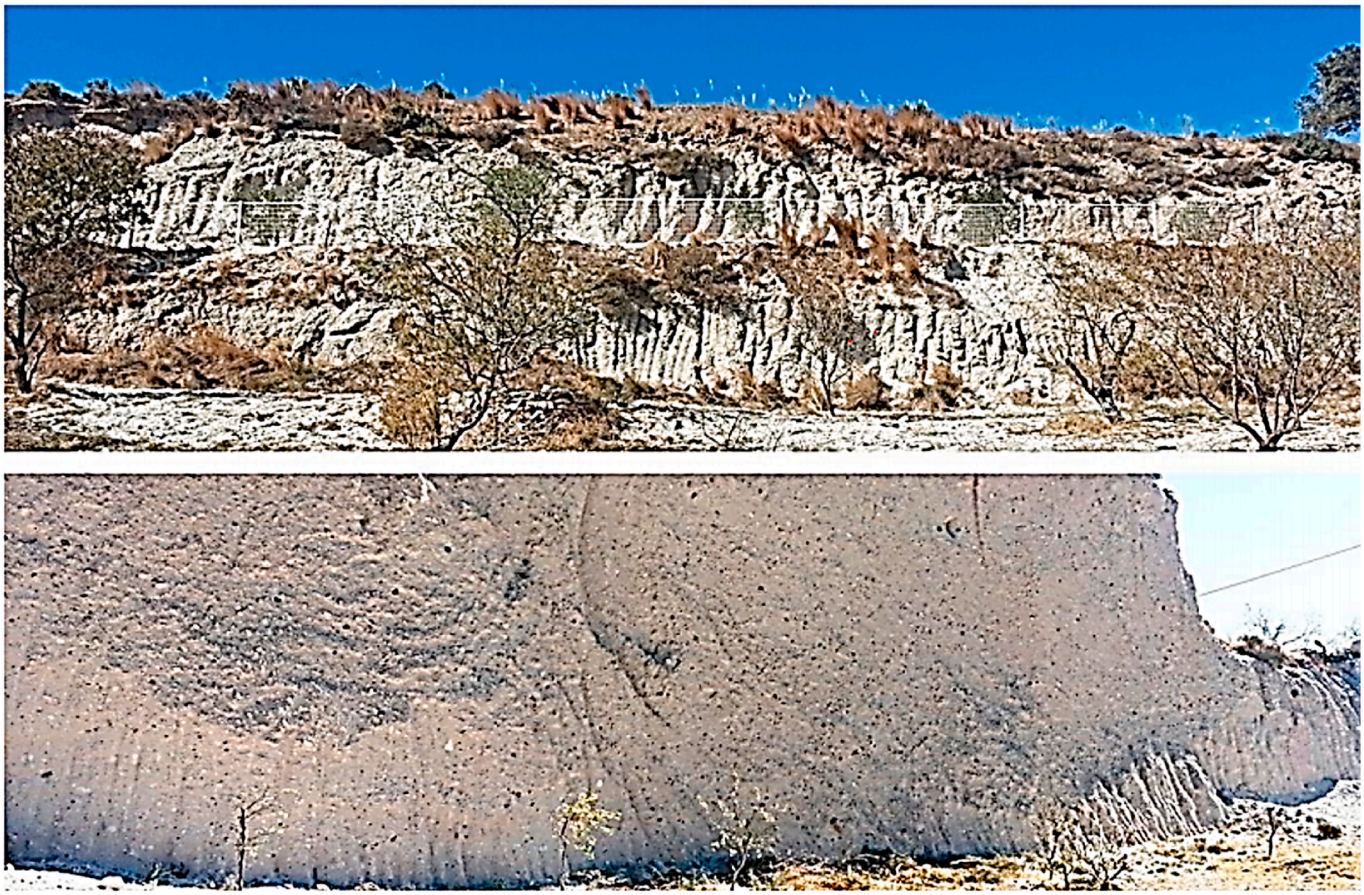

Fig. 1. View of the Earth of Datça as found in the nature.

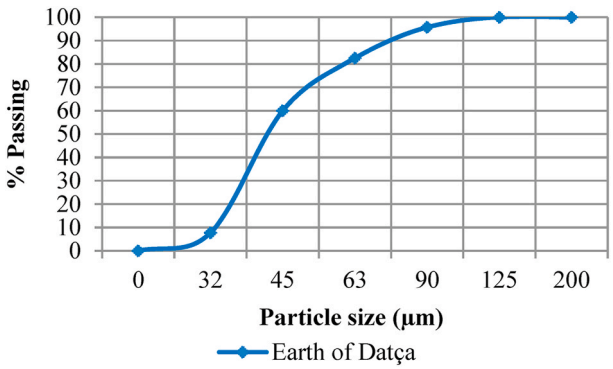

Fig. 2. Particle size distribution of Earth of Datça.

Table 1
Chemical composition of the ED, quicklime, and hardened mortar (ED)T.

Component	CaO (%)	Earth of Datça (%)	(ED)T Mortar (%)
SiO_2	–	75.289	58.934
Al_2O_3	–	15.991	11.510
Fe_2O_3	–	0.984	0.829
Na_2O	–	2.211	1.361
K_2O	–	3.026	2.408
CaO	85	1.222	21.377
CO_2	5	–	–
MgO	1.5	0.622	2.870
P_2O_5	–	0.072	0.077
TiO_2	–	0.149	0.148
MnO_2	–	0.047	0.050
Cr_2O_3	–	0.005	–
NiO	–	0.004	0.005
CuO	–	0.002	–
ZnO	2.23	0.002	0.006
Rb	–	0.006	0.007
SrO	–	0.015	0.019
V_2O_5	–	–	0.022
Y_2O_3	–	0.002	0.006
ZrO_2	–	0.010	0.015
Nb_2O_5	–	0.001	0.008
BaO	–	0.097	0.075
Cl	–	0.092	0.078
SO_3	0.8	0.1	0.194
PbO	–	–	–
ThO_2	–	–	–
LOI	-	0.15	-
Total	93.8	100.00	–

(=1.5405 Å) respectively. XRF results of the ED fulfil the requirements of TS 25 [33] in order to be used as a natural pozzolan in cement, concrete and also other binder types because its SiO_2+Al_2O_3+Fe_2O_3 content (92.26%) is higher than 70.0%, SO_3 and Cl contents (0% and 0.092%) are lower than 3.0% and 0.1%, respectively [33]. In addition, the flexural and compressive strength results of the pozzolanic activity test (1.43 and 6.12 MPa, respectively) meet the minimum requirements of TS 25. It may be revealed from the XRF analysis that ED has similar chemical properties with other natural pozzolan types which are mentioned in the introduction. According to the qualitative phase (XRD) analysis, ED contains cristobalite in lower quantity, quartz, feldspar, and a compound in amorphous structure and (ED)T indicates the hydration products and contains cristobalite, quartz, feldspar, portlandite and a compound in amorphous structure (Fig. 3).

Sodium hydroxide (NaOH), potassium hydroxide (KOH) and sodium silicate (Na_2SiO_3) solutions (SS) were supplied from a chemical company. NaOH and KOH solutions were prepared by dissolving the pellets with 99% purity in deionised water one day prior to usage. The mass of NaOH and KOH solids in the solutions are expressed as Molar (M). The molecular weights of NaOH and KOH solids are 40 and 56.1, respectively. NaOH solution with the concentration of 7.5 M consists of 7.5 × 40 = 300 g of NaOH pellet per litre of the solution and 7.5 M KOH solution consists of 7.5 × 56.1 = 420.7 of KOH pellet per litre of the solution. Molar ratio of SS is SiO_2/Na_2O = 3.4. CEN standard sand based on TS EN 196-1 [34] was used for geopolymer mortar.

2.2. Methods

In the current study, the parameters which may be effective on the physical and mechanical properties of ED based geopolymer were investigated in three stages given below. Each consecutive stage determines the parameters of the following stage:

(i) The effect of alkali activator type (NaOH (N)), KOH (K), SS + NaOH (SN), and SS + KOH (SK);
(ii) The effect of thermal curing conditions;
(iii) The effect of alkali hydroxide (NaOH, KOH) concentration (7.5, 10, 12.5, and 15 M) and the effect of activator ratios (SS/NaOH and SS/KOH) (1.0, 1.5, 2.0, 2.5, by weight).

The effective parameters researched in each stage, mixture proportions and their codes, and curing conditions of the experimental design for the development of geopolymerization reactions are given Table 2.

At the first two stages, to understand the behaviour of the ED to

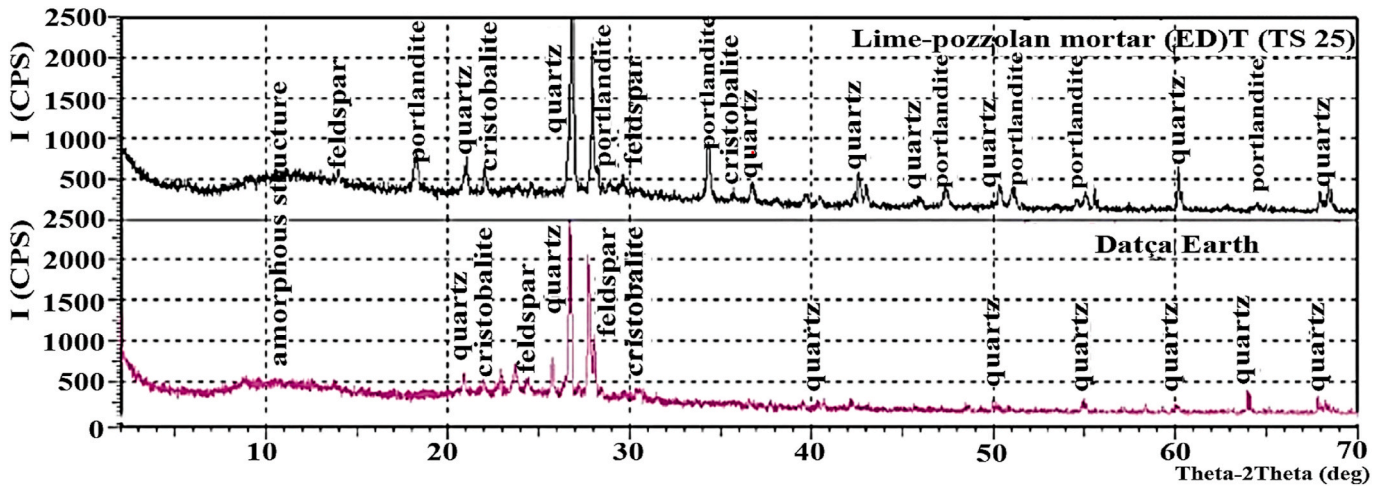

Fig. 3. XRD data of ED and hardened mortar (ED)T.

Table 2
Mixture proportions and curing conditions of the ED-based geopolymer.

Stage	Specimen Code	Alkali activator type	Concentration of alkali hydroxide (MOH)*	Activator ratio (SS/MOH)	Activator/dry pozzolan (by weight)	Dry Pozzolan/ sand (by weight)	Total water/ total solid (by weight)	Flow (%)	Curing conditions
i	7.5N-0-100	N	7.5	–	0.3	–	0.27	100	70 °C, sealed
	7.5K-0-100	K		–					
	7.5SN-1-100	SN		1.0					
	7.5SK-1-100	SK		1.0					
ii	7.5SK-1-100	SK	7.5	1.0	0.3	–	0.27	100	70 °C, sealed 70 °C, unsealed 90 °C, sealed 90 °C, unsealed
iii	7.5SK-1-60	SK	7.5	1.0	0.3	1/3	0.10	60	90 °C, sealed
	7.5SK-1.5-60			1.5					
	7.5SK-2-60			2.0					
	7.5SK-2.5-60			2.5					
	10SK-1-60		10	1.0					
	10SK-1.5-60			1.5					
	10SK-2-60			2.0					
	10SK-2.5-60			2.5					
	12.5SK-1-60		12.5	1.0					
	12.5SK-1.5-60			1.5					
	12.5SK-2-60			2.0					
	12.5SK-2.5-60			2.5					
	15SK-1-60		15	1.0					
	15SK-1.5-60			1.5					
	15SK-2-60			2.0					
	15SK-2.5-60			2.5					

(*) M: Na and/or K.

produce a geopolymeric binder, sand was not added to the mixtures. Alkali activator to dry pozzolan ratio was kept constant as 0.3 by weight throughout the study.

At first, only the effects of activator types on the geopolymeric reactivity were tested. Because the molarity of alkali hydroxides ranges from 5 M to 10 M for the activation of natural minerals [35], the concentration of NaOH and KOH was selected as 7.5 M. The workability of the fresh binder was specified with flow table test in accordance with ASTM C 1437-01 [36]. Although the flow of cement-based mortars is given as 110 ± 5 as per ASTM C 109 [37], the optimum flow where no water seepage from the mould was found 100 for ED. Total water/total solid ratio was calculated as 0.27.

At the second stage, the effects of thermal curing conditions on the binder properties were investigated. After the evaluation of the results of the previous stage, SK (SS + KOH) was determined as the base activator type due to having the highest strength for the following stages of the study. After the samples were moulded, they were sealed with Polyethylene (PE) film to avoid rapid moisture loss and pre-cured at 20 ± 2 °C and 95 ± 5% RH for 24 h. This initial curing was applied to all the test specimens in the study. After this initial curing, the samples were demoulded. Then, to find out the effects of curing temperatures and humidity, the samples were cured at 70 °C and 90 °C for 24 h both in unsealed and sealed conditions with PE film. Throughout the study, the cured specimens were left to air curing at 20 ± 2 °C and 50 ± 5% RH for 7 days, prior to testing.

After the determination of the suitable activator type (SK═SS + KOH) and curing conditions (90 °C in sealed conditions for 24 h) for ED-based geopolymer binder in the previous two stages, at the third stage, geopolymer mortars were prepared with 1/3 dry pozzolan/sand ratio. The workability of the fresh mortar was specified with flow table test and the optimum flow was found as 60. The effects of alkali hydroxide concentrations (7.5, 10, 12.5, and 15 M) and activator ratios (1.0, 1.5, 2.0, 2.5, by weight) were investigated respectively.

The specimens were coded in [XX-X-XX] format. The first symbol group represents the concentration and type of alkali hydroxide; the second symbol group points out the activator ratio, the third group indicates the flow of the fresh mortar.

Both the physical and mechanical tests were applied on the prismatic test specimens 40 mm × 40 mm × 160 mm in size. Three specimens were prepared for each test. To calculate the bulk density according to TS EN 1015-10 [38], the specimens were dried at 105 ± 5 °C in a ventilated oven until the constant mass was reached. Bulk density is the ratio of the recorded dry mass to the volume of the specimens.

The capillarity coefficient (C) test was applied on oven-dried specimens. The weight of the specimens and the area of the base were recorded. Then the specimens were covered with a film on the sides and placed on the thin supports in a water tank (a depth of 3 ± 1 mm) to absorb water vertically from the base. At time intervals of 1, 4, 9, 25, 36, 49, 64, 81, and 100 min the weights of the specimens were recorded. The capillarity coefficient was calculated by using Eq. (1):

$$C = (m_i - m_d) / A.t_i^{1/2} \quad (1)$$

where m_d = mass of the dry specimen, (gr); m_i = successive masses of the specimen at specified time intervals, (gr); A = area of the base immersed in water, (m^2); t_i = times elapsed from the beginning of the test until the times at which the successive masses m_i was measured, (s) [39].

Water absorption ratios (A_b) of the specimens were calculated according to TS EN 13755 [40]. The specimens were weighed after drying at 105 ± 5 °C until the constant mass (m_d). Then, they were placed in a tank, and clean water at 20 ± 10 °C was added gradually over a period of 2 h until they were completely immersed in water. 48 h later, the saturated specimens were taken out of the water, wiped off the surface water with a damp cloth and weighed (m_s). The water absorption ratio

of the specimens was calculated by Eq. (2):

$$A_b = ([m_s - m_d] \times 100) / m_d \quad (2)$$

The UPV test was done in accordance with TS EN 14579 [41] using a portable ultrasonic non-destructive device (Proceq).

The flexural strength was determined with a universal testing machine with a capacity of 300 kN, at a rate of loading of (50 ± 10) N/s according to TS EN 196-1 [34]. As per the standard, for all flexural tests the specimen sizes are 40 × 40 × 160 mm. The specimen was placed in the apparatus with one side face on the supporting rollers and with its longitudinal axis normal to the supports. The load was applied vertically by means of the loading roller to the opposite side face of the specimen and increased at the rate of (50 ± 10) N/s until fracture. The flexural strength (R_f) was calculated by using Eq. (3):

$$R_f = (1.5 \times F_f \times l) / b^3 \quad (3)$$

where b is the side of the square section of the specimen, (mm); F_f is the load applied to the middle of the specimen at fracture, (N); l is the distance between the supports, (mm).

Compressive strengths were determined by tests applied on the six specimens that were previously broken into two pieces in flexural tests. The specimen was centered laterally to the plates of the machine, and the load was increased at a rate of (2400 ± 200) N/s over the entire load application until fracture. Compressive strength (R_c) was calculated from Eq (4):

$$R_c = F_c/1600 \quad (4)$$

where F_c is the maximum load at fracture, (N); 1600 is the area of the plates (40 mm × 40 mm), (mm^2).

Arithmetic mean and standard deviation of the test results were calculated and the results were statistically analysed according to the Chauvenet's Criterion, which specifies that a reading may be rejected if the probability of obtaining the particular deviation from the mean is less than 1/2 N. N is the number of measurements. In the study, 3 measurements were done in the physical tests, and 6 measurements were done in the compressive strength tests. Thus, the ratio of maximum acceptable deviation of the measurements were recorded as 1.38 and 1.73, respectively, according to the Chauvenet's Criterion table for eliminating a reading [42]. All measurements that deviate from the mean more than the maximum acceptable deviation were rejected. Then, a new arithmetic mean was calculated from the remaining measurements. The coefficient of variation which defines the extent of deviation according to the mean was also calculated. All the test results are given in Table 3.

3. Results and discussion

3.1. Chemical and mineralogical properties of Earth of Datça as a geopolymer precursor

Of the common oxides in geopolymer chemistry, i.e. SiO_2, Al_2O_3, Fe_2O_3 and CaO, alumina and silica are the two major oxides which control the geopolymeric network formation and strength development [43] and it is declared that, the available calcium content of reacting components should be low to enable formation of three-dimensional polymeric chain and ring structure consisting of Si–O–Al–O bonds [16]. On the other hand, the calcium content is also an important factor affecting the setting time and final strength in concrete, while there are indications that it may also affect the properties of geopolymers [44]. ED has comparatively lower calcium content than other natural pozzolans except Nevşehir volcanic tuff (Table 4).

Samples containing high amount of amorphous silica (SiO_2) and alumina (Al_2O_3) and, low SiO_2/Al_2O_3 molar ratio of amorphous phase have higher values of compressive strengths [43,45,46]. For example, metakaolin which has SiO_2/Al_2O_3 molar ratio in the range of 3.4–3.8 showed high strength development [47]; fly ash materials with reactive SiO_2/Al_2O_3 molar ratio higher than 3.3 induced formation of zeolitic phases, and led to low strength development [48], and Jordanian natural pozzolan, which has low molar ratio SiO_2/Al_2O_3 of 2.9, showed strength development up to 17 MPa [25] (Table 4). ED, parallel to its comparatively higher SiO_2/Al_2O_3 ratio (4.70) has lower compressive strength. However, since the interrelationship between chemical composition and reactivity of individual minerals is such an extremely complex issue that the prediction of geopolymeric reactivity based only on the bulk chemical composition of the starting materials may not be of much value [43].

3.2. Influence of alkali type on the properties of the ED based geopolymer binder

The influence of alkaline solution types on the mechanical properties of the ED based geopolymer binder is given in Fig. 4. Specimens produced with KOH based activators have higher strengths than specimens based on NaOH activators. The flexural and compressive strengths of KOH-activated binder were respectively 1.06 and 1.11 times higher than its NaOH-activated counterpart. In addition, the flexural and compressive strengths of SS + KOH activated specimens were found 1.21 and 1.54 times higher compared to SS + NaOH activated specimens under the same conditions. Differences determined in the mechanical properties may be related to the influence of the alkali activator type, on the extent of dissolution and polycondensation reactions. Na^+ having a smaller ionic size leads to a higher extent of dissolution and displays cation-anion ion-pair reaction with the smaller silicate oligomers. As these types of ion-pairs don't match with another silicate anion, the formation of larger silicate oligomers is prevented. In addition, the compound of Al $(OH)^{4-}$ cannot readily bind with smaller silicate oligomers like monomers. On the other hand, K^+ having larger ionic size, leads to the formation of larger silicate oligomers with which Al $(OH)^{4-}$ can readily bind. Thus, more geopolymer precursors form in the KOH-activated mixtures, which lead to the production of materials having higher mechanical properties [49]. Furthermore, KOH leads to the increase of the degree of polycondensation reaction that further increases the mechanical properties of the matrix and produces stronger geopolymer material [50]. That KOH-activated specimens possessed higher mechanical properties in comparison to NaOH-activated specimens were also observed in Taftan and Shahindej natural pozzolan-based [35], Cameroonian volcanic ash-based [51], blast furnace slag and Taftan pozzolan-based [52,53] and bottom ash-based [8] geopolymer binders in the literature.

The existence of SS solution in ED-based geopolymer binder leads to the increase of the mechanical properties. The flexural and compressive strengths of SS + NaOH activated binders were 1.10 and 1.24 times, respectively, higher than those of only NaOH-activated binder. In addition, the flexural and compressive strengths of SS + KOH activated binders were 1.25 and 1.73 times, respectively; higher compared to only KOH-activated specimens. The alkaline mixture SS + KOH, was found more effective than the use of only KOH regarding the compressive strength development (5.78 > 3.33 MPa). The increment in the mechanical properties due to the addition of SS to the alkaline mixtures based on either NaOH or KOH may depend on: (i) SS enhances the dissolution rate of Si and Al of the aluminosilicate source; (ii) Al ions dissolve easily in the alkali medium due to the fact that Al–O bonds of the aluminosilicate source are weaker than Si–O bonds. Consequently, existence of additional Si ions in the reaction medium before the dissolution of raw material may increase the degree of geopolymerization [52]. It is known that the compressive strength is an indication of the degree of geopolymerization [54]. In addition, the existence of SS in the mixture also provides stronger ion-pair formation, and this leads to more long-chain silicate oligomers in addition to Al–O–Si compounds, in other words geopolymer precursors. The presence of the more long-chain silicate oligomers means that geopolymer

Table 3
Test results of the ED-based geopolymer.

Stage	Specimen code	Curing	Bulk Density (g/cm^3)		Capillarity coefficient (cm/$\sqrt{dk}$)		Water absorption by weight (%)		Porosity (%)		Ultrasound pulse velocity (km/s)		Flexural strength (MPa)		Compressive strength (MPa)	
			Mean	Coefficient of variance (%)	Mean	Coefficient of variance (%)	Mean	Coefficient of variance (%)	Mean	Coefficient of variance (%)	Mean	Coefficient of variance (%)	Mean	Coefficient of variance (%)	Mean	Coefficient of variance (%)
i	7.5N-0-100	70 °C, sealed											1.33	1.16	3.02	1.23
	7.5K-0-100												1.40	1.08	3.33	1.54
	7.5SN-1-100												1.45	1.01	3.75	1.72
	7.5SK-1-100												1.75	1.10	5.78	1.65
ii	7.5SK-1-100	70 °C, sealed											1.75	0.87	5.78	1.11
		70 °C, unsealed											0.45	0.94	4.26	1.20
		90 °C, sealed											1.90	0.72	9.36	1.05
		90 °C, unsealed											0.68	0.71	5.60	0.96
iii	7.5SK-1-60	90 °C, sealed	2.00	0.29	0.1040	1.35	7.61	1.36	23.08	0.26	2.51	1.12	2.59	0.95	4.62	0.75
	7.5SK-1.5-60		2.00	0.10	0.1070	1.32	7.43	0.86	22.90	0.45	2.63	1.18	2.75	0.76	4.80	0.71
	7.5SK-2-60		2.01	0.11	0.1119	1.32	7.31	0.64	22.50	0.52	2.73	1.04	2.83	0.84	5.51	0.52
	7.5SK-2.5-60		2.01	0.15	0.1200	1.24	7.19	1.25	22.25	0.48	2.84	0.98	3.25	0.77	6.16	0.87
	10SK-1-60		2.02	0.22	0.1220	1.16	7.01	0.86	22.52	0.57	2.80	0.95	3.41	0.62	4.82	1.14
	10SK-1.5-60		2.03	0.17	0.1251	1.23	6.98	0.12	22.30	0.62	2.86	0.90	3.67	0.85	5.00	1.26
	10SK-2-60		2.03	0.29	0.1270	1.32	6.92	1.20	22.17	0.54	2.90	0.84	3.74	1.00	5.64	1.03
	10SK-2.5-60		2.04	0.14	0.1292	1.14	6.86	1.07	21.84	0.41	2.94	0.76	4.23	1.04	6.29	1.01
	12.5SK-1-60		2.05	0.23	0.1255	0.96	6.38	0.42	21.47	0.38	2.90	0.78	4.63	0.87	6.17	1.14
	12.5SK-1.5-60		2.06	0.28	0.1270	0.84	5.87	0.06	21.29	0.64	2.93	0.57	4.76	0.62	6.25	1.52
	12.5SK-2-60		2.07	0.29	0.1291	0.82	5.76	1.23	21.20	0.42	2.96	0.62	4.82	0.82	7.9	1.38
	12.5SK-2.5-60		2.08	0.13	0.1310	0.89	5.48	0.60	21.00	0.48	3.13	0.81	5.47	0.95	10.57	1.21
	15SK-1-60		1.99	0.27	0.1051	1.25	8.00	1.12	23.85	0.85	2.46	1.25	2.49	1.14	4.34	1.15
	15SK-1.5-60		1.99	0.25	0.1085	1.17	7.76	1.08	23.76	0.74	2.67	1.20	2.70	1.26	4.65	1.23
	15SK-2-60		2.00	0.29	0.1120	1.19	7.55	0.96	23.45	0.86	2.80	1.18	2.87	1.33	5.38	1.01
	15SK-2.5-60		2.00	0.18	0.1156	1.12	7.31	0.91	23.30	0.63	2.86	1.31	3.16	1.29	6.00	0.94

Table 4
Comparison of 7 day compressive strengths of the various types of natural pozzolan-based geopolymer mortars.

Ref	Aluminosilicate source	Chemical composition of pozzolan				Mix parameters				Curing	Compressive strength range at 7 days (MPa)	
		SiO_2 (%)	Al_2O_3 (%)	SiO_2/Al_2O_3 (%)	CaO (%)	Alkali hydroxide concentration	Activator ratio (by weight)	Activator/ pozzolan (by weight)	Sand/ pozzolan ratio (by weight)			
[25]	Jordanian Natural Pozzolan, and oil-shale ash, Jordan	40.17	13.86	2.89	9.70	10–12 M	2.0–2.5	0.3	3.0	24 h at 60 °C	10 M SS/ NaOH = 2.0	17.0
											10 M SS/ NaOH = 2.5	17.5
											12 M SS/ NaOH = 2.0	15.2
											12 M SS/ NaOH = 2.5	15.5
[26]	Natural zeolite, Bigadic Turkey	68.3	10.97	6.22	3.24	8–12 M	1.0–3.0	0.3	3.0	7 days 30 °C	10 M SS/ NaOH = 2.0	~3
											10 M SS/ NaOH = 3.0	~4
											12 M SS/ NaOH = 2.0	~5
											12 M SS/ NaOH = 3.0	~6
[27]	Volcanic tuff, Nevşehir, Turkey	77.22	18.89	4.08	0.27	10–16 M	1.2–2.5	0.35	2.0	24 h at 90 °C	10 M SS/ NaOH = 1.5	9.5–13
											10 M SS/ NaOH = 2.0	13–15
											10 M SS/ NaOH = 2.4	10.5–14
Current Study	**Earth of Datça, Turkey**	75.28	15.99	4.70	1.22	7.5–15 M	1.0–2.5	0.3	3.0	24 h at 90 °C	10 M SS/ KOH = 1.0	4.82
											10 M SS/ KOH = 1.5	5.00
											10 M SS/ KOH = 2.0	5.64
											10 M SS/ KOH = 2.5	6.29
											12.5 M SS/ KOH = 1.0	6.17
											12 M SS/ KOH = 1.5	6.25
											12 M SS/	7.9

(*continued on next page*)

Table 4 (*continued*)

Ref	Aluminosilicate source	Chemical composition of pozzolan				Mix parameters				Curing	Compressive strength range at 7 days (MPa)	
		SiO_2 (%)	Al_2O_3 (%)	SiO_2/Al_2O_3 (%)	CaO (%)	Alkali hydroxide concentration	Activator ratio (by weight)	Activator/ pozzolan (by weight)	Sand/ pozzolan ratio (by weight)			
											KOH = 2.0 12 M SS/ KOH = 2.5	10.57

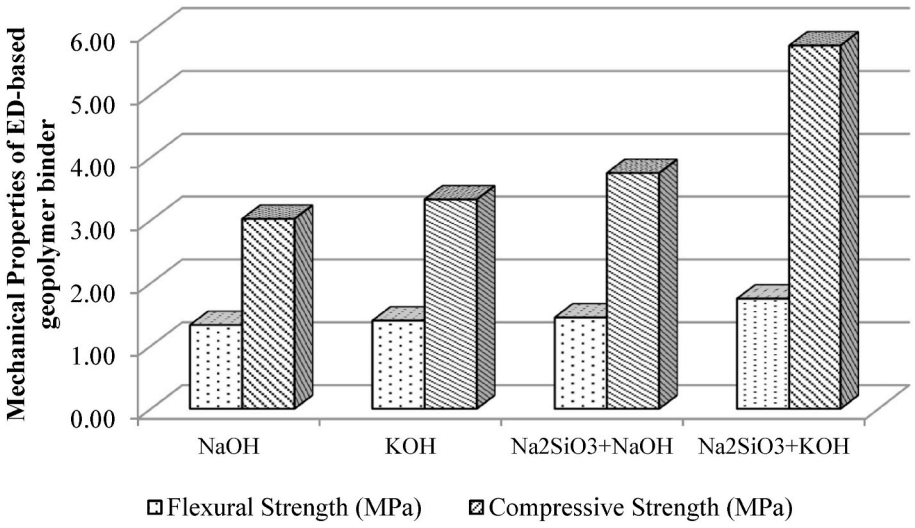

Fig. 4. Mechanical properties of ED-based geopolymer binder activated with various alkaline solutios.

precursors are more easily formed [49]. Thus, the addition of SS into the mixture is important in order to support the development of geopolymerization reactions.

3.3. Influence of curing conditions on the properties of the ED based geopolymer binder

It is well known that mechanical strength of the geopolymer binder is very sensitive to the curing conditions. Curing temperatures between 25 and 90 °C are necessary for geoplymerization reactions [21]. The influence of different curing conditions on the mechanical properties of the ED based geopolymer binder is given in Fig. 5. At 24 h curing duration, when the temperature increases from 70 °C to 90 °C, the compressive strengths of the specimens under unsealed conditions (10 ± 5% RH) increased 1.31 times; while that of the specimens with PE sealing (90 ± 5% RH) increased 1.62 times. Parallel to this, the flexural

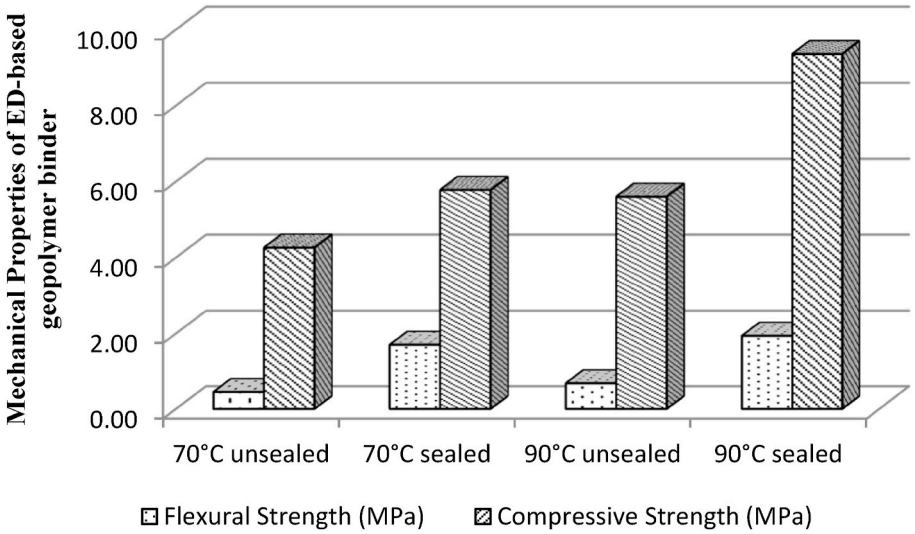

Fig. 5. Mechanical properties of 7.5 M SS + KOH activated ED under different curing conditions.

strengths increased 1.51 and 1.10 times in the specimens cured under unsealed and sealed conditions, respectively. Increasing of the curing temperature increases the dissolution rate of silica and alumina compounds of the solid aluminosilicate source and the rate of polycondensation reactions [12]. On the other hand, under higher temperatures, faster polycondensation and quick formation of geopolymer gel may prevent further dissolution of silica and alumina compounds and this situation has negative effects on the mechanical properties. In addition, under higher temperatures the water content of the specimens evaporates quickly, and this may prevent the completion of geopolymerization [12], and may lead to the formation of shrinkage cracks and loss of strength in the structure of the material [55,56]. Because of this reason, sealed conditions by providing extra humidity may prevent this quick evaporation.

The compressive strengths of the specimens cured at 70 °C and 90 °C inside PE cover are respectively 1.35 and 1.67 times higher than their counterparts cured at unsealed conditions. Similarly, the flexural strengths of the sealed specimens cured at 70 °C and 90 °C are 3.88 and 2.79 times higher respectively, compared to the specimens cured at unsealed conditions. The deterioration of strength under unsealed curing may result from the substantial loss of moisture of the specimens. In addition, the existence of visible cracks, caused by drying shrinkage, on the surface of the specimens cured under unsealed conditions are also in conformity with the lower mechanical strength results. Thus, in order to produce a crack-free geopolymer with good mechanical properties, quick drying should be prevented [56], and heat curing under humidity conditions should be supplied.

When the temperature increased from 70 °C to 90 °C under PE sealed conditions, the ratio of flexural to compressive strength decreased from 1/3.3 to 1/4.9. In addition, when the temperature increased from 70 °C to 90 °C under unsealed conditions, the ratio of flexural to compressive strength decreased from 1/8.2 to 1/9.4. This result reveals that the temperature increase makes the geopolymer binder brittle and more susceptible to fracture. In general, the tensile strength of brittle materials is about 1/10 of their compressive strength [57]. In the current study, aforementioned ratios are higher than 1/10, which means that the specimens show more resistance to crack formation due to various load and impact effects.

3.4. Comparison of ED based geopolymer binder and mortar

The binder requires addition of aggregate in order to be used for the production of a building brick or block. Aggregate is the major component of mortars and referred as the "skeleton" of the mortars' systems [58]. Along with the binder, addition of aggregates brings some advantages like volume stability, durability and strength to the mortar. In the current study, standard sand with a specified granulometry was added to the binder. Sand addition, however, reduces the compressive strength of the geopolymer binder. i.e., compressive strengths of the binder and mortar cured under the same conditions (90 °C, sealed) were found 9.36 and 3.15 MPa, respectively. Three folds decrease of strength may be on account of the thinning effect of sand addition. Similar results were also determined by other researchers [59,60]. Even if it leads to a reduction in strength, addition of sand into the geopolymer binder is important in terms of reducing the amount of binder, and consequently the production cost, as long as the material meets the performance criteria expected from its function. In the current study the pozzolan/sand ratio by weight of the mortar was kept constant as 1/3; therefore, the effects of various pozzolan/sand ratios on the mechanical properties of the mortar needs further investigation.

3.5. Influence of alkali hydroxide concentration on the properties of the ED based geopolymer mortar

The influence of the alkali hydroxide concentration on the physical and mechanical properties of SS + KOH activated geopolymer mortar

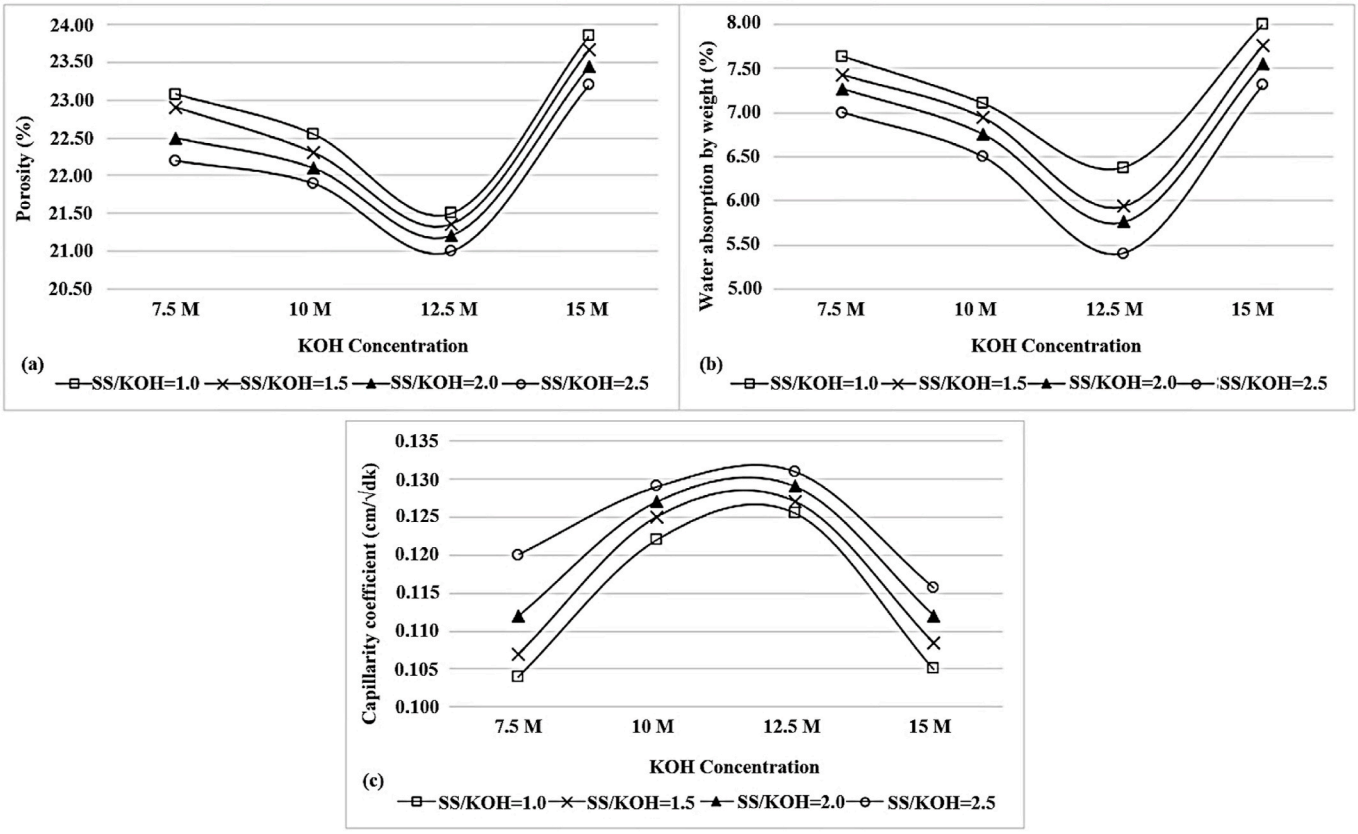

Fig. 6. Influence of KOH concentration on the physical properties of the specimens having different activator ratios.

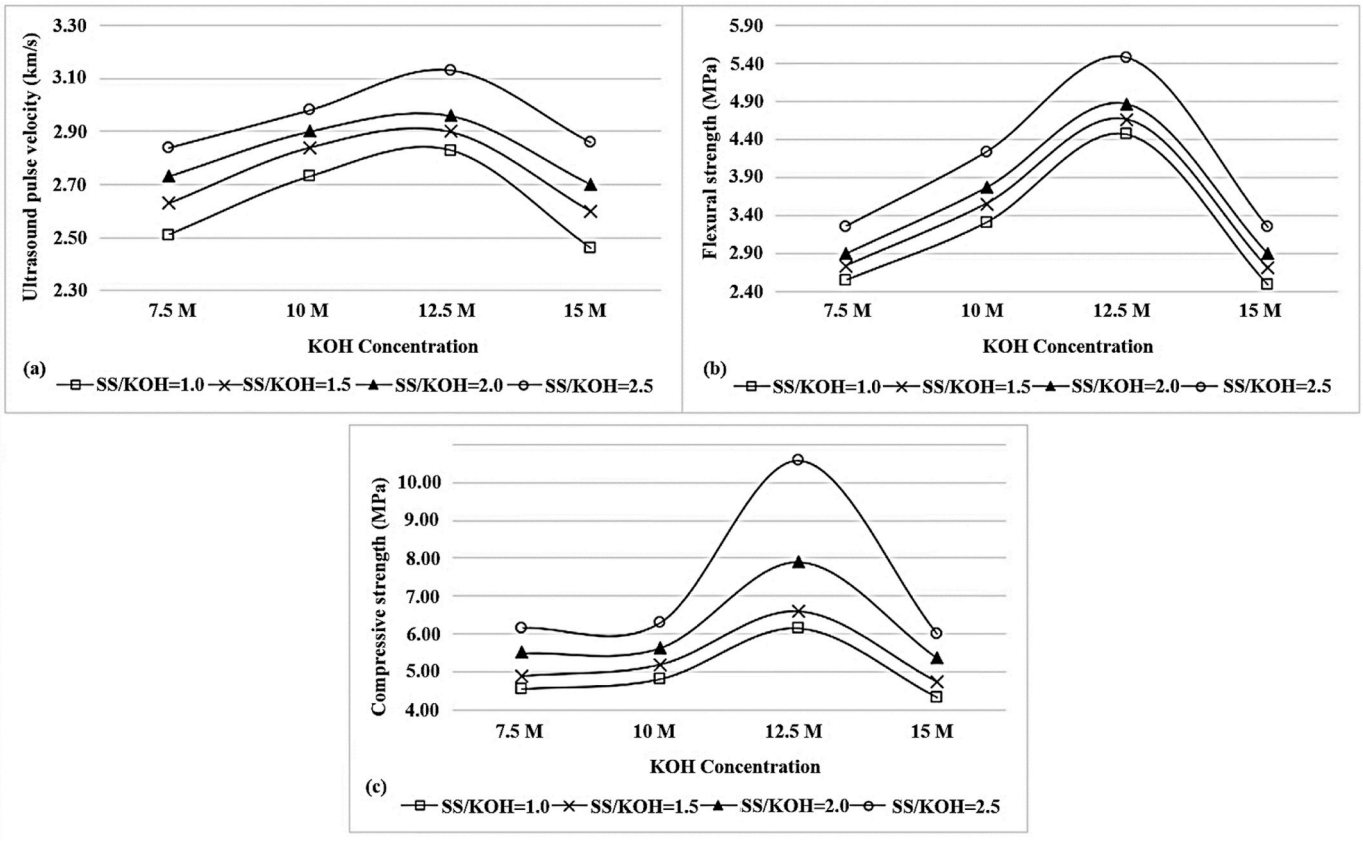

Fig. 7. Influence of KOH concentration on the mechanical properties of the specimens having different activator ratios.

are given in Fig. 6 and Fig. 7. About 12.5 M KOH there was reversal of properties towards observed. When activator ratio was kept constant, and KOH molarity was increased from 7.5 M to 10 M and 12.5 M, these changes resulted in a decrease in the porosity and water absorption ratios of the samples. The lowest porosity (21.00%) and water absorption ratio (5.48%) were observed in the specimens having 12.5 M KOH concentration, and activator ratio of 2.5. Ascending values of capillarity coefficients revealed that the internal pores of the mortar were narrow and better connected, and consequently, the capillary pressure was high. The aforementioned changes in the physical properties indicate the structural improvement of the mortar. The properties of ultrasound velocity, compressive, and flexural strengths of the specimens increased depending on the increase of KOH molarity. Especially, the rate of increase in mechanical properties of the specimens activated with activator ratio of 2.5 peaked at 12.5 M KOH concentration and had the highest ultrasound pulse velocity (3.13 km/s), compressive (10.57 MPa) and flexural strengths (5.47 MPa). This improvement may be explained by the enhancing dissolution of the aluminosilicate source (Earth of Datça) with the increasing OH^- concentration [61] and formation of higher amount of dissolved Si and Al compounds in the reaction medium for the development of following polycondensation reactions [62]. So, in the current study, activating Earth of Datça with 12.5 M KOH solution leads to the higher extent of dissolution of the aluminosilicate resulting in less porous and permeable geopolymer structure as well as higher mechanical properties.

When activator ratio was kept constant, the increase in the KOH molarity from 12.5 M to 15 M results in an increase in the porosity and water absorption ratios of the samples. The highest porosity (23.85%) and water absorption ratio (8.00%) were observed in the specimens with 15 M KOH concentration and activator ratio of 1.0. A decrease in the capillarity coefficient was an indication that the internal pores of the mortar become larger, giving rise to lower capillary pressure. The aforementioned changes in the physical properties indicate the structural deformation of the mortar. The mechanical properties of the 15 M KOH-activated samples decreased to a point at which they were lower than those of the 7.5, 10, and 12.5 M KOH-activated samples. The lowest ultrasound pulse velocity (2.46 km/s), compressive (4.34 MPa) and flexural strengths (2.49 MPa) were detected with the 15 M KOH-activated samples where activator ratio is 1.0. Hence, when the alkali hydroxide concentration was increased more than its optimum level, the physical and mechanical properties were affected adversely, causing immature molecular structure. This result may be explained by the existence of excess OH^- in the reaction medium which brings about faster and sooner polycondensation. Thus, the development of the dissolution reactions may have been prevented and the aluminosilicate source can not have enough time for dissolution [61].

3.6. Influence of activator ratio on the properties of the ED based geopolymer mortar

The influence of the activator ratio on the physical and mechanical properties of SS + KOH activated geopolymer mortar are given in Fig. 8 and Fig. 9. It has been observed that when the KOH concentration was kept constant, and activator ratio was increased from 1.0 to 1.5, 2.0, and 2.5, this led to a slight decrease in the porosity and the water absorption ratio, and a slight increase in capillarity coefficient values of the samples. The lowest porosity (21.00%) and water absorption ratio (5.48%) and the highest capillarity coefficient (0.1310 cm/$\sqrt{}$dk) were observed in the specimens having activator ratio of 2.5. Although the change in physical properties is not very significant, the increasing ratio of the mechanical properties is remarkable, especially in the specimens having 12.5 M KOH concentration and activator ratio of 2.5. The samples activated with activator ratio of 2.5 (when KOH molarity is 12.5 M) had the highest ultrasound pulse velocity (3.13 km/s), compressive (10.57

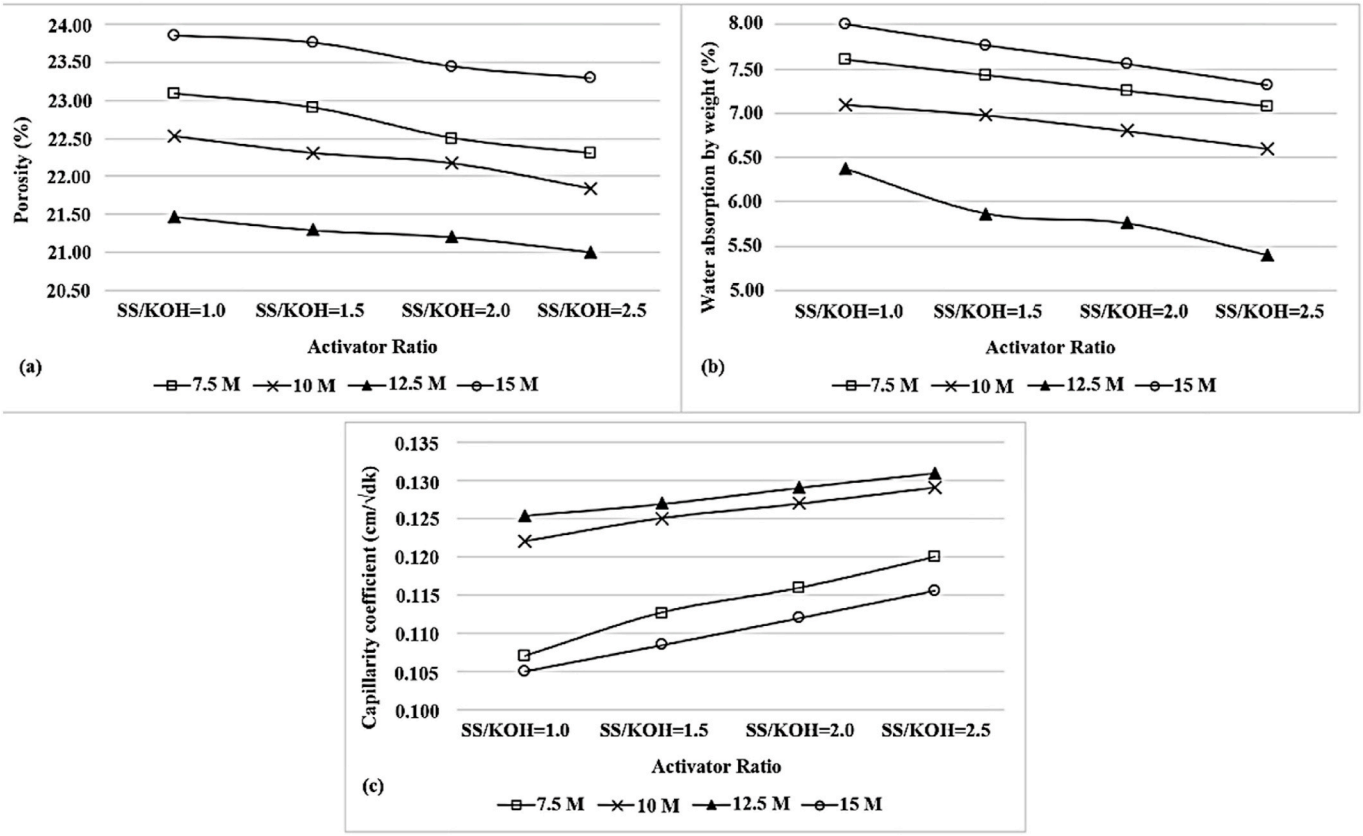

Fig. 8. Influence of activator ratio on the physical properties of the specimens having different KOH concentrations.

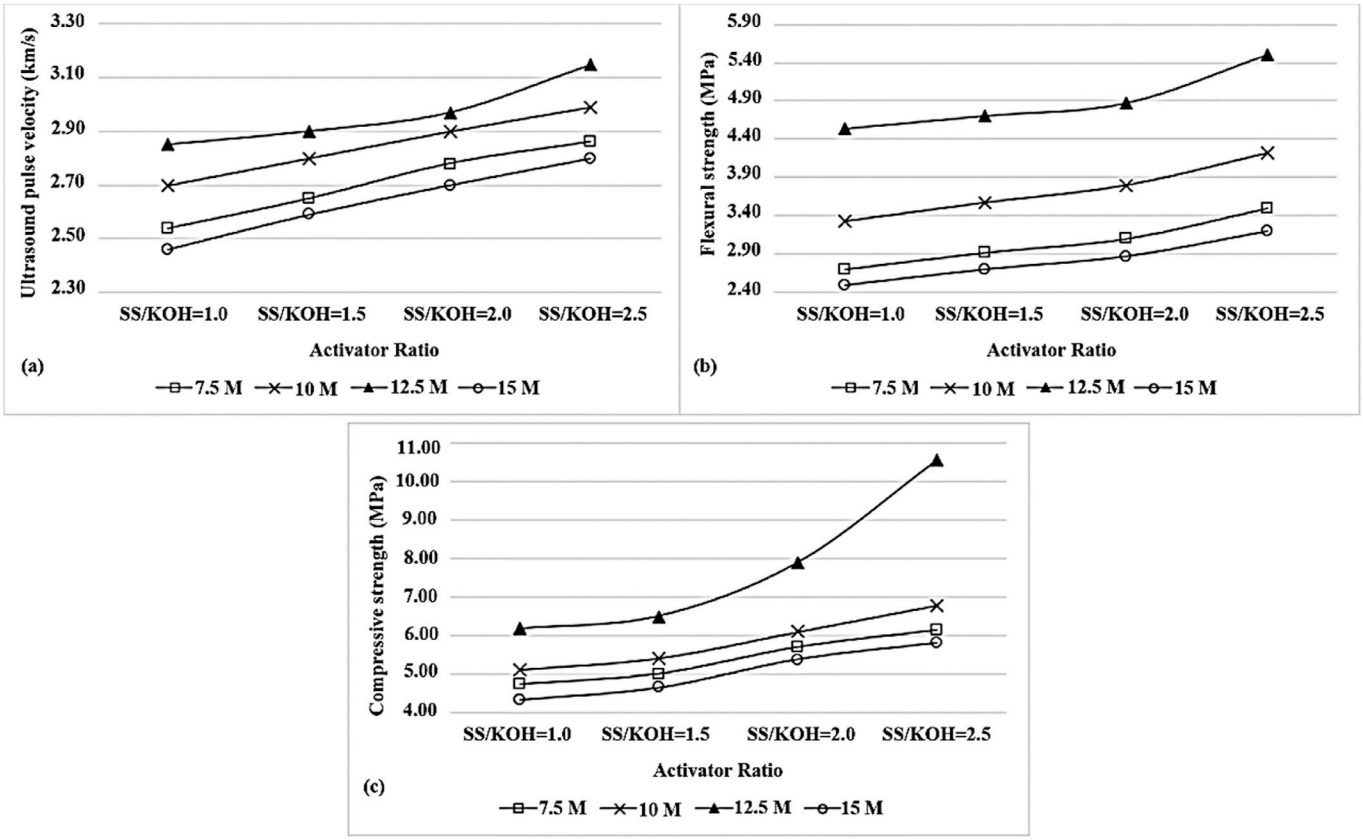

Fig. 9. Influence of activator ratio on the mechanical properties of the specimens having different KOH concentrations.

MPa) and flexural strengths (5.47 MPa). The reason of this increment in the mechanical properties owing to the addition of SS was explained in Section 3.2. The higher the activator ratio is, the higher the physical and mechanical properties of ED-based geopolymer mortar becomes. However, care must be taken to regulate the ratio between them. If more SS is added, the mentioned properties may decrease since excess SS may prevent the evaporation of water in the reaction medium and may hinder the structure formation of the material [63]. Hence in the current study, the optimum activator ratio was found to be at 2.5. However, the optimum activator ratios may fluctuate depending on the various types of the raw materials, alkali solutions, and alkali/binder ratios. E.g. the optimum activator ratio is 2.5 for the combination of fly ash and Pasuruan Trass [64], 2.0 for the geopolymer binder and concrete produced from granulated blast furnace slag [65] and 1.5 for the fly ash based geopolymer concrete [66].

The compressive strengths of ED-based geopolymer mortars are compared with those of mortars made with various natural aluminosilicate sources and given in Table 4. Taking into consideration the factors given in Table 4 such as chemical composition, alkali hydroxide concentration, activator ratio, activator/pozzolan, sand/pozzolan ratios, and curing conditions, the compressive strength range of geopolymer mortars produced from Natural zeolite of Bigadic [26] and volcanic tuff of Nevşehir [27] shows close values to that of ED-based mixtures. However, the compressive strength of Jordanian Natural Pozzolan-based geopolymer mortar [25] is approximately 1.60 times higher than that of ED-based geopolymer mortar, which may be result of lower SiO_2/Al_2O_3 ratio (2.9) compared to that of ED (4.70). On the other hand, there are other factors whose efficacy on geopolymeric reactivity was not evaluated in this study such as: particle shape, particle size, particle size distribution; glass content, glassy phase of pozzolanic raw materials [67,68]. All of these factors are interrelated and may not be considered individually when a specific raw material is studied [43].

4. Conclusions

Following conclusive remarks can be stated from the obtained results:

- ED has lower calcium content (1.22) and comparatively higher SiO_2/Al_2O_3 ratio (4.70) than other natural pozzolans given in literature; its properties as a geopolymer precursor can be enhanced by mixing it with suitable alkali activators.
- The physical and mechanical characteristics of the tested ED based geopolymer gradually increased with the increase of curing temperature from 70 °C to 90 °C. Hence, 90 °C can be suggested.
- Sealed curing at 90 ± 5% RH provides higher strength results, whereas, curing at lower relative humidity around 10% caused visible cracks on the surface of the specimens due to drying shrinkage. Thus, humidity conditions during curing should be provided.
- A combination of SS solution with KOH is selected as the optimum activator type for ED-based geopolymer binder due to the highest gain in mechanical characteristics.
- Sand addition (pozzolan/sand ratio = 1/3, by weight) leads to a decrease in the mechanical properties of the binder due to its thinning effect. However, sand addition is important in terms of reducing the cost of the mixture as long as the material meets the performance criteria expected from its function.
- Compressive strengths up to 10.5 MPa can be achieved by controlling the parameters such as the alkali activator type, alkali hydroxide concentration, activator ratio, and curing conditions.
- Physical and mechanical properties of the ED-based geopolymer mortar increase with the KOH concentration, peaking at 12.5 M and declining thereafter. Using of 12.5 M KOH solution together with activator ratio of 2.5 leads to the greatest amount of dissolution of the precursor, which is important for the following polycondensation, and less porous and permeable matrix formation together with the highest mechanical properties.
- The higher the activator ratio, the higher is the physical and mechanical properties of ED-based geopolymer mortar. When the economic and strength properties are considered together, activation the Earth of Datça with 12.5 M KOH concentration and activator ratio of 2.5 is seen as an optimum choice, without compromising the performance criteria expected from the material. Trying to produce a high quality geopolymer by increasing the alkali concentration and the activator ratio above a certain level is not recommended when material properties and its economical aspects are taken into account.
- The mortar produced in this research has promising characteristics to be used as a building material. On the other hand, factors such as particle shape, particle size, particle size distribution, glass content, glassy phase of the pozzolanic raw material which were not evaluated in this study should be undertaken for further research.

Author statement

Kübra Ekiz Barış: Corresponding Author, Conceptualization, Design of methodology, Investigation, Experimental research, Preparation and creation of the published work, Data presentation, Writing.

Leyla Tanaçan: Conceptualization, Design of methodology, Preparation and creation of the published work, Reviewing and Editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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