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Earth of Datça: Development of pozzolanic activity with steam curing

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HIGHLIGHTS

- Earth of Datça-lime binder gained high strength in a short time with steam curing.
- Materials having low environmental impact were produced by steam curing.
- Steam curing provided more energy-efficiency and saving of time than the other methods.
- Earth of Datça-lime binder with steam curing may be used as 'bricks or blocks'.
- Earth of Datça-lime binder without steam curing may be used as 'mortar ingredient'.

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ABSTRACT

Environment friendly binders with relatively low energy consumption and low emissions may be an alternative to energy-intensive cement. To contribute to this knowledge, physical and mechanical characteristics of Earth of Datça-lime binder under steam curing conditions at atmospheric pressure was investigated. To evaluate the effects of the steam curing conditions, air, water, and heating curing were also examined. Tests results showed that Earth of Datça-lime binder through steam curing can be used for the production of materials similar to fired clay or calcium silicate bricks. Steam curing provided more strength, energy-efficiency and time saving compared to other curing conditions.

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1. Introduction

As an industrial material, cement is manufactured intensively in building trade. Cement production significantly consumes natural resources and causes CO_2 emissions. CO_2 emissions represent approximately 5% of anthropogenic global CO_2 emissions [1]. Thus, there is an urgent need to produce alternative binders which are more environment friendly with lower energy consumption and lower CO_2 emissions instead of energy-intensive cement.

Lime-pozzolan is a binder with hydraulic properties mainly consisting of air lime with added pozzolanic material. Addition of a pozzolan to air lime enables the mortars to obtain hydraulic properties, resistance to water, higher compressive strength, but also durability and some resistance to weathering [2]. So, lime-pozzolan binder can be used as an alternative to the cement for both structural and non-structural purposes in modern and historical buildings. In addition, it is possible to use them in the production of alternative bricks to the cement-based or calcium silicate bricks.

Mechanical properties of lime-pozzolan binders increase slower than that of cement, depending on the type of the pozzolan used [2]. In order to obtain satisfactory strength in an acceptable time period heating curing is usually necessary [3]. The objective of curing is to provide appropriate temperature and humidity conditions to ensure the progress of hydration reactions causing the filling and segmentation of capillary voids by hydration products [4]. The progress of hydration reaction, which influences the binders' porosity, microstructure and strength, are directly connected to the curing conditions used [5]. Except for heating curing, diverse curing methods are used comprising steam curing at atmospheric pressure and high pressure steam curing. The primary objective of steam curing is to obtain a sufficiently high early strength so that the products may be handled soon after casting: the moulds can be removed, or the prestressing be vacated earlier than would be the case with ordinary moist curing, and less curing storage space is required; all these mean an economic advantage. When steam is at atmospheric pressure, i.e. the temperature is below 100 °C, the process can be regarded as a special case of moist

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Table 1
Chemical analysis of the main components of the hydrated lime, Earth of Datça, and Earth of Datça-lime binder.

Component	Hydrated Lime (%)	Earth of Datça (%)	Earth of Datça-lime binder (%)
SiO_2	1	72.07	49.20
Al_2O_3		13.06	7.90
Fe_2O_3		1.39	1.25
MgO	1	0.44	0.61
CaO	85	2.34	29.46
CO_2	5	–	–
Na_2O		3.27	1.66
SO_3	0.6	–	–
K_2O		4.60	3.04
TiO_2		0.20	0.25
P_2O_5		0.05	0.05
MnO		0.06	0.06
Cl		0.10	0.13
S		218PPM	503PPM
LOI		2.15	6.01
Total	92.6	99.73	99.63

curing in which the vapour saturated atmosphere ensures supply of water [6]. However, when the pressure is above atmospheric level, the curing method is called as high pressure steam curing or autoclaving, which is extensively used for the manufacturing of cement-based building products such as dense concretes, lightweight or aerated concretes and calcium silicate bricks. To obtain such products, high temperatures (170–210 °C) are necessary in order to impart to the silica the necessary reactivity towards lime [7]; it means higher consumptions of energy. But, there is no need high amounts of curing energy for lime-pozzolan products due to the fact that amorphous silica (and/or alumina) of pozzolan is more reactive than sand towards lime.

In a previous study [8], three types of curing methods (air curing, water curing and heating curing) was compared on the development of pozzolanic activity of volcanic tuff settled at the far west end of the Datça Peninsula in Turkey. It was determined that physical and mechanical characteristics of Earth of Datça-lime binder developed considerably with water and heating curing individually. However, the effects of both water and heating curing on this binder were not evaluated together. The purpose of the present study is to investigate the development of physical and mechanical characteristics of Earth of Datça-lime binder under steam curing conditions at atmospheric pressure which contains both of water and heating effects together. In this context, the objectives of the study can be summarized as:

- To produce a hydraulic binder as an alternative to cement;
- To obtain satisfactory strength in an acceptable period of time;
- To shorten the production time and consequently to minimize the energy consumption under the constant temperature;
- To investigate the possibility of preparing products with this binder similar to 'bricks'.

2. Materials and methods

Materials: The pozzolanic activity of Earth of Datça (ED) was examined according to the Turkish Standard 25 [9]. Quicklime was slaked in order to obtain calcium hydrated lime putty complying with TS EN 459-1 [10] and stored in the moist state by covering it for minimum 15 days until used. CEN standard sand based on TS EN 196-1 [11] was used. ED was put in an oven at 70 °C and dried until constant weight was determined. Afterwards, ED was milled by a tumbling ball mill. Maximum grain size (fineness) of the pozzolan was determined as 90 μm as per Turkish Standard 25 [9] and was measured by sieving it on standard sieves according to the TS EN 196-6 [12]. Table 1 reports the chemical characterization (XRF) and Fig. 1 presents mineralogical analysis (X-ray diffraction_XRD) of the hydrated lime, Earth of Datça, and Earth of Datça-lime binder (ED)B produced for the pozzolanic activity test. The mixture proportion of lime, pozzolan and sand is 1:2.3:9 by weight. XRF and

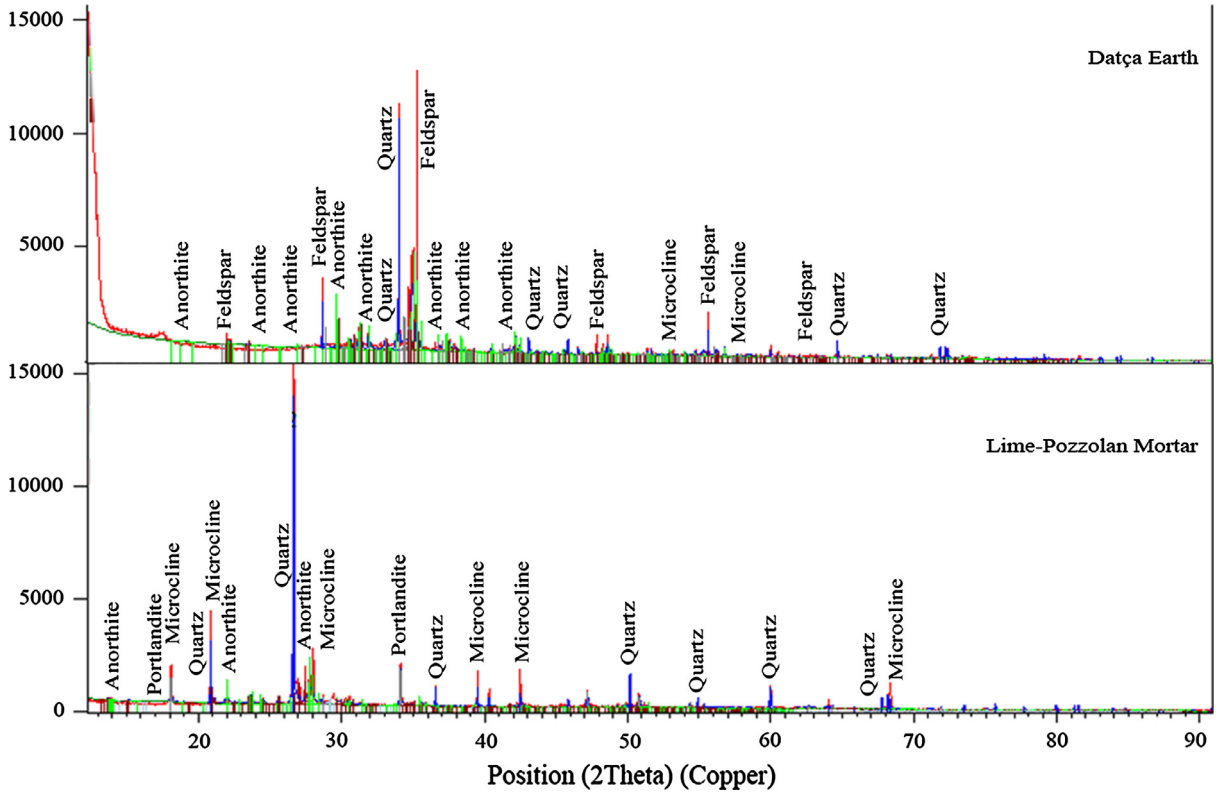


Fig. 1. Results of XRD in Earth of Datça, and Earth of Datça-lime binder.

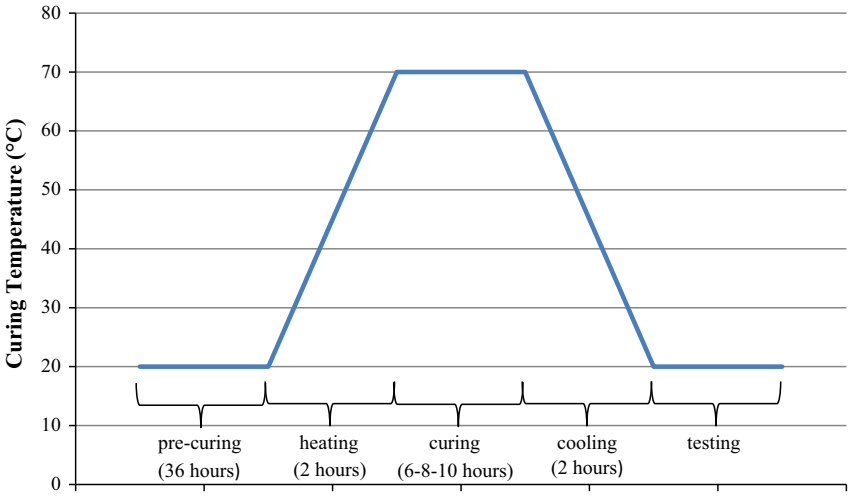


Fig. 2. Steam-curing cycle of the specimens cured at 70 °C.

Table 2
Properties of Earth of Datça-lime binder.

Curing Conditions		Unit weight (g/cm^3)	Capillarity coefficient (cm^2/s)	Water absorption by weight (%)	Apparent porosity (%)	Porosity (%)	Compactness (%)	Ultrasound Velocity (km/s)	Flexural Strength (MPa)	Compressive Strength (MPa)	Young's Modulus of Elasticity (MPa)
Air Curing	A-35/20/7	1.39	1.29×10^{-3}	24.05	33.42	42.57	57.43	1.43	0.82	5.74	2840
	A-35/20/28	1.41	0.95×10^{-3}	21.36	30.11	39.25	60.75	1.50	1.00	6.60	3170
	A-35/20/90	1.36	0.80×10^{-3}	27.26	37.07	45.18	54.82	1.40	0.42	4.72	2660
Water Curing	W-35/20/7	1.43	0.84×10^{-3}	19.59	28.01	41.16	58.84	2.16	1.61	6.68	6670
	W-35/20/28	1.47	1.10×10^{-3}	16.26	23.90	38.22	61.78	2.66	2.19	10.91	10400
	W-35/20/90	1.56	1.41×10^{-3}	9.60	14.97	28.37	71.63	3.61	2.45	13.11	20330
Heating Curing	H-35/70/7	1.36	0.51×10^{-3}	21.40	29.10	42.38	57.62	2.33	1.01	9.8	7380
	H-35/90/7	1.35	0.38×10^{-3}	24.80	33.48	43.62	56.38	1.79	0.80	8.52	4320
Steam Curing at Atmospheric Pressure	S-35/70/6	1.49	1.12×10^{-3}	13.92	20.74	38.94	61.06	2.40	1.47	10.11	8580
	S-35/70/8	1.41	0.90×10^{-3}	19.68	27.74	41.71	58.29	2.15	0.86	9.45	6510
	S-35/70/10	1.39	0.73×10^{-3}	21.32	29.63	42.29	57.71	2.05	0.51	8.79	5840
	S-35/90/6	1.39	0.80×10^{-3}	18.72	26.02	39.16	60.84	2.02	1.00	8.52	5670
	S-35/90/8	1.31	0.66×10^{-3}	23.77	31.13	45.05	54.95	1.35	0.51	8.00	2380
	S-35/90/10	1.29	0.52×10^{-3}	25.64	33.07	46.32	53.68	1.33	0.30	7.57	2280

XRD were performed on dry, grinded and sieved samples with a cumulative passing percentage at 45 μm of 100%. XRF of the samples was measured by using Bruker-S8 Tiger model XRF equipment. XRD of the samples was conducted using D8-Advance Bruker-XRD equipment with Cu X-ray tube (1.5406 Angstrom). According to the XRD data, anorthite, quartz, feldspar and microcline are observed in ED and the binder (ED)B contains anorthite, quartz, portlandite and microcline.

Total amount of $SiO_2 + Al_2O_3 + Fe_2O_3$ (86.52%) and the chemical limitations for MgO (0.44%), and SO_3 (0%) of pozzolan fit well the requirements of TS 25. The specific surface area of the pozzolan measured with Blaine method according to the TS EN 196-6 [12] (5278.71 cm^2/g) is greater than the required 4000 ± 25% cm^2/g. The flexural and compressive strength test results of pozzolanic activity, 1.43 and 6.12 MPa, respectively, are also in conformity with the standard [9] requirements.

Test Methods: Physical and mechanical characteristics of (ED)B were investigated under the steam curing at atmospheric pressure. The binder consisted of hydrated lime and pozzolan with cumulative passing percentage at 200 μm of 100%. Sand was not added into the mixture.

The ratio of lime/pozzolan was kept 35% by weight for all series. The water content was determined by flow table test according to ASTM-C593-06 [13]. The binder was moulded in prismatic 40 × 40 × 160 mm casts and coded in [X-XX/XX/XX] format. The first letter represents curing condition (A:air curing, W:water curing, H:heating curing, S: steam curing at atmospheric pressure), the second letter group points out the mixture proportion of lime/pozzolan, the third letter group indicates the curing temperature and the fourth letter group indicates the curing time.

To find out the effects of steam curing conditions at atmospheric pressure, the freshly moulded samples were placed inside polyethylene bags and pre-cured at the temperature of 20 ± 2 °C and 95 ± 5% RH for 36 h. After this initial curing; the samples were demoulded and cured at 70 °C and 90 °C and under saturated steam in a closed container named Kerman for 6, 8, and 10 h. The rate of the temperature rise of the container was arranged 35 °C/h and curing cycle at 70 °C was given in Fig. 2.

In order to evaluate the effects of the steam curing at atmospheric pressure compared to other curing conditions (air curing, water curing and heating curing) on the physical and mechanical characteristics of Earth of Datça-lime binder, these curing

conditions were also included in the experimental program. The curing conditions employed were: A – Air curing where the binders were pre-cured at 20 ± 2 °C and 95 ± 5% RH in moulds for 7 days, and then cured at 20 ± 2 °C and 50 ± 5% RH without moulds until 28th and 90th days; W – Water curing where the specimens were pre-cured at 20 ± 2 °C and 95 ± 5% RH in moulds for 7 days, and then immersed in water at 20 ± 2 °C without moulds until the testing day at 7th, 28th, and 90th days; H – Heating curing with pre-curing at 20 ± 2 °C and 95 ± 5% RH in moulds for 36 h and after, curing in an oven at 70 °C and 90 °C and 95 ± 5% RH without moulds for 6 days. The specimens of air, heating and steam curing were tested directly without drying, while the specimens of water curing were dried for 24 h at 50 °C prior to tests.

The specimens were tested for unit weight according to the TS EN 1015-10 [14], capillary water absorption according to the TS EN 1925 [15], water absorption under the atmospheric pressure according to the TS EN 13755 [16], dynamic ultrasound velocity/Young's modulus of elasticity according to the TS EN 14579

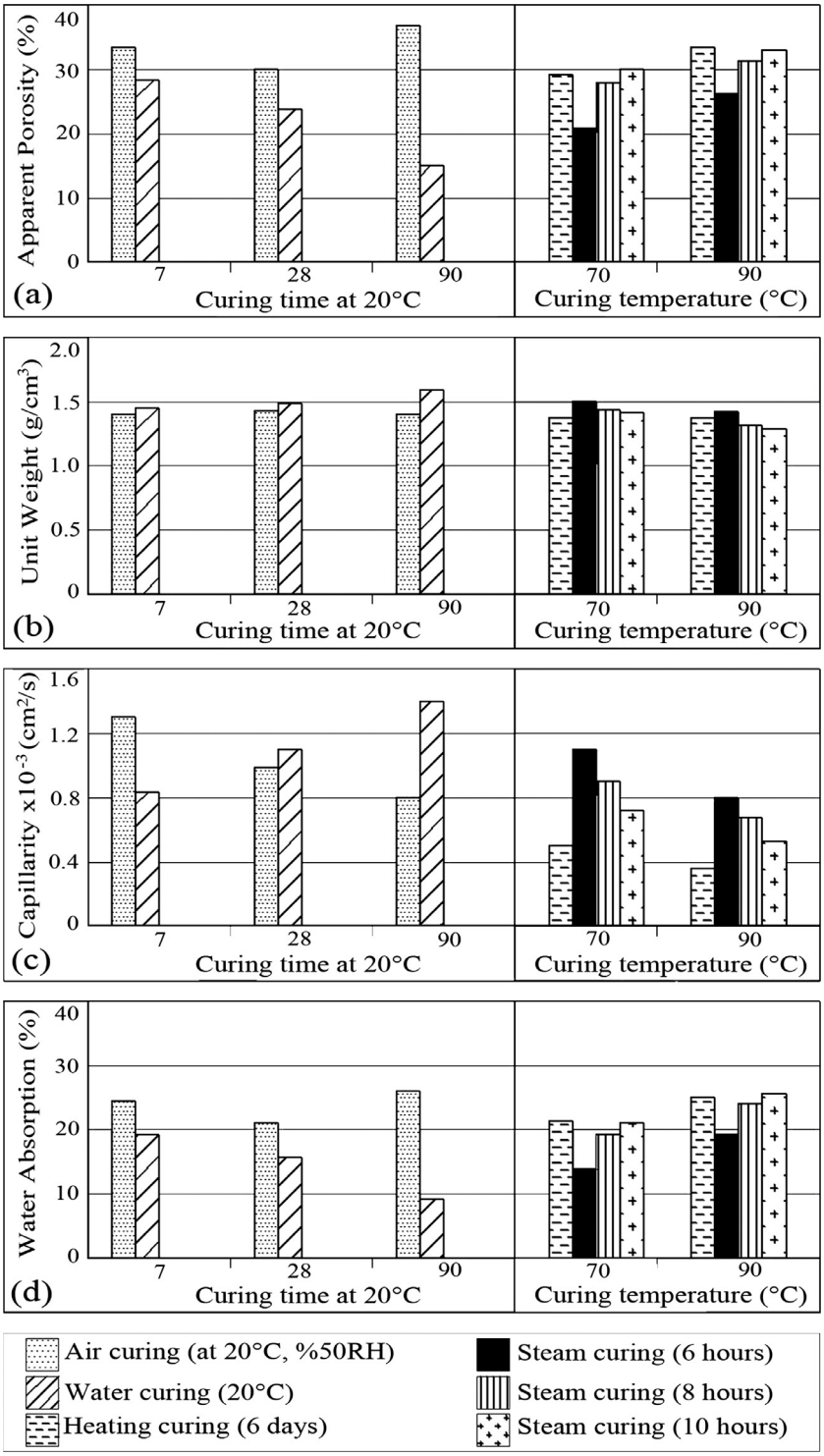


Fig. 3. Comparisons of the physical properties of binders cured at different conditions: (a) apparent porosity; (b) unit weight; (c) capillarity coefficient; (d) water absorption by weight.

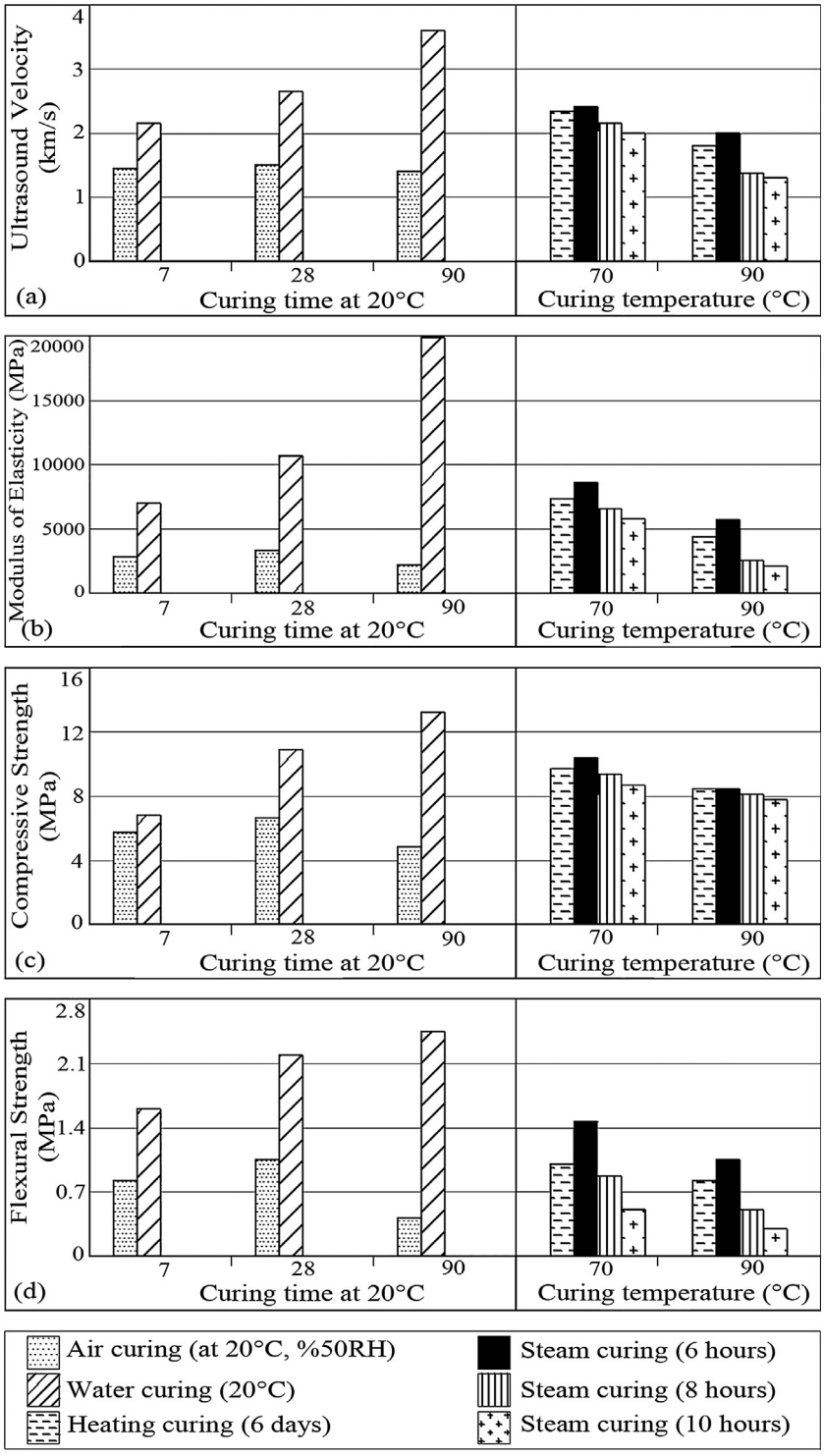


Fig. 4. Comparisons of the mechanical properties of binders cured at different conditions: (a) ultrasound velocity; (b) modulus of elasticity; (c) compressive strength; (d) flexural strength.

[17], flexural and compressive strengths according to the TS EN 196-1 [11].

3. Results and discussion

3.1. Influence of curing conditions on the characteristics of the binder

Curing conditions were examined so as to evaluate their influence on the development of the physical and mechanical properties of (ED)B. The properties presented in Table 2 are averages of three specimens.

After 90 days of water curing, apparent porosity of the specimens decrease 0.50 times on average compared to 7th days (Fig. 3a). Depending on the decreasing of the porosity, while the unit weight and capillarity coefficient increase 1.1 and 1.67 times (Fig. 3b and c), respectively, the water absorption decreases 0.50 times on average (Fig. 3d). The increasing values of capillarity coefficient mean that the internal pores of the binder are narrow

(10–0.005 μm) and better connected; and consequently, the capillary pressure is high. This denser microstructure presents that the water curing enhance the formation of hydrated products. The ascending values obtained by unit weight and descending values obtained by porosity and water absorption indicate the structural improvement of the binder. The ultrasound velocity, the modulus of elasticity, the compressive, and the flexural strength of the water curing specimens increase 1.67, 3.04, 1.96 and 1.52 times, respectively (Fig. 4a –d), with respect to the values obtained after 7 days. The increasing of mechanical strength conforms with the improvement of pore structure and is derived from the formation of hydrated calcium silicates and calcium aluminates which are produced from the pozzolanic reactions [8,18].

After 28 days of air curing, apparent porosity of the binder decreases 0.90 times (Fig. 3a) and unit weight increases 1.01 times compared to 7th days (Fig. 3b). In addition, capillarity coefficient

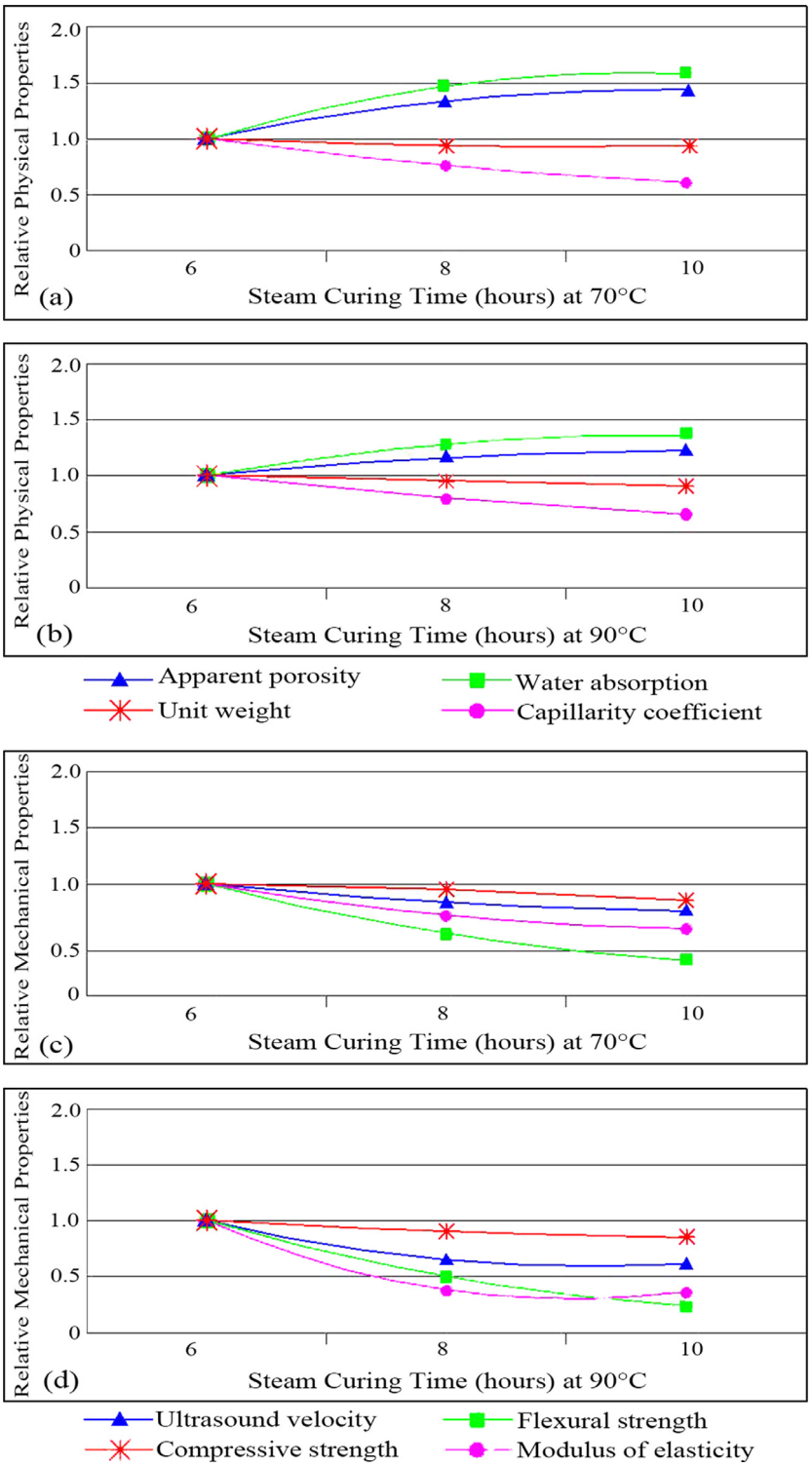


Fig. 5. Relative properties as function of steam curing time: (a) physical properties at 70 °C; (b) physical properties at 90 °C; (c) mechanical properties at 70 °C; (d) mechanical properties at 90 °C.

and water absorption decrease approximately 0.70 and 0.90 times, respectively (Fig. 3c and d). While the ultrasound velocity remains almost same (Fig. 4a), the modulus of elasticity, the compressive and flexural strengths of the specimens increase 1.11, 1.14 and 1.21 times, respectively (Fig. 4b–d). The evolution of the properties until 28th days show that (ED)B already have enough humidity for the development of successfully hydration reactions. However, after 90 days of air curing, cracks develop in the structure of the binder due to the shrinkage. Because of the cracks, porosity and water absorption increase 1.23 and 1.27 times, whereas the unit weight and capillarity coefficient decrease 0.96 and 0.84 times, respectively, compared to 28th days results. Furthermore, the ultrasound velocities of some specimens could not be measured owing to the failures; the compressive and flexural strength decrease correspondingly. It is clear that the effect of the humidity during curing is a major consideration that cannot be ignored [19]. Hence, other researchers [5,20–24] notified that physical and mechanical characteristics of the lime–pozzolan binders did not show any improvement when the binders were exposed to dry curing conditions for a long time. There are several factors that affect the sensitivity of pozzolanic binders to dry curing conditions such as instability of hydrated calcium silicates and calcium aluminates due to dehydration at low humidity, microcracks derived from the shrinkage and deterioration of the contiguity of micro components of binders [2].

When the temperature increases from 70 °C to 90 °C in heating curing, the apparent porosity increases in parallel with the water absorption. However, although the ascending values of the apparent porosity (Fig. 3a), the descending values of the capillarity coefficient (0.74 times) (Fig. 3c) reveal that internal pore size of the binder may not decrease. It is well known that ultimate strength of the lime-pozzolan pastes is very sensitive to the curing temperature. The higher the curing temperature, the lower the ultimate compressive strength of lime-pozzolan pastes [25]. Due to the increasing of curing temperature, inversely proportional to the porosity, the ultrasound velocity, the modulus of elasticity, the compressive and the flexural strength decrease 0.76, 0.58, 0.86 and 0.79 times respectively (Fig. 4a–d). The decreasing of mechanical strength may result from the broken down of bound-water in hydrated calcium silicates and calcium aluminates after 95 °C, which means that the matrix is destroyed [8].

Although porosity and unit weight values of 7th days air curing (at 20 ± 2 °C and 50 ± 5% RH) and heating curing specimens (at 90 °C and 95 ± 5% RH) are almost same, the capillarity coefficient of the air curing ones are approximately 3.4 times more than those of heating curing. In other words, the total pore volume of the binder may remain constant, but, the size of the pores may expand. Thus, it can be concluded that heating curing at 90 °C can not contribute to the hydration process compared to air curing during the same period.

3.2. Steam curing at atmospheric pressure

Two essential parameters should be taken into account to evaluate steam curing conditions at atmospheric pressure: curing time and temperature. Firstly, the effects of curing time on the development of the physical and mechanical characteristics of Earth of Datça-lime binder were tested. When the curing time increases from 6 to 10 h at both 70 °C and 90 °C, while the apparent porosity and water absorption of the all specimens increase, unit weight and capillarity coefficient decrease as expected (Fig. 5a and b). The rate of the fall is relatively high between 6 and 8 h of steam curing and gradually decreases towards 10 h. Concerning mechanical properties, the ultrasound velocity, the modulus of elasticity, the compressive and the flexural strength decrease correspondingly (Fig. 5c and d). The decreasing values of the properties shows that a curing period longer than optimum (6 h) may not contribute to any improvement in the pore structure of (ED)B. The reason of this, continued steam curing after completion of the reaction between calcium and silicate may lead to a variation of the crystallinity of calcium silicate hydrates, but, may not lead to any refinement of the pore size of binders [26]. Thus, 6 h steam curing at atmospheric pressure is seen as optimum period for (ED)B both at 70 °C and 90 °C.

When the steam curing temperature increases from 70 °C to 90 °C for 6 h, the apparent porosity and accordingly water absorption of the specimens increase 1.25 and 1.34 times (Fig. 3a and d); on the other hand, the unit weight and capillarity coefficient decrease 0.93 and 0.71 times, respectively (Fig. 3b and c). In a study regarding with the low temperature steam curing of pozzolan-lime binder [27], it is stated that temperature increase from 75 °C to 90 °C gives rise to an increase in total porosity and a shift of the pore sizes towards the coarser size classes. These results, however, cannot be extrapolated to temperatures higher than 100 °C, since, during the high pressure steam treatment, the reaction between lime and the silica contained in the pozzolana could follow a different mechanism [7]. The evolution of the physical properties adversely affect mechanical properties; so, the ultrasound velocity, the modulus of elasticity, the compressive, and the flexural strength of the steam curing specimens decrease 0.84, 0.66, 0.84 and 0.68 times, respectively (Fig. 4a–d).

At 70 °C, the compressive strength of the specimens obtained after steam curing for 6 h is 1.04 times higher than that obtained after heating curing for 6 days. In terms of electricity consumption, steam curing for 6 h (86.4 MJ) is approximately 20 times lower than that of heating curing for 6 days (1710 MJ). Thus, it may be concluded that steam curing at atmospheric pressure is more energy-efficient and time saving in comparison to heating curing.

Compressive strength of the specimens cured 28 days in water is 1.07 times higher than that of steam curing specimens at 70 °C for 6 h. That is, although the steam curing consumes energy during treatment, its curing time is shorter; thus saves time.

Correlation between flexural (F_f) and compressive strengths (F_c) of the specimens after water and steam curing (at 70 °C) is given in Fig. 6. This correlation for the specimens of water curing is:

$$F_f = 0.5022 \times (F_c)^{0.6164} \quad (1)$$

The correlation factor of 0.99 shows high level of conformity with the previous study [8]. The relation obtained for steam curing specimens is:

$$F_f = 4E-08 \times (F_c)^{7.539} \quad (2)$$

and the correlation factor is 0.98.

Table 3 presents a comparison of the specimens produced under steam curing at atmospheric pressure at 70 °C for 6 h with fired

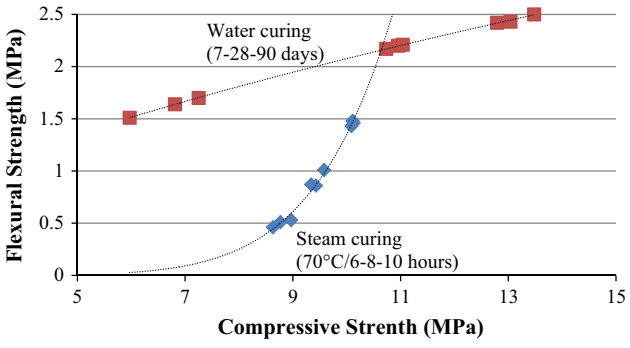


Fig. 6. Correlation between the flexural and the compressive strengths of the water and steam curing (at 70 °C).

Table 3
Comparison of the properties of Earth of Datça-lime binder with fired clay bricks and calcium silicate bricks.

Properties	Loadbearing Fired Clay Bricks (TS EN 771-1) [28]	Non-Loadbearing Calcium Silicate Bricks (TS EN 771-2) [29]	Earth of Datça-lime binder blocks (steam curing at 70 °C for 6 h)
Specific Weigh (g/cm^3)	2.4–2.7	–	2.44
Unit Weight (g/cm^3)	1.2–1.8	0.8–1.6	1.49
Capillarity Coefficient (cm^2/s)	10^{-4}	–	1.12×10^{-4}
Water Absorption (%)	8–18	8–18	13.92
Compressive Strength (MPa)	5–20	5–25	10.11

clay bricks and calcium silicate bricks in terms of main physical and mechanical properties. According to this comparison, Earth of Datça-lime binder under steam curing (at 70 °C for 6 h) may fulfill the requirements of the properties and thus, it can be used instead of loadbearing fired clay bricks and non-loadbearing calcium silicate bricks in building sector.

4. Conclusions

Following conclusive remarks can be stated from the obtained results:

- A vast increment of the physical and mechanical characteristics of Earth of Datça-lime binder with water curing reveals that high level of humidity benefits hydration reactions. The existence of moisture promotes the infilling of the voids. Thus, the binder structure becomes more dense and strong. On the other hand, long-term air curing at low relative humidity adversely impacts the characteristics of the binders which may develop cracks due to the shrinkage.
- As for the curing temperature, the physical and mechanical characteristics of the specimens gradually decrease in contrast to the increasing of temperature above 70 °C. Hence, 70 °C under the high level of humidity conditions can be suggested as optimum curing temperature for Earth of Datça-lime binder.
- Pre-curing at 20 ± 2 °C and 95 ± 5% RH for 36 h in mould is found as ideal conditions for (ED)B in terms of obtaining adequate consistency. Regarding the steam curing period, curing longer than 6 h contributes less to further improvement in the pore structure. So, 42 h treatment in total (36 h for pre-curing +6 h for steam curing) is suggested as optimum steam curing period at atmospheric pressure for Earth of Datça-lime binder.
- All the results obtained at this study show that Earth of Datça-lime binder through steam curing at atmospheric pressure may be used for the production of materials similar to that of fired clay bricks or calcium silicate bricks.
- Using steam curing method at atmospheric pressure provides more mechanical strength, energy-efficiency and time saving compared to that of heating curing. In addition, although the energy consumption for steam curing is higher than the water curing, the saving from time and the requirements of less storage space during production may compensate this disadvantage.
- Binder without steam curing may also be used as a mortar ingredient in the range of several construction applications of preservation as well as new construction; so this means that it may be an alternative to the energy-intensive cement.

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