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Manufacture of Clay Aggregate Doped with Pozzolan Destined for Lightweight Concrete

Hamza Mesrar[1], Laila Mesrar[2*], Abdelhamid Touache[3], Raouf Jabrane[1]

[1] Laboratory of Intelligent Systems, Georesources and Renewable Energies, Geology Department, Sidi Mohamed Ben Abdellah University, Fes, Morocco

[2] Laboratory Waves and Complex Environnement, Normondie University, UMR 6294 CNRS, France

[3] Laboratory of Mechanical Engineering, Mechanical Department, Sidi Mohamed Ben Abdellah University, Fes, Morocco

* Corresponding author's e-mail: hamza.mesrar@usmba.ac.ma

ABSTRACT

In this work, marl clay was used because these materials have a very important industrial potentiality in several fields, namely ceramics. The objective was manufacturing expanded clay aggregate (ECA), with two main ingredients of marl and pozzolan at different percentages in order to integrate them into the concrete as aggregate. The physicochemical parameters of the mixture marl / Pozzolan was discussed and the results of the analyses, allowed deducing that the sample with 15% pozzolan has the most expansion rate of 16.8%, and its density of 1232 kg/m^3 is in accordance with the international standard of expanded aggregates. The density of the concrete decreases with the quantity of ECA added and reaches its minimum with 1671 kg/m^3 according to concrete with 50% of the expanded aggregate. The bending tests show the increase of the mechanical strength as a function of the quantities of aggregate added. The results show a very important potential with the addition of clay aggregates, density and water absorption decrease with the increase of the mechanical resistance.

Keywords: marl, pozzolan, density, expanded clay aggregate, concrete, mechanical resistance.

INTRODUCTION

In recent years, there is an increasing demand for granular materials used in the construction sector in Morocco, the depletion of natural reserves is a matter of time, these intense extractions of aggregates made along the beds of rivers or natural sand beaches destabilize the geomorphological and biological balance of the aquatic ecosystem. The preservation of ecosystems by reducing the intense extraction of natural environments and energy savings in order to reduce the emission of greenhouse gases has become paramount.

The study area is the quarry that is located in northwest Morocco, along the outer limit of the southern Rifan Range at the contact of the Sais Basin, which consists mainly of chaotic-structured gravity nappes resulting from the destruction of outward moving nappe fronts [Leblanc and Olivier, 1984]. The samples were collected northwest of the city of Fez and are composed mainly of an Upper Miocene marl matrix [Tejera De Leon et al., 1995]. Marl is a type of clay found on the surface or at depth in the form of sediments or unconsolidated rocks, it is composed mainly of clay minerals and limestone [Durand, 1972]. Clay minerals are formed by physical or chemical alteration of the silicon and aluminum rich parent rock and reveal an amazing diversity of mineralogical composition as they exhibit a wide range of solid solutions and a great ability to form mixed crystals by interstratification [Reynolds, 1980]. The large reserve of this material and its ease of surface extraction make it very important for industrial exploitation. Its physical properties such as its plasticity and interesting refractory clay minerals give it a very interesting exploitation potential [Grim, 1953].

It is used for various industrial applications (ceramics, inks, purification oils, pharmaceuticals, paper, paint, oil industry, etc.). Clay is one of the most used industrial raw materials, the latest studies on the Miocene marls of Benjalik show an impressive potential, and they have been doped with MnO_2, Fe_2O_3, and Al_2O_3 [Mesrar et al., 2014, 2021]. Conventional concrete is very expensive, and causes irreparable damage to the environment, hence the idea of preparing a thermal insulation material with acceptable mechanical properties [Yew et al., 2021]. Clay is used as a construction product by preparing Expanded Clay Aggregate (ECA), either by clays like vermiculite which has a very high expansion capacity [Becker et al., 2022], or by polypropylene fibers, which have a very low density [Yew et al., 2021; Ye et al., 2022], and then their incorporation in lightweight concrete [Zhang et al., 1992; Wasserman and Bentur, 1997].

On the other hand, the marl shrinks with the increase of temperature because the porosity created during the firing enables the gases released by the decomposition of carbonates and organic matter to escape [Allison et al., 1965]. This is why the authors had the idea of adding pozzolan because iron and quartz close the pores of the surface with a phenomenon called vitrification [Akwilapo et al., 2003], to store the gases and obtain the expansion of the aggregates. Marls were mixed with 10%, 15% and 20% pozzolan to know the percentage that gives the greatest expansion and specifying the temperature and time necessary for firing. The addition of calcium and aluminum powder [Azarhomayun et al., 2022], metakaolin and silica fume [Güneyisi et al., 2012] were used in previous studies.

In the formulation of concrete, others use fly ash [Wasserman and Bentur, 1997] or rice husk ash and coal bottom ash [Bheel et al., 2021] or newsprint ash [Wong et al., 2022], and they have also studied the physicochemical properties [Khudhair et al., 2018; Khudhair et al., 2017], thermal parameters, microstructural and acoustic parameters [Becker et al., 2022], fire resistance [Alireza et al., 2021], or have conducted research on the pre-wetting of clay aggregates and its effect on durability [Musial et al., 2021], liquefaction attenuation [Ghorbani et al., 2021] and also on the flexural strength of lightweight concrete [Baronet et al., 2022]. The lightweight concrete mix was performed according to the world standards as it has been done in several studies before [Melanie, 2003; Bogas et al., 2014]. Then, it was monitoring during curing by measuring the shrinkage of concrete until the curing age of 28 days [Baronet et al., 2022]. After curing, the study of physical properties, such as density [Wang et al., 2022], water absorption [Zheng et al., 2021] and mechanical characteristics using three-point bending strength was carried out [Azarhomayun et al. 2022; Baronet et al., 2022].

MATERIALS AND METHODS

Samples preparation

In the present study, the used marl originated from Quarry of Ben Jallik, Fez, Morocco (Fig. 1). The marl samples were oven dried at 105°C for 24 hours, quartered to select a representative sample of the materials. Then, they were manually ground to a reduced particle shape. Subsequently, they were finely ground for 5 minutes in an agate mill, and doped the marls with pozzolan at different percentages (0%, 10%, 15%, and 20%). Afterwards, the mixture was ground for 5 minutes to better homogenize the materials. The expanded clay aggregate is prepared by the composite of marl/pozzolan with an addition of water. The light aggregate was dried in the oven at 105°C for 24 hours to start the firing process in a muffle furnace for 5, 10, 15, 30 minutes, to evaluate the expansion of ECA, on each temperature, namely 900–1100°C in 100°C increment (Fig. 2). Then, the higher expansion aggregate with cement (CPG45) and sand was used, to formulate the lightweight concrete according to the procedure described in NF EN 133699 (Fig. 3).

Chemical analysis

The chemical composition of clays was determined by X-ray and fluorescence spectrometry (Axios, ELE03-PROT v01) using pressed powder pellets. For X-ray fluorescence, irradiation with a primary X-ray bundle from an X-ray tube causes the emission of fluorescent x-rays with discrete energies characteristic of the elements present in the sample. The analysis were performed at the National Center for Scientific and Technological Research (NCSTR) (Morocco).

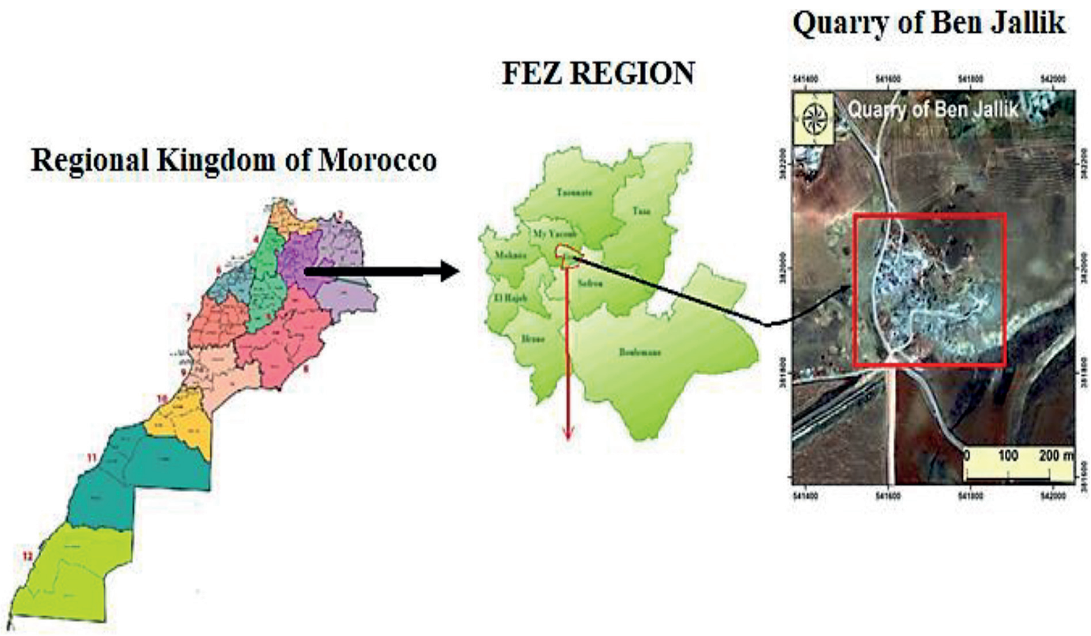

Figure 1. Location of the sites investigated (Quarry of Ben Jallik, Fez, Morocco)

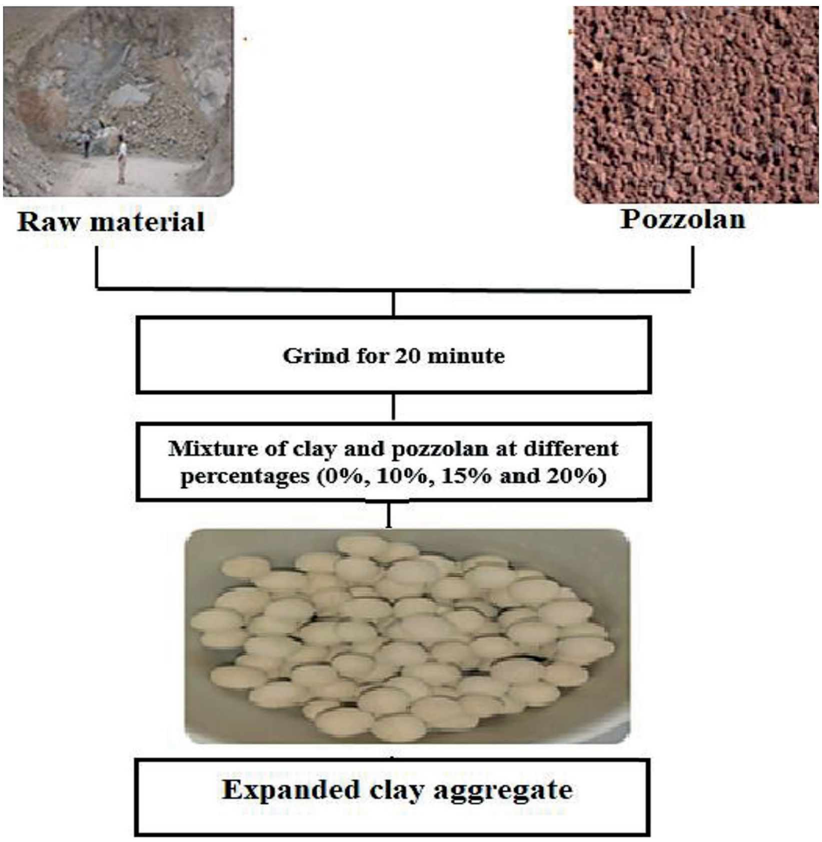

Figure 2. The schematic process of composite material

Physical analysis

Scanning electron microscope (SEM)

The SEM image is effected by a pressure controlled instrument, the cathode which produces the electron beam, the condenser system which forms a reduced image of the cross-over which is then projected by an objective lens on the object, the scanning system formed by the deflection coils to move the probe on the object and finally the detector which picks up the signals coming from a fluorescent screen and produces a black and white image.

Atterberg limits

The Atterberg limits are a basic measure of the critical water contents of a fine-grained soil: its plastic limit, and liquid limit. The test is obtained by using standard (NF P 94-051).

Differential scanning calorimetry (DSC)

Thermoanalytical technique in which the difference in the amount of heat required to raise the temperature of a sample and a reference is measured as a function of temperature. The sample and reference are kept at approximately the same temperature throughout the experiment. In general, the temperature program for a DSC analysis is designed, so that the temperature of sample holder increases linearly with time. The reference sample must have a well-defined heat capacity over the temperature range to be analyzed.

Density

The procedure for measuring density is essentially based on mass and volume of sample, which is placed in a small cup (pycnometer) of known volume at a given temperature. In order to calculate the density, the weight obtained was taken and divided by the volume of the liquid tested.

Water absorption

This test was performed on hardened concrete and water absorption was measured by immersion using international standard (NBN B 15-215(1989)). This test gives some information about the porosity and consequently the resistance of the material.

Mechanical analysis

The bending strength tests were realized in the mechanical laboratory of FST Fez. Testing was carried out by W500 machine (Fig. 4), with a maximum effort of 50 KN and connected to a software- driven computer system (EM 506), which gives the evolution of the bending resistance (N) according to the displacement (mm). To calculate the maximum resistance of flexural strength, the equation of three point bending was used [Bogas et al., 2014]:

$$\sigma_{max}=\frac{3}{2}\left(\frac{FL}{bd^2}\right) \tag{1}$$

The results are expressed in MPa. The flexural strength is tested for the comparison between fired and unfired bricks.

RESULTS AND DISCUSSION

Chemical composition

The chemical composition of the marls is shown in (Tables 1 and 2), it is formed by 46.6% of SiO_2 (it has an effect of refractoriness and contraction of the mass) [Kornmann et al., 2005]; 13.4% Al_2O_3 (the fact that these marls contain 15% of Al_2O_3 means that they are considered moderately plastic); 8.87% CaO (it influences the porosity of the material and its presence indicates that the mineral contains plagioclases); 6.91% Fe_2O_3 (which acts as a flux; this oxide is the cause of the brown or red color of the products after firing) [Arib et al., 2007]; 19.9% of loss on ignition and 4.32% of minor elements (P_2O_5; MnO; Na_2O; k_2O; MgO and Cl) they promote the formation of a glassy phase with a low melting point during the firing process (energetic flux role). From these results, it can be concluded that these marls can be used in ceramics [Nshimiyimana et al., 2020]. It was noticed that the addition of pozzolan leads to an increase in the rate of iron and titanium oxides and a decrease in the loss on ignition.

Physical analysis

Scanning electron microscope

The SEM gives the magnified images of the size, shape, composition, crystallography, and other physical and chemical properties of a specimen. The raw sample presented in (Fig. 6) shows quartz grains and phyllosilicate in sheet form, while the sample doped with 15% pozzolan shows a significant structural change (Fig. 7), notably an increase in closed porosity compared to the raw sample.

Atterberg limits

The curve presents the limits of Atterberg as a function of the percentage of pozzolan, indeed, the addition of pozzolan in marls introduces an increase in plasticity and a decrease in the limit of liquidity [Pimraksa et al., 2009; Silva et al., 2014], which reveals that the pozzolan plays a role of degreaser and contributes to the phenomenon of the expansion [Aineto et al., 2006; James et al., 2018] (Fig.8).

Differential scanning calorimetry

The differential calorimetry curves represent the variation of the absorbed energy as a function of time (Fig. 9). The results show the decrease in absorbed thermal energy with the amount of pozzolan added. It is obvious that the effect of the pozzolan has modified the thermal properties of the marl. Therefore, the material becomes more insulated with the addition of the pozzolan. On the industrial level, this parameter is of great use on the economic level of the energy in particular in the building and public works sector (BPWS).

Figure 3. The schematic process of lightweight concrete

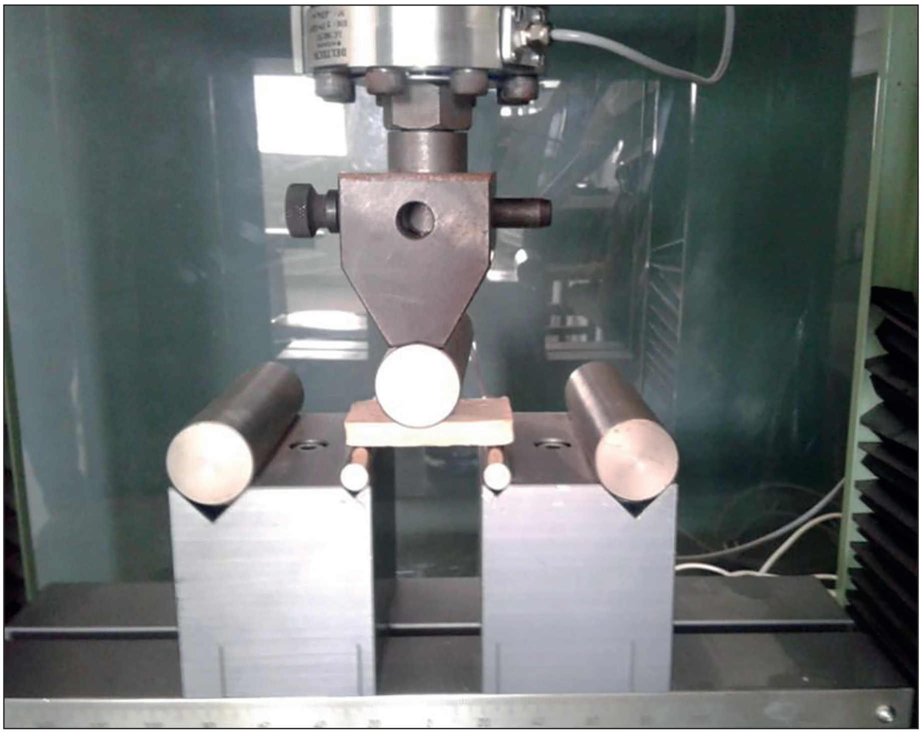

Figure 4. Photographs of the flexural strength machine (W500)

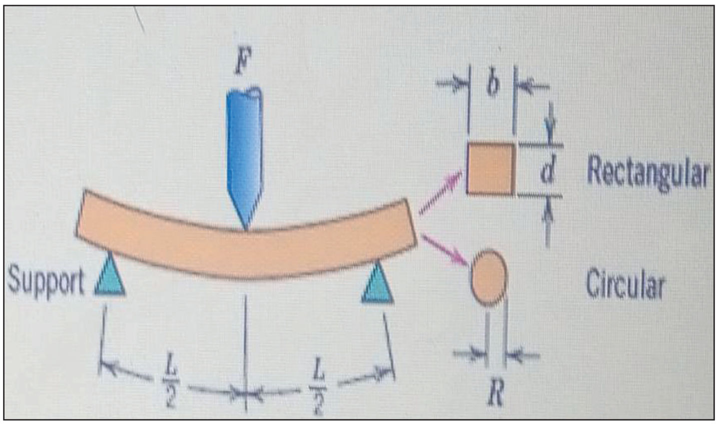

Figure 5. Arrangement of specimen for flexural strength test.

Table 1. Chemical component analyses of raw and mixed materials (Wt. %)

Percentages of pozzolan in the raw material	SiO_2	L.O.I	Al_2O_3	CaO	Fe_2O_3	MgO	Cr_2O_3	Na_2O	K_2O	SO_3	TiO_2	NiO	P_2O_5	MnO_2	Cl
0%	40.6	19.9	13.4	8.87	6.91	3.44	2.16	1.58	1.19	0.629	0.355	0.234	0.207	0.206	0.165
15%	40.7	18.8	13.5	8.65	7.9	3.43	2.27	1.37	1.17	0.586	0.525	0.262	0.244	0.273	0.156
20%	39.9	18.8	13.3	8.71	8.41	3.62	2.34	1.39	1.07	0.589	0.58	0.288	0.279	0.247	0.156

Table 2. Fluorescence analysis of Pozzolan

Oxides names	Concentrations (%)
SiO_2	37.8
Al_2O_3	17.0
Fe_2O_3	17.3
MgO	6.2
CaO	3.88
Na_2O	0.82
K_2O	0.355
P_2O_5	0.72
TiO_2	2.3
NiO	0.47
MnO_2	0.435
Cr_2O_3	2.87
SO_3	0.33
Cl	0.16
Loss on ignition	9.00

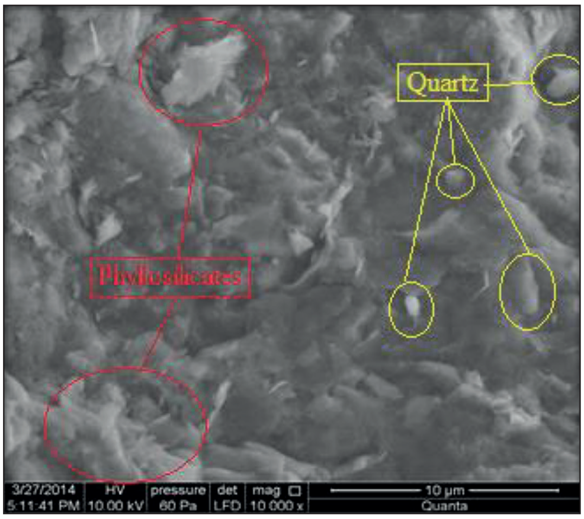

Figure 6. SEM image of clay used in the formulation

Firing process

The curves show that the aggregate diameter increases with firing time, since pozzolan intensifies the porosity [Matias et al., 2014] and reduces the pore size [Li et al., 2020]. The raw sample reveals an increase in the expansion rate with time and reaches its maximum at 6.25% around 30 min. However, the diameter of the expanded clay aggregate doped with 15% and 20% pozzolan shows significant expansion at 30 min (Fig. 10). The highest expansion rate which reaches 16.8% for (15% PZL) with a density of 1232 kg/m^3 in agreement with other authors [Nie et al., 2011; Mydin et al., 2015], shows an interest for its use in lightweight concrete [Wasserman and Bentur, 1997; Bogas et al., 2014].

Density of concrete

Indeed, lightweight concrete is less heavy than conventional concrete. It can be placed on

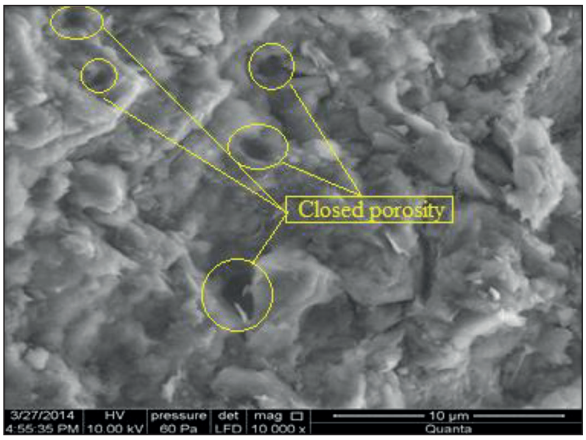

Figure 7. SEM image of formulation clay and 15% of pozzolan

a structure requiring a lower load resistance. This can be an advantage because the structure is easier to put in place and consequently, savings are made. In addition to its low density, lightweight concrete has the particularity of being both a good thermal and sound insulator (Fig. 11).

For the placement of lightweight concrete, the authors complied with the NF EN 13369 (2013) standards. For 1 volume of cement + 2 volume of sand + % of EC). The results show a negative correlation between density and the proportion of expanded clay aggregate added in the concrete samples (Fig. 9). The employed ECA marl and pozzolan achieved the desired objective since the densities of the concretes

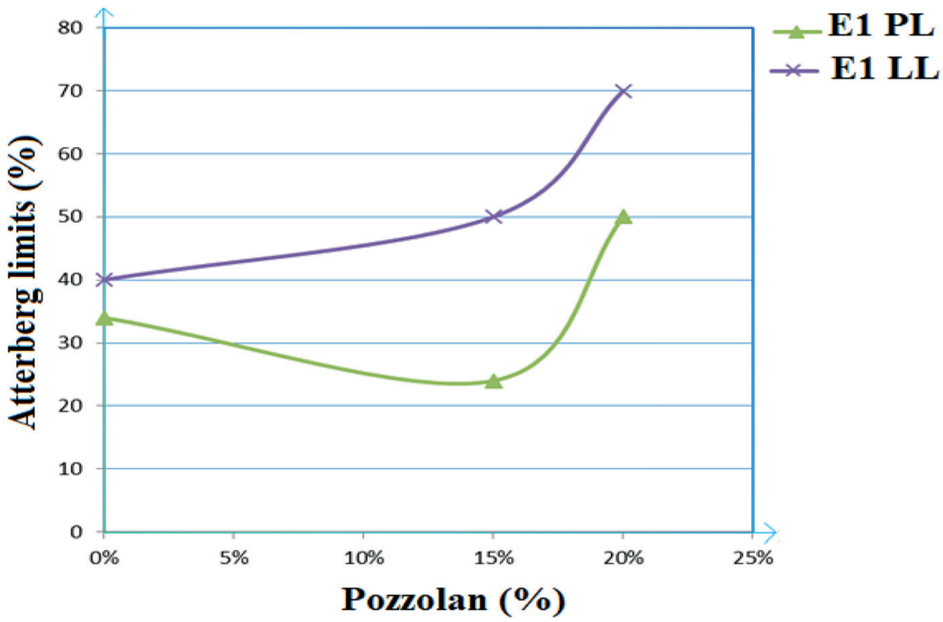

Figure 8. Atterberg limits (PL – plastic limit, LL – liquid limit)

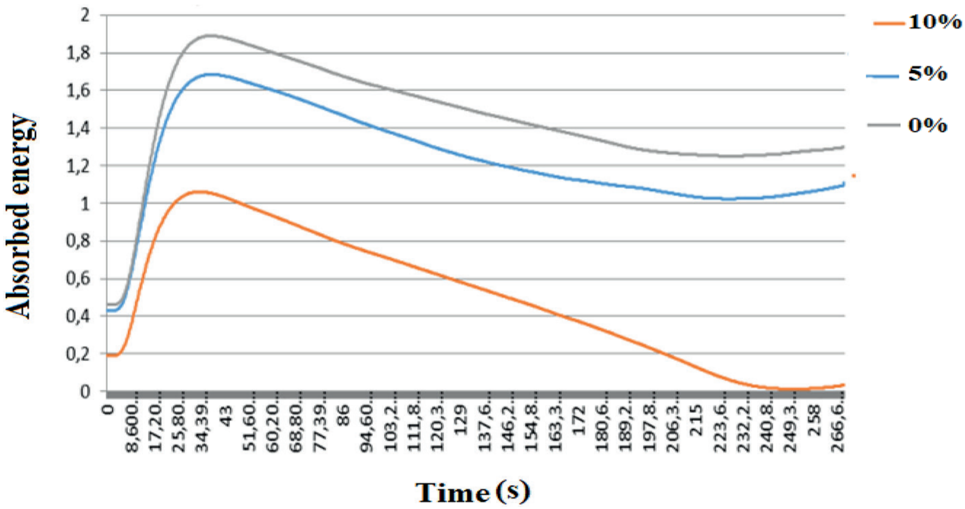

Figure 9. Result of the differential scanning calorimetry test

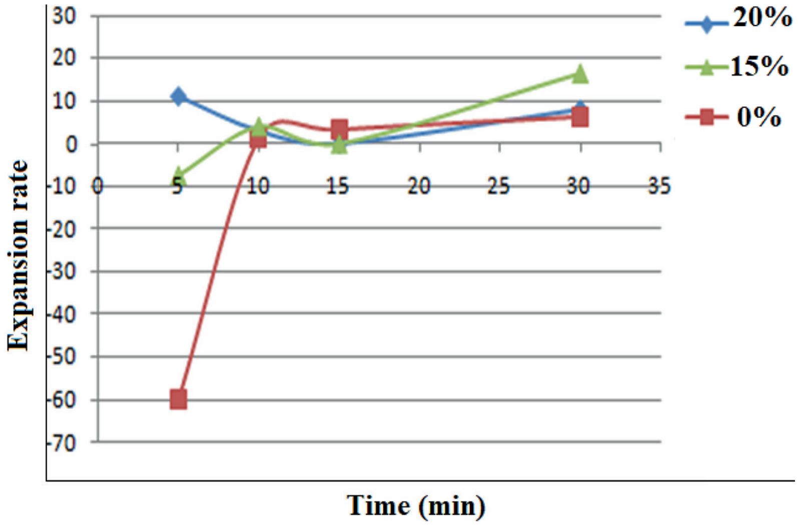

Figure 10. Diameter of expanded clay aggregate as a function of firing time

are all lower than 1800 kg/m^3 [Ye et al., 1992], in agreement with several studies performed by other authors [Samson et al., 2017; Vandanapu et al., 2018]. The lowest density is that of 50% ECA which reaches 1671 kg/m^3 [Kan et al., 2009].

Water absorption

The water absorption informs us about the open porosity of the specimen [Raimondo et al., 2009]. Therefore, Figure 12 shows the values of absorption of lightweight concrete as a function of percentage of ECA added. This figure reveals that the concrete doped with 50% of aggregate has a maximum value 14%, while these percentages of absorption are low, testifying to a good mechanical strength [Tharakarama et al., 2017].

Mechanical analysis

Flexural strength

Figure 13 shows the specimens made from the mixture of different percentage of expanded clay aggregate (0, 5, 10, 20, 30, 40 and 50%). The following test is designed to determine the required proportion of expanded aggregates that must be added to the lightweight concrete mix to ensure satisfactory strength according to US construction standards. Flexural strength is carried out on the specimens composed of (cement + sand + expanded clay aggregate). The unreinforced lightweight concrete is marked by the failure of the matrix [Melanie, 2003]. When the concrete could no longer resist the stress, the micro-cracks widened

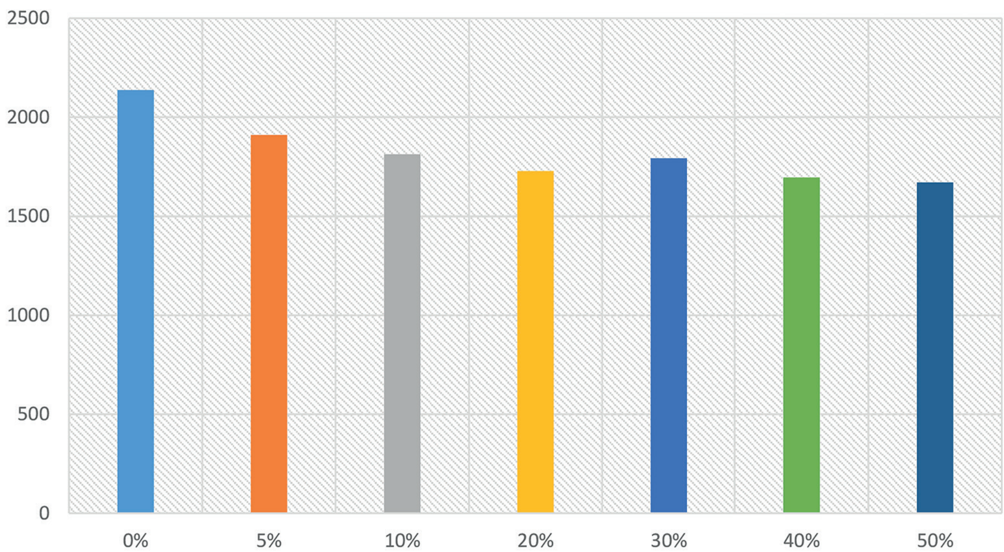

Figure 11. Density of cement samples with percentages of ECA added

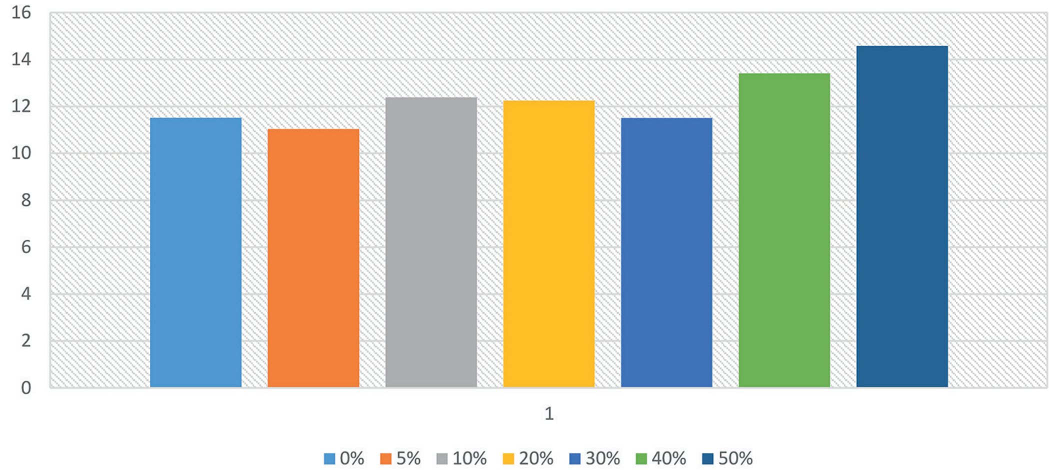

Figure 12. Water absorption of samples

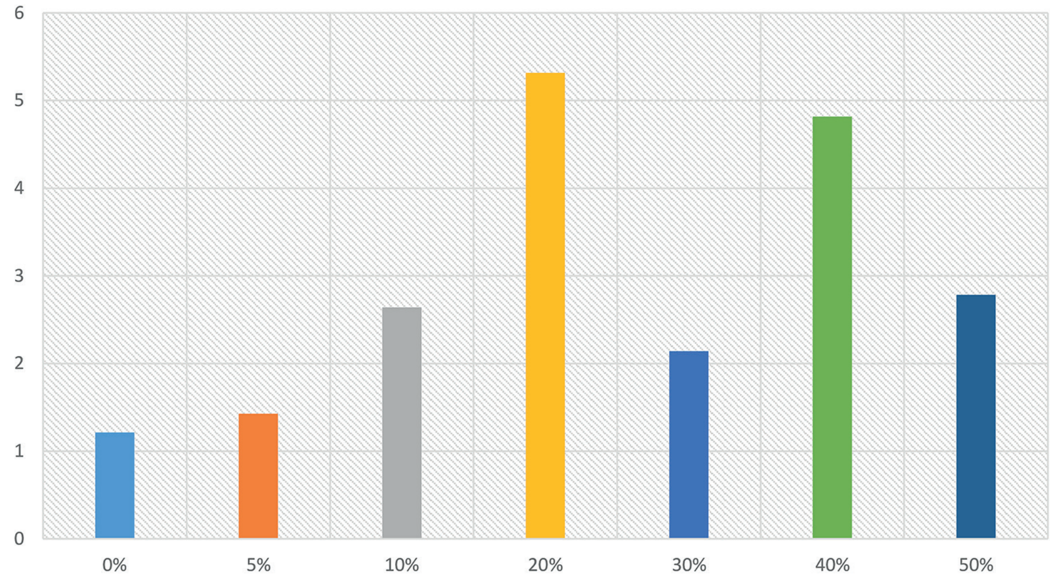

Figure 13. Flexural strength curves of lightweight concrete doped with expanded clay aggregate

and turned into macro-cracks [Wong et al., 2022]. It was noticed that the mechanical resistance increases with the percentage of expanded clay aggregate added until 40%. The highest resistance is observed in the mixture of 20% of clay aggregate added. It is apparent that with the increase of the percentage of expanded clay aggregate the density decreases as well as the strength increases. This is mainly due to the semi-porous surface and the pozzolanic activity of the studied aggregates with the cementitious matrix which reinforces the structure of the lightweight concrete [Awoyera et al., 2018; Wasserman et al., 1997]. The results of the maximum bending stresses of the specimens containing 20% aggregate which reached 5.31 Mpa and a density of 1728 kg/m^3, can be used for splitting concrete according to the research already carried out by others authors [Zhang et al., 1991; Wilson et al., 1988].

CONCLUSIONS

This study allowed concluding the following. The mixture of marl and 15% pozzolan has the best expansion result (16.8%), and the lowest density of 1232 kg/m^3 according to the international standard. The decrease in absorbed energy is proportional to the increase in the percentage of pozzolan added. The density of the concrete decreases with the amount of aggregates added and reaches its minimum at 1671 kg/m^3 at 50% expanded clay aggregate. Flexural tests show the increase in mechanical strength as a function of the amount of expanded clay aggregate added. The maximum strength was found in the mixture containing 20% of clay aggregate added.

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