



Mechanical and physical properties of stabilised compressed coal bottom ash blocks with inclusion of lateritic soils in Niger[☆]



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ABSTRACT

This paper describes the results from an investigation on the production of compressed block made with coal combustion by-products and local clayey soil in Niger. Stabilisation with Portland cement and a mixture of Portland cement and lime was adopted. Mechanical, physical, hydric, and thermal characterisation of the produced bricks was carried out. Blocks with satisfactory compressive strength were produced, with samples consistently exceeding the compressive strength of 4 MPa. Stabilisation with Portland cement proved to be the most effective in terms of strength development. However, satisfactory results were obtained with partial substitution of cement with lime (up to 30%). Porosity was found to be in the range 33% to 40% for all samples. The low thermal conductivity (in the range 0.31 to 0.48 W/m·K) was presumably influenced by the high porosity. Water absorption of the bricks was found to be very fast, although total water absorption (in the range 20.6 to 28.7%) was lower than the calculated porosity, suggesting that some of the pores were not accessible by water. Samples subjected to heating showed very promising results in terms of strength and mass loss. An increase in compressive strength was recorded up to temperatures of 400 °C. This might be due to the triggering of other reactions in the binding matrix due to the chemistry of the bottom ash. A change in colour of samples (from grey to red) was observed, due to the dehydroxilation of iron hydroxide turning into ferric oxide.

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Introduction

Background

The consumption of raw materials for energy production or other industrial activities and the management of waste/by-products influence the sustainability of targeted industrial sectors, the quality of local and global environment and the local economic development. This is particularly true in the case of sub-Saharan countries where the exploitation of local resources offers opportunities for economic growth but poses treats to the local environment.

Despite being responsible for approx. 35% of the global CO₂ emissions (data for 2017) [1], coal remains a privileged energy source that plays and will probably still play a major role in energy production in the 21st century, according to the International Energy Agency (IEA) [2]. Coal is still the primary source for the production of electricity, being the 39% of the world electricity produced from coal in 2015 [2]. World largest coal reserves are in the USA, China, Russia, Australia, and India [3].

Coal combustion results in the production of a number of solid residues in a range of sizes and qualities, depending mainly on combustion technology and quality and composition of the fuel. Some of the residues are widely recycled (e.g. fly ash), used as additive in the production of cement or concrete for their pozzolanic properties, as road building materials and in agriculture to improve soil properties [4–6]. Others are still underexploited, as in the case of bottom ash (BA), which is a granular material rich in silicates and aluminates. In 2016, BA production in Europe accounted for 9% of the total coal combustion products, and only 29% was utilised in high value applications, whilst about 71% was used as bulk material for filling, reclamation/restoration or stockpiled in temporary (or permanent) dumps [7].

Population growth dynamics and rapid urbanisation in developing countries cause concerns on the availability of building materials and on their environmental impact [8]. Earthen construction is a vernacular technique that has been gaining interest in the last decades because of its low environmental impact compared to modern building materials [9]. Deterioration of earthen elements (mainly by water) is one of the main issues faced by these materials. Methods for improving the mechanical and hydraulic performance of earth blocks (EB) involve stabilisation with other substances, typically binders (cement, lime, bitumen emulsions, but also cow dung) [10]. Previous studies indicated that most common stabilisation techniques consist of addition of cement or lime, or a combination of the two [11–13]. Lime was used as a stabiliser in the manufacture of EB in order to activate pozzolanic reactions from silicoaluminates present in clay (due to the high pH of the pore solution), whilst reducing the cement content in the mix. Nagaraj et al. [11] reported that substitution of 50% by weight of cement with lime led to uniaxial compressive strengths of bricks (under moist conditions) higher than these obtained with neat cement, when sufficiently long curing periods (> 6 months) are ensured. Water absorption rates were found to be lower than these obtained from cement-stabilised EB [11]. Other investigations suggested that high substitution rates of cement with lime did not negatively affect the water absorption of bricks [13].

Several studies indicated that inorganic wastes such as fly ash from coal combustion can be used in different proportions for the manufacture of cement stabilised bricks [4,14–18]. In some cases, earthen bricks produced using high proportions of fly ash showed properties challenging fired bricks [19].

Current situation in Niger and justification for the study

In 2014, Niger was the world's fourth-ranked producer of uranium, accounting for about 7% of world production [45]. Other mineral commodities produced in the country included clinker, coal, crude oil, gold, gypsum, limestone, salt, silver, and tin. Clinker, phosphate rock, oil, and uranium operations were privately owned, whilst coal and gold mining operations were owned by the government. Over the last forty years, the number of inhabitants has quadrupled from 5 million in 1977 to nearly 20 million in 2018, with 68% of the population under the age of 24 [46]. Housing is a concern for many people, particularly low-income households and young civil servants. The Niger government, within the framework of its national housing policy, committed to build 25,000 housing units from 2016 to 2021 [47].

The Niger Coal Company (SONICHAR) produces electricity through the combustion of coal mined in Tefereyre, 75 km northwest of Agadez. In order to address the environmental issues that could result from the storage of coal combustion residues in the proximity of the exploitation sites, SONICHAR commissioned a feasibility study of the potential valorisation of BA produced by their power plant in the construction sector. The study initially focussed on determining the technical feasibility of recycling BA in the production of compressed bricks for housing construction. Physical, chemical, mineralogical and environmental properties of BA were assessed and the material was found to be suitable for utilisation. The study was then focussed on the manufacture of compressed masonry bricks with BA in combination with other local materials such as loose lateritic soil sourced in Burkina Faso (Ouagadougou). The effects of mix proportions (i.e. proportions of Portland cement, slag and laterite in the mix) on the compressive strength of the blocks were assessed and suitable mix proportions achieving a target strength of 4.5 MPa were suggested [20]. Outcomes from the investigation indicated further developments for the understanding of the potential production of stabilised earth blocks with inclusion of BA from SONICHAR, focussing on the evaluation of the reproducibility of the obtained results using natural materials sourced near Agadez (for obvious economic reasons). Furthermore, the assessment of the effects of using lime for partial or total substitution of Portland cement for the stabilisation of bricks, and the quantitative analysis of the behaviour of bricks under elevated temperature, compared with that of Portland cement-based concrete were addressed as possible research developments

Table 1
Physical properties and chemical composition (Wt%) of BA (from [20], modified).

Property	Results
Grain size distribution	Sandy gravels
Specific density	2.2 t/m ³
Bulk density	0.7 t/m ³
Porosity	67%
SiO ₂	62.32%
Al ₂ O ₃	27.21%
FeO	3.57%
K ₂ O	2.58%
TiO ₂	2.15%
MgO	0.95%
Na ₂ O	0.7%
CaO	0.5%
MnO	0.01%

Building on these recommendations and on the available results [20], this paper focusses on the evaluation of the physical, mechanical, hydric and thermal properties of compressed bricks produced with BA and clay materials sourced in north-easter Niger, with the addition of Portland cement and lime. Clay deposits around SONICHAIR site with similar properties to the laterite utilised in the preliminary study were characterised and used for the production of blocks.

The partial substitution of cement with lime was also investigated. Although the expected quantity of clay in the block mixes would not justify the use of lime as stabilisation agent, this experimental step was carried out also for assessing possible beneficial effect of portlandite dissolved in the pore solution in activating amorphous silicates and aluminates from BA (through dissolution and subsequent reorganisation), which would provide additional strength. The interest in investigating the possible utilisation of lime was also due to other considerations. Lime require much less energy than cement for its manufacture and it slowly returns to its original state as limestone through carbonation processes. Lime is permeable to water vapour, absorbing and then releasing it quickly and therefore allowing to control moisture from walls and floors. Eventually, lime also has antibacterial and antiseptic properties as well as good fire resistance.

The effect of high temperatures (200, 400 and 600 °C) exposure on the compressive strength of bricks was also analysed.

The objectives of this study were therefore two: (a) to investigate if bricks with suitable mechanical properties could be produced in Niger with local geomaterials and by-products (thus being low-cost and low-carbon building materials); (b) to assess if recycling bottom ash for brick production was a technically viable strategy for the management and recycling of coal combustion residue in Niger and any other developing country where coal is a primary source to produce energy.

The experimental programme followed the sequence described hereafter:

- Geotechnical characterisation of local soils retained for the study.
- Production of compressed blocks containing bottom ash and local soils and stabilised with cement and/or lime.
- Evaluation of the physical, mechanical and thermal properties of each mix.
- Evaluation the hydric properties (i.e. water absorption tests and capillarity rise tests) of blocks that gave satisfactory strength results.
- Evaluation of the mechanical behaviour of blocks exposed to high temperature. Fire resistance tests have been performed only on the mix that gave the highest compressive strength.

The economic and environmental analysis of the developed building elements, assessed against existing blocks produced from earth materials, falls outside the scope of this paper and it is mentioned in the conclusions as a further development of the study.

Materials and methods

Materials

Bottom ash

The main factors affecting the quality and the properties of coal combustion residues are the mineral components of coal and the combustion technique. Typically, fly ash (FA), bottom ash (BA), boiler slag (BS) and fluidised bed combustion (FBC) ash are the main combustion products. Furthermore, dry or wet flue gas desulphurization operations result in the production of semi dry absorption (SDA) product and flue gas desulphurization (FGD) gypsum. BA accounts for about 9% of the total combustion residues production, whilst FA represents about 64% and FGD gypsum nearly 24% [7]. The physical and chemical properties determined on the BA produced by SONICHAIR were discussed in [20] and shown in Table 1.

Table 2
Grain size distribution parameters and Atterberg limits of investigated soils.

Label	Gravel fraction%	Sand fraction%	Silt fraction%	Clay fraction%	LL%	PL%	PI%	A
AGA	21.5	60.2	6.5	11.8	49.6	24.9	24.7	2.0
BCS	5.5	62.6	13.0	18.9	53.4	24.8	28.6	1.4
CEP	13.5	42.9	29.5	14.1	42.9	28.1	14.8	1.0
BFA	36.3	28.4	6.2	29.2	53.0	29.0	24.0	0.8



Fig. 1. Visual appearance of the lateritic soil deposit at AGA site.

Binders

The cement used for this study was “CPA 45”, supplied by the Togo cement company (CIMTOGO) and equivalent to the CEM I 42.5 according to EN 197-1 [26]. The following properties were specified: specific density 3.10 t/m^3 , apparent density 1.06 t/m^3 , BET specific surface area $1.47 \text{ m}^2/\text{g}$, initial setting time 180 min [27]. Investigated cement dosage was 8.5% in volume, in conformity with [20].

In order to improve the economic and environmental profile of the produced bricks, lime (Ca(OH)_2 calcium hydroxide) was used as stabiliser as partial cement substitution for investigating the possible pozzolanic reaction of BA. Substitution rates of cement with lime were 30%, 50% and 70% in volume. Milk of lime was preferred to lime powder in order to avoid the appearance of white lime stains on the bricks as well as to improve the homogenization of materials during the mixing phase. The required mass of lime was mixed with required water volume before adding these to the others materials in the mixer.

Lateritic soils

Several clayey soil deposits were identified in the surroundings of Agadez and Tchirozérine (northeaster Niger). Soil samples from each deposit underwent a geotechnical classification, and materials from three sites already exploited for earth brick production were found to be similar to the lateritic soil sourced in Burkina Faso described in a previous work [20], and therefore retained for further experiments. The local materials were labelled CEP (site “Carrière Ecole Publique” in Tchirozérine, N $17^\circ 15' 30.6''$, E $7^\circ 49' 55.5''$), BCS (site “Banco carrière Sonichar” in Tchirozérine, N $17^\circ 16' 49''$, E $7^\circ 51' 02.1''$) and AGA (site “Agadez carrière” in Agadez, N $16^\circ 58' 18.1''$, E $8^\circ 02' 01.6''$), whilst the soil used in [20] was referred to as BFA (Burkina Faso).

Results from geotechnical characterisation (grain size distribution and Atterberg limits) are shown in Table 2, whilst Fig. 1 shows the typical appearance of the lateritic soil deposits (the picture was taken at AGA site). For a complete characterisation of geomaterials, free swell data of tested soil would have been useful, as well as X-ray diffraction (XRD) for detecting the presence of smectite and other clay minerals. However, considering the reduced quantity of lateritic soil used in the mix (around 17%), these tests were not carried out.

The activity (A) of a soil is calculated as the plasticity index (PI) divided by the amount of clay-sized particles (less than $2 \mu\text{m}$). Typically, the activity of clay is between 0.75 and 1.25. When A is less than 0.75, it is considered inactive. Swelling behaviour would be expected from materials AGA and BCS according to their activity index (equal to about 2 and 1.4 respectively, see Table 2). Laterite is generally composed of silica, kaolinite and iron oxides (hematite, goethite) [21,22].

The particle size distribution of soils was obtained in accordance with French standards NFP 18–560, using dry sieving method until grain size $75 \mu\text{m}$ (retained), whilst the fraction passing $75 \mu\text{m}$ was assessed with a sedimentation test using hydrometer method.

According to the United Soil Classification System (USCS), the three materials sourced in Niger AGA, BCS and CEP can be classified as SC (sand and clay), CH (inorganic clay with high plasticity) and SM (sand and non-plastic material) respectively. As a comparison, material BFA (i.e. soil used in [20] and sourced in Burkina Faso) was classified as a mixture of sand and clay. Grain size distribution curves of the investigated soils are shown in Fig. 1, where upper and lower suitability limits suggested by CRATerre guidelines [23] are also included (dashed lines). Such limits provide a range for grain size distributions suitable for the production of compressed earth blocks. This application requires soils showing a good grading (in order to ensure satisfactory compaction properties) and a balanced amount of fine and coarse material (enough fine fraction to

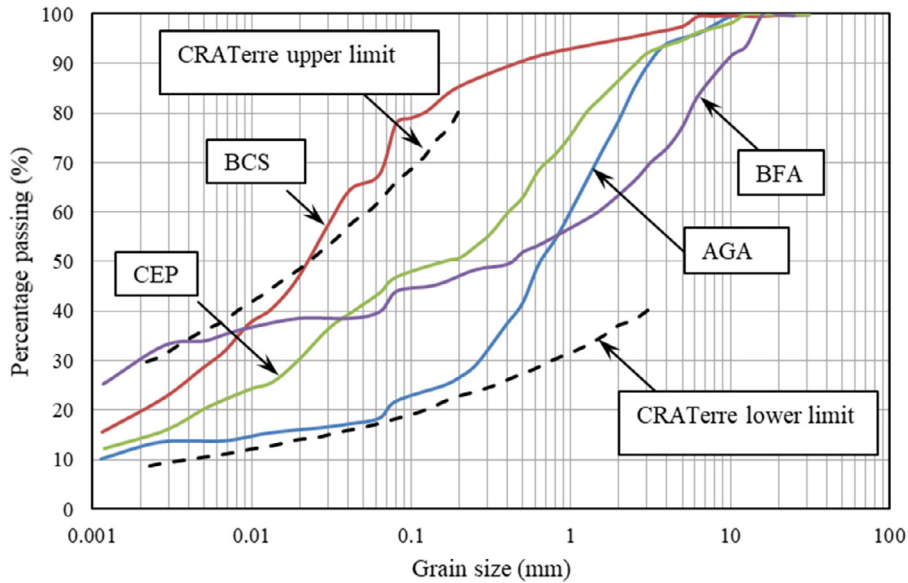


Fig. 2. Grain size distribution curves of investigated soils. Upper and lower suitability limits suggested by CRATerre [23] are plotted as dashed lines. As in the original publication, limits of the grading envelope do not reach 100% but are truncated as shown.

Table 3
Mix proportions for the investigated samples (volume percentages).

Batch	Label	BA	Soil	Cement	Lime	Water
1	MC10	79.4	0	7.9	0	12.7
	MC7L3	82.9	0	5.8	2.5	8.8
	MC5L5	84.7	0	4.2	4.2	6.8
2	MLAGA20C10	68.4	17.1	8.5	0	6
	MLBCS20C10	68.4	17.1	8.5	0	6
	MLCEP20C10	68.4	17.1	8.5	0	6
3	MLBCS20C7L3	66.9	16.7	5.9	2.5	8
	MLBCS20C5L5	66.9	16.7	4.2	4.2	8
	MLBCS20C3L7	66.9	16.7	2.5	5.9	8

provide cohesion to the block, enough coarse fraction to ensure the rigid ‘skeleton’ would avoid excessive shrinkage). Soils with grain size distribution exceeding these limits might still be suitable for block production, provided that appropriate physical and chemical properties are met.

The analysis of grain size distribution curves shown in Fig. 2 indicated that a significant fraction of the investigated soils had grains larger than 3–5 mm. CRATerre guidelines [24,25] suggest that a minimum of 50% of material passing 5 mm is required.

The three pre-selected soils sampled in Niger fitted within the range recommended by CRATerre for the production of cement-stabilised compressed earth blocks, as it was the case for BFA material (see Fig. 2). The clayey fraction of sample BCS exceeded the recommended upper limit. The pre-selected materials were therefore considered suitable for the production of cement-stabilised compressed earth blocks.

Experimental methods

Samples production

According to the practice followed by local compressed earth block producers, soils were sieved and only the fraction passing 5 mm was used for brick production.

Nine formulations, divided into three batches, were investigated in this study (see Table 3). The label of each samples indicates the composition of each mix. “M” stands for bottom ash, “LAGA”, “LBCS” or “LCEP” indicate the source soil, “C” is for cement and “L” is for lime. Numbers in the label inform on the amount of each constituent included in the mix.

Following the practice adopted by compressed earth block producers, quantities of constituent were mixed in volume proportions (see Table 3).

1. *Effect of binder:* the first batch (labels MC10, MC7L3, MC5L5) was produced with neat BA stabilised either with cement or with a mixture of cement/lime. A certain volume of BA was poured into the mixer, then binder was added as 10%

in volume, and eventually water was added until the consistence of the mix was deemed suitable by the operator. The difference in water content for different mixes was due to the effects of the binder blend on the mix consistency. The quantity of added water was recorded and then the volumes of each constituent was recalculated as a percentage. When Portland cement was blended with lime, the volume of this latter was measured according to the required proportions (30% or 50% of the cement volume for samples MC7L3 and MC5L5 respectively) and then mixed with a certain amount of water before being poured into the mixer. Further water was then added in the mixer and the total volume of water was noted.

2. *Effect of locally sourced soil*: the second batch of blocks was produced with the three soils sourced in Niger (LAGA, LBCS, LCEP), adopting the optimised formulation described in [20] as far as BA, soil and cement proportions were concerned (labels MLAGA20C10, MLBCS20C10, MLCEP20C10). The aim of this investigation was to assess the suitability of locally sourced materials for producing blocks with physic-mechanical performances similar to previous results [20]. A defined volume of "solid" fraction (i.e. BA and lateritic soil with proportions 80% and 20% respectively) was poured into the mixer, then binder was added as 10% of the total (i.e. BA + soil) volume, and eventually water was added at a constant volume for the three soils. Although effects of fine content and fine fraction plasticity might play a role in the consistency of the mix, this parameter was not further investigated in the experimental plan. The quantity of added water was recorded and then the volumes of each constituent was recalculated as a percentage.
3. *Combined effect of binder blend and locally sourced soils*: the third batch was produced with BA and LBCS material, investigating the effect of partial substitution of Portland cement with lime. The choice of limiting this analysis to the material LBCS related to the higher proportion of clay fraction compared to other materials (i.e. LAGA and LCEP). When Portland cement was blended with lime, the volume of this latter was measured according to the required proportions (30%, 50% and 70% of cement volume for samples MLBCS20C7L3, MLBCS20C5L5 and MLBCS20C3L7 respectively) and then mixed with a certain amount of water before being poured into the mixer. Further water was then added in the mixer for achieving the desired consistency and the total volume of water was noted.

Blocks were produced with a Terstaram hand press that could apply variable pressures from 50–60 bar to more than 100 bar on the material (according to the mould filling rate). A vertical shaft mixer with maximum capacity equal to 100 l was used. Blocks were produced in two sizes: (a) blocks with dimensions 14 cm × 14 cm × 9 cm and (b) blocks with dimensions 29.5 cm × 14 cm × 9 cm. Mechanical, thermal and fire resistance tests were carried out on type (a) blocks, while tests for hydric characterisation (i.e. water absorption test and capillarity rise test) were carried out on type (b) blocks. Blocks were kept moist with water spraying during the first fourteen days after production, and covered with a black plastic sheet in order to prevent evaporation.

Compressive strength

Block compressive strength was measured on all samples (MC10, MC7L3, MC5L5, MLAGA20C10, MLBCS20C10, MLCEP20C10, MLBCS20C7L3, MLBCS20C5L5, and MLBCS20C3L7) according to the recommendations from the literature [28]. Tests were carried out with a 1500 kN hydraulic press. An automated data acquisition system consisting of an LVDT type displacement sensor with a maximum length of 65 mm and a pressure sensor with a capacity of 400 bar was coupled to the press. Compressive strength was measured at 28, 60 and 90 days of curing with an Instron press in accordance with NF EN 772-1 [31]. Four blocks were tested for each mix at each curing time.

Thermal properties

Thermal tests were carried out on all samples (MC10, MC7L3, MC5L5, MLAGA20C10, MLBCS20C10, MLCEP20C10, MLBCS20C7L3, MLBCS20C5L5, and MLBCS20C3L7) with a KD2 PRO analyser (Fig. 3). The principle of the KD2 PRO analyser method consists of a handheld controller and sensors that can be inserted into the sample. Sensors measure thermal conductivity, resistivity, volumetric specific heat capacity and diffusivity. Thermal measurements were carried out on blocks of 90 days of age.

The porosity of samples were determined according to the relationship that links the apparent density and the grain density as follows:

$$n = \left(1 - \frac{\rho_d}{\rho_s}\right) \times 100 \quad (1)$$

where ρ_d and ρ_s are the apparent and grain densities respectively. The value of ρ_d was determined through the water immersion method. The sample brick was initially dried in oven at 105 °C, then it was broken in pieces of suitable dimension (between 1 and 10 cm). Obtained pieces were coated with paraffin in order to prevent water absorption, weighed, then lowered in a beaker with a known amount of water. The volume variation was then measure for assessing the volume of the brick fragment, and the ratio between the dried mass and the volume was used for determining ρ_d . Grain density ρ_s was measured with a gas pycnometer according to the standard ASTM D5550-06 "Standard Test Method for Specific Gravity of Soil Solids by Gas Pycnometer".

Hydric characterisation

Hydric characterisation of blocks was determined through water absorption test (until saturation), and capillary rise tests. Water absorption tests were carried out on samples MC10, MC7L3, MLAGA20C10, MLBCS20C10, MLCEP20C10, MLBCS20C7L3,

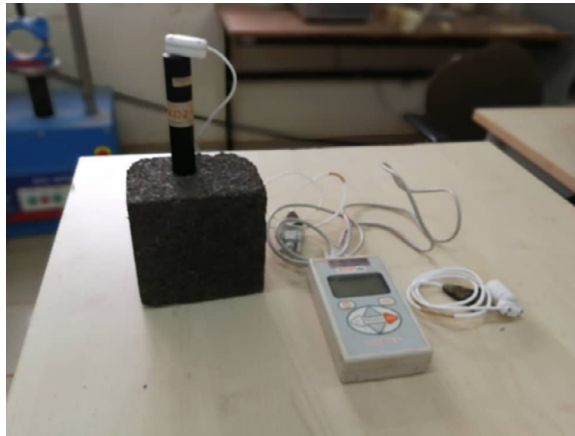


Fig. 3. Testing equipment for thermal properties measurements KD2 PRO.

and MLBCS20C3L7. Blocks were firstly oven-dried at 105 °C for 24 h before the test, then weighted to determine their dry mass. For each mix, different blocks were tested (each one for a specific immersion time). Blocks were immersed in water for 30 s, 1 min, 3 min, 5 min, 30 min, 60 min, 90 min, 2 h, 3 h, 4 h, and 24 h. After each time step, blocks were removed from water, wiped with a cloth, and then weighted for determining the mass of absorbed water.

The procedure was repeated at each stage for a total test duration of 24 h.

The degree of water absorption was calculated as

$$Hp = \frac{G_{ab} - G_s}{G_s} \% \quad (2)$$

where G_{ab} and G_s were the masses of moist and dry samples respectively. Results were plotted against time.

The capillary rise test was carried out on samples MLAGA20C10, MLBCS20C10, MLCEP20C10 to determine the maximum water rise height through the pores of the blocks. The interest in testing these samples was due to their potential for replication in terms of use of local lateritic soil and suitable compressive strength. Blocks were first dried in oven at a temperature of 40 °C until a constant mass was obtained, then cooled down to room temperature for 6 h. Blocks were subsequently partially immersed 5 mm under water level for 24 h. Marks were drawn on the water containers as reference for keeping the water level constant during the experimentation. The water-to-block contact surface was 14 cm x 9 cm (see Fig. 4).

Fire resistance

Fire resistance tests were carried out at the Laboratory of Mechanics and Materials of Civil Engineering of the University of Cergy-Pontoise (France) on samples MLCEP20C10 (i.e. the soil-blended sample giving the highest compressive strength) cured for 90 days. Tests were carried out following the same procedure suggested for concrete in accordance with the process described below [29]. Blocks underwent heating-cooling cycles in a furnace as per the following heating profiles:

- The temperature was increased at a rate of 0.5 °C/min until reaching the desired temperature (first phase).
- The temperature in the furnace was kept constant for about an hour in order to ensure a uniform distribution of temperature within each block (second phase).
- The temperature was decreased at the average rate of 0.5 °C/min until reaching room temperature (third phase).

Bricks were equipped with thermocouples in order to control the actual test temperature on their faces. Mass loss and residual compressive strength were measured at the end of the cycle. Three temperature levels were investigated, i.e. 200 °C, 400 °C and 600 °C (see Fig. 5). As earth blocks were intended for low-cost housing, those three temperatures were retained in comparison to concrete products, which typically show no residual strength at 800 °C [30]. 21 samples were tested, including 5 control samples tested at room temperature (i.e. 20 °C). Compressive strength of blocks exposed to high temperature was measured with an Instron press following the standard NF EN 772-1.

Results and discussion

Compressive strength

Results from block compressive strength testing on the investigated mixes at 28, 60, and 90 days of curing are shown in Fig. 6. As a general observation, the strength seemed to show an increasing trend with time, being samples at 90 days on average stronger than samples at 60 days (samples MC10, MLBCS20C5L5 and MLBCS20C3L7 did not follow this trend).

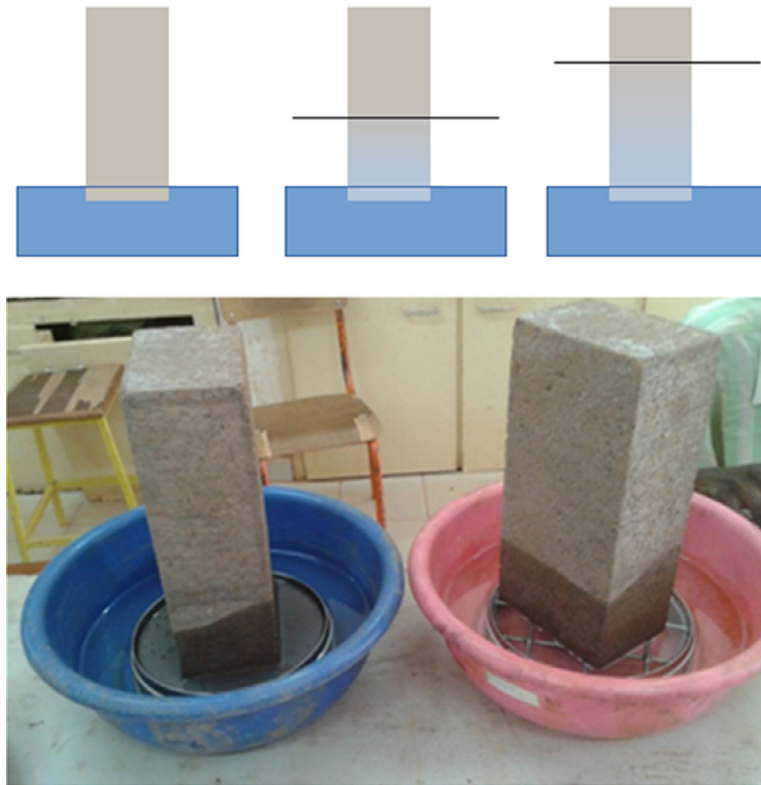


Fig. 4. Capillarity rise test. Top: sketch of the test. Horizontal lines represent the capillarity rise that was measured. Bottom: testing of two blocks.

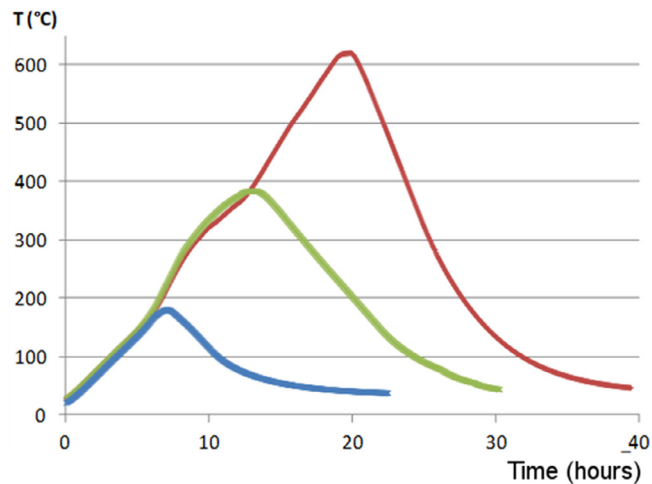


Fig. 5. Evolution of the temperature as a function of time during the fire resistance tests.

However, it is to be noted that error bars (which represent the standard deviation) are often (if not always) overlapping, suggesting that the strength increase after 28 days and up to 90 days is not significant (variation are in the range of 0.5 MPa). The strength decrease at 90 days observed for samples MLBCS20C5L5 and MLBCS20C3L7 can be explained as statistical variation. The strength decrease for sample MC10 is more pronounced and might be due to other reasons. Mix MC10 was produced with the highest water content, and drying in hot climate might have resulted in microcracks in the binding matrix.

Batch 1: the obtained compressive strength values were in line with those found in the literature for cement-stabilised earth blocks [10,19,32]. Results were also similar to those obtained in [20] for neat BA block stabilized with cement (mix MC10). Blending Portland cement with lime in samples of neat BA resulted in a decrease of the mechanical strength. MC7L3

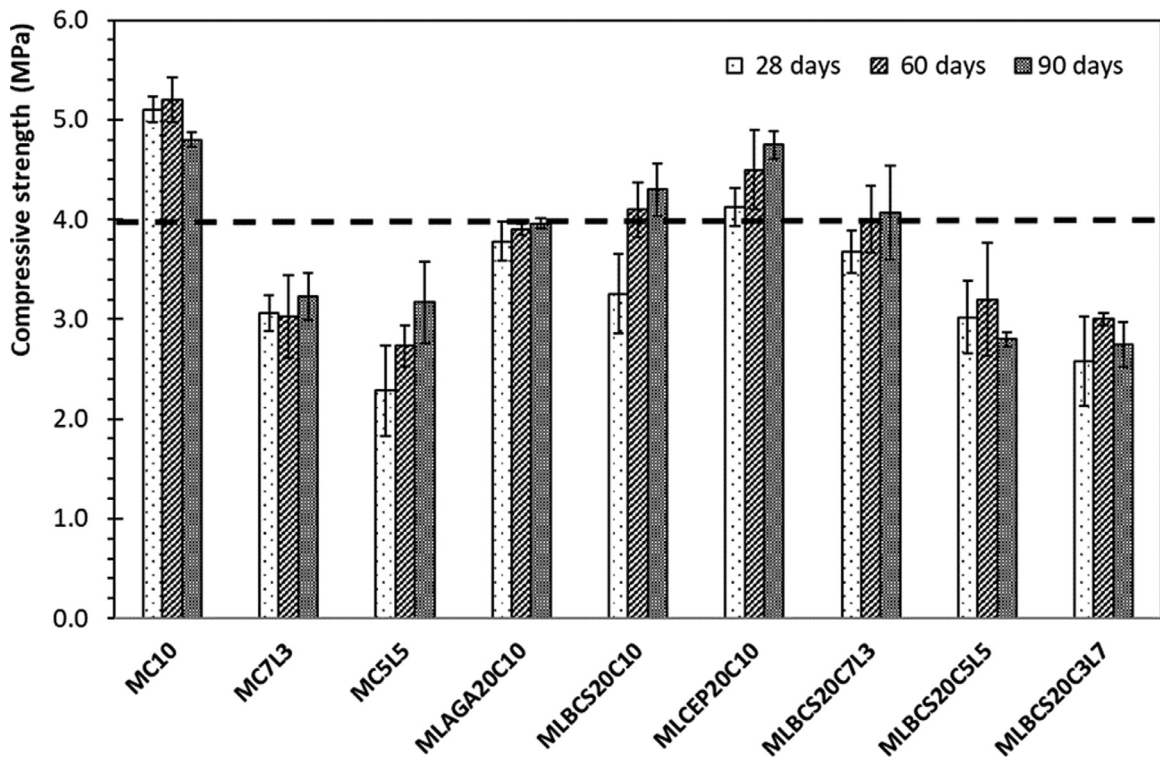


Fig. 6. Compressive strength of tested blocks at 28, 60, and 90 days of curing. The dashed line shows the reference value of 4 MPa as suggested by Craterre [23].

and MC5L5 formulations showed a significant strength decrease when compared to MC10, in the range of 40% and 50% respectively (results at 28 days). This decrease at early age can be explained considering that the production of calcium-silicate-hydrate compounds (CSH) due to the pozzolanic reaction between lime or $\text{Ca}(\text{OH})_2$ and the silica (SiO_2) in BA did not fully replace the missing CSH due to reduction of cement in the mix. Several previous studies confirmed that the partial substitution of cement with other reactive materials is not always beneficial, as this can change the optimum content of calcium, silicate and aluminium minerals that leads to the most efficient reaction [33–37]. In addition, lime does not replace other reaction products such as calcium aluminate hydrates (due to the lack of Al in lime). Observing the results at 90 days, the strength decrease seemed less pronounced, with a reduction of 33%. This might suggest that lime needs longer time for complete reaction, which might involve not only the production of CSH but also the production of CaCO_3 due to the reaction of portlandite with atmospheric CO_2 [38].

MC7L3 samples (i.e. 5.8% cement and 2.8% lime, volume percentages) showed higher 28-day strength than MC5L5 samples (i.e. 4.2% cement and 4.2% lime, volume percentages), whilst at 90 days results were very similar. This seemed to confirm that the substitution of cement by lime had a negative impact on the mechanical strengths in the short and medium term, whilst the slower pozzolanic reaction between the $\text{Ca}(\text{OH})_2$ and the silica from BA can form additional CSH (and possibly CaCO_3 through reaction with atmospheric CO_2) in the long term. Nevertheless, obtained strengths were lower than the value (4 MPa) recommended by CRATerre for compressed earth block [23].

Batch 2: blocks produced with 8.5% cement and 17.1% lateritic soils matched the target strength after 90 days of curing. Samples MLAGA20C10 and MLBCS20C10 resulted in very similar strength at each testing time, whilst MLCEP20C10 showed significantly higher strength. It was also observed that strengths obtained at 90 days of cure were lower than those obtained by Vinai et al. [20], i.e. 7.72 MPa at 45 days of curing, where a lateritic soil from Burkina Faso (BFA) was used. This observation seems to suggest that mineralogical composition and clay content have a relevant impact on the development of the strength of the blocks. BFA soils had higher clay content (29.2%) compared to soils sourced in Niger, but lower activity index ($A=0.8$). An in-depth study of the effect of clay soils properties on the strength of the blocks would improve the understanding of this result, but this falls outside the scope of this paper. It is interesting to observe that MLAGA20C10 and MLBCS20C10 had similar results in terms of strength despite the grading characteristics of soil LAGA and LBSC were very different. This might be due to the clay mineralogy as both soils had an activity index higher than 1, therefore considered “active” (being $A > 1.25$). This property is typically associated to large shrinkage during drying, and this could have affected the strength of blocks (MLAGA20C10 samples were slightly weaker than MLBCS20C10 samples).

Batch 3: strength results from samples MLBCS20C10 and MLBCS20C7L3 were comparable despite the partial substitution of cement with lime. The introduction of clayey soil in the mix therefore compensated the negative effects of substitution

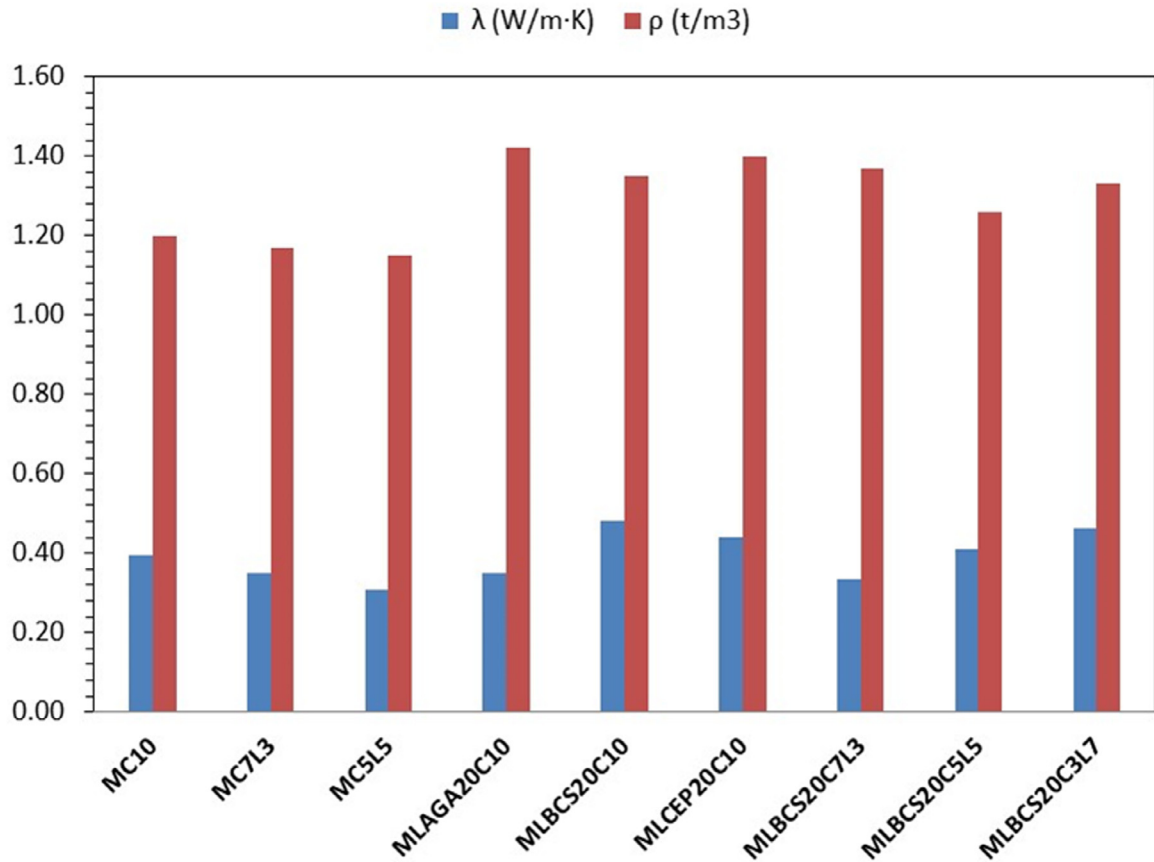


Fig. 7. Thermal properties of produced blocks.

Table 4

Thermal conductivity of typical building materials compared to experimental values obtained for the block produced.

Material	Thermal conductivity (W/m·K)
Mortar [39]	1.40
Clayey or silty soil [39]	1.50
Solid concrete [39]	1.15 – 2.00
Hollow concrete blocks [39]	0.70
Bricks containing lateritic soil with 8% cement [43]	0.75 – 1.15
Bricks with 45% lateritic soil, 45% natural pozzolana, 10% cement [43]	0.65 – 0.71
Bricks with 81% lateritic soil, 9% sawdust, 10% cement [43]	0.50 – 0.65
MC10, MC7L3, MC5L5	0.31 – 0.40
MLAGA20C10, MLBCS20C10, MLCEP20C10	0.34 – 0.48
MLBCS20C7L3, MLBCS20C5L5, MLBCS20C3L7	0.33 – 0.46

of cement with lime discussed previously. A higher substitution rate (see samples MLBCS20C5L5, MLBCS20C3L7) led to a decrease in mechanical strengths, although no significant differences between samples MLBCS20C5L5 and MLBCS20C3L7 were recorded.

Thermal properties

The thermal characterisation method described in section 2.2 was used to determine the thermal conductivity λ and the volumetric heat capacity C . Results from these tests, carried out with the KD2 PRO analyzer at the 2iE Eco-Materials Building Laboratory (LEMC), are shown in Fig. 7, along with the value of the density of the blocks ρ (t/m³).

Values of thermal conductivity for blocks produced with BA (and stabilised with different combinations of cement, laterite and lime) were found to be lower than those found in literature for typical building materials (see Table 4). Common concrete blocks that are typically used for masonry have a thermal conductivity in the range of 0.70 – 1.30 W/m·K, the lower value being representative of hollow blocks. Lower thermal conductivity values are typically obtained when special

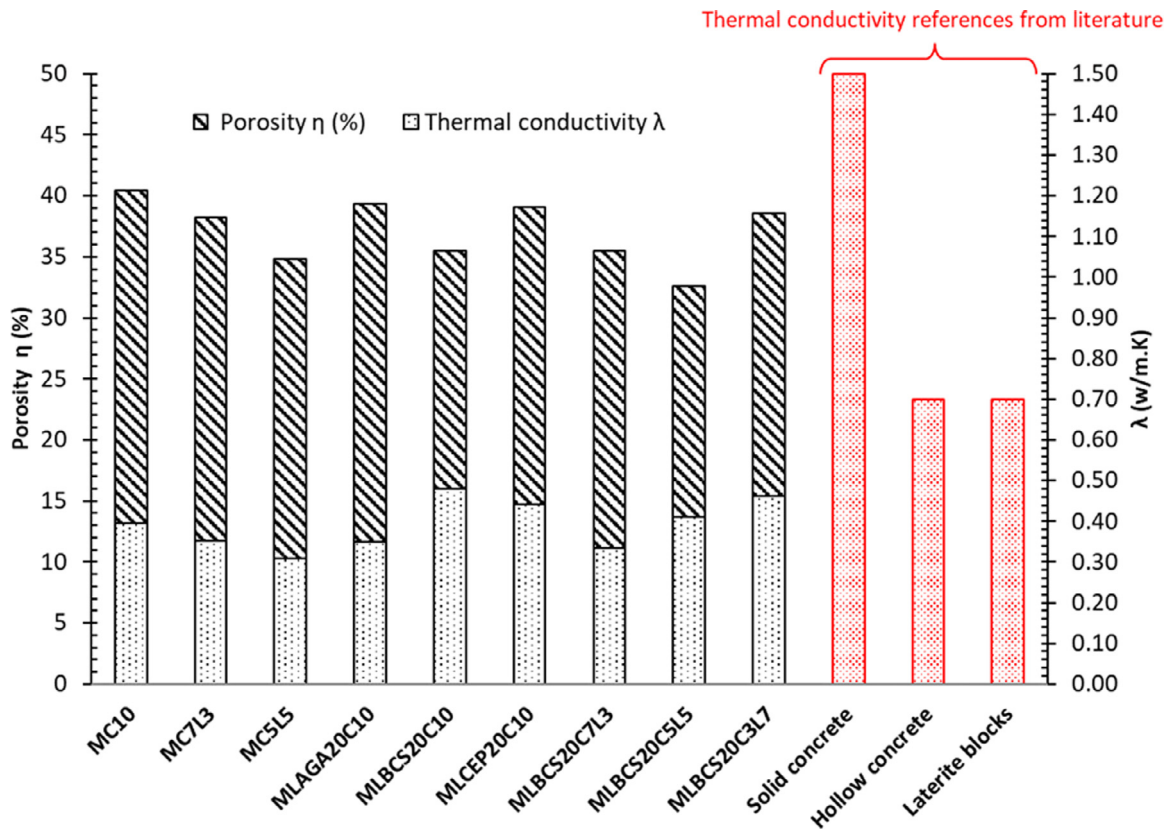


Fig. 8. Porosity and thermal conductivity of investigated samples.

building elements, such as perlite (or other lightweight) aggregate-based blocks or aerated autoclave concrete blocks, are used, although these technologies might not represent a fair benchmark for compressed earth blocks produced in developing countries. Meukam et al. [43] investigated the thermal and physical properties of building materials available in developing countries. Thermal conductivity values were measured in the range of 0.5 – 1.15 W/m.K for bricks produced with different lateritic soil, cement and other constituents. These outcomes suggest that BA-based materials can have superior thermal insulation properties than lateritic earth compressed blocks, see Table 4. It was also observed that measured thermal conductivities were lower than those found in literature for compressed earth blocks (either with or without stabilisation). This result could be due to the dry apparent densities of the blocks produced in this investigation, which were lower than those suggested by the literature. Several studies demonstrated that the variation in thermal conductivity is proportional to the apparent density of bricks [12,29].

The addition of lime seemed to decrease the thermal conductivity of blocks. This is presumably correlated to the density reduction. Formulations with lime gave the lowest thermal conductivity values. The low apparent densities of the bricks can be interesting for non-load bearing structures.

As the thermal conductivity of a material can be related to its porosity, the porosities of samples were investigated and the obtained values are shown in Fig. 8.

The produced blocks had porosity equal to 30 to 40%, irrespective of the mix proportions. Obtained values of porosity and apparent density were in the same range reported in the literature. These latter demonstrated that the increase in compaction pressure led to a more “efficient” granular arrangement of the internal structure and therefore contributed to the decrease of macroscopic and microscopic porosity of the blocks. The high porosity obtained in the present investigation can be therefore justified by the low compaction pressure applied by the hand press, which was significantly lower than that of hydraulic presses. This property influenced the thermal conductivity values, which were low and therefore of interest for the production of materials with high thermal insulation potential. The optimum (50%) rate of substitution of cement with lime seemed to reduce the porosity of blocks (MC10, MC7L3, MC5L5 and MLBCS20C10, MLBCS20C7L3, MLBCS20C5L5, MLBCS20C3L7). It could be explained by the effect of deposition of $\text{Ca}(\text{OH})_2$ reaction products (with silicate or with CO_2) into the pores of the blocks. Further detailed investigation would need to be carried out for confirming this hypothesis.

Irrespective of the laterite source, porosities of blocks stabilised at 17% and 8.5% were all in the same range, although the porosity of samples MLBCS20C10 was slightly lower than the other two mixes. This result could be due to the higher presence of fine fraction (78% of sample mass passing the sieve 75 μm , see Table 2), which could have influenced the final

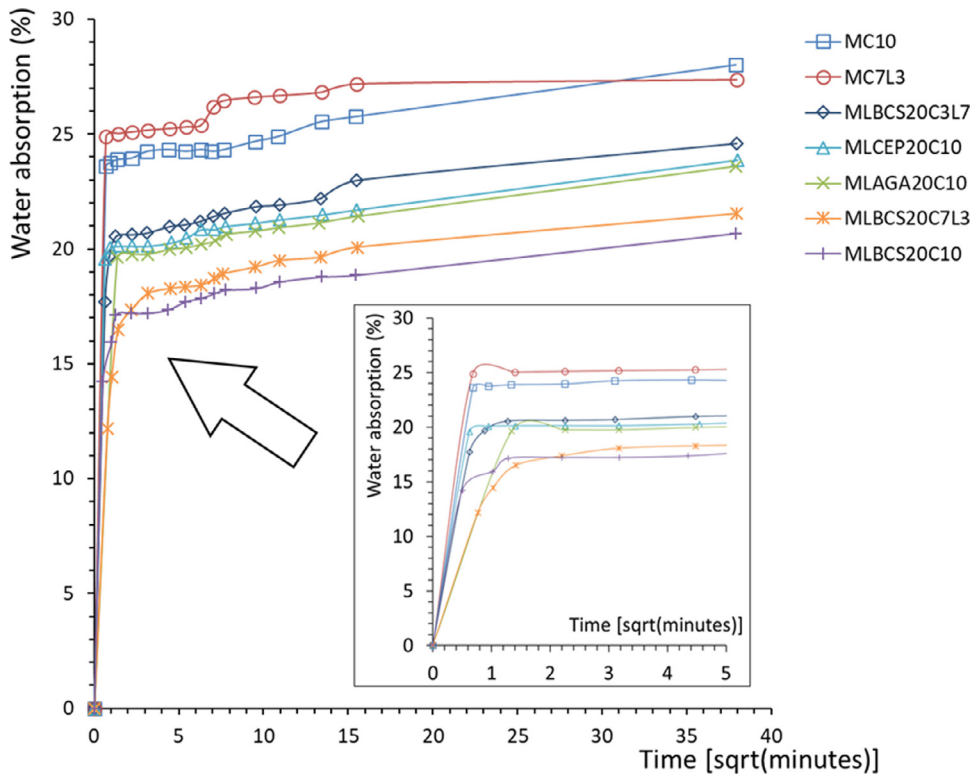


Fig. 9. Kinetics of the water absorption for some of the investigated blocks.

porosity. The other two soils MLAGA20C10 and MLCEP20C10 had fine fraction (i.e. sample mass passing the sieve 75 μm) equal to 22% and 46% respectively.

No clear trend was observed when plotting the thermal conductivity vs. porosity, although some kind of correlation was expected. This might be due to the high porosity values, which led to similar thermal conductivities in the range 0.31 to 0.48 W/m.K. The lack of clear trend between porosity and thermal conductivity might also be due to the overcoming effect of other physical properties of the blocks and of the mix constituents. From data shown in Fig. 7, it was observed that a quasi-linear relationship existed between thermal conductivity and apparent density of blocks, with only two outlying mixes (MLAGA20C10 and MLBCS20C7L3) that showed thermal conductivities lower than expected. Albeit in the case of MLAGA20C10 this evidence could be ascribed to the relatively high porosity, further investigations are requested to understand the complex relationships driving the final thermal conductivity. Bhattacharjee and Krishnamoorthy [44] suggested that trends between porosity and thermal conductivity of construction materials become apparent only for porosity > 50%, and therefore thermal conductivity depends not only on the porosity but also on the solid type.

Hydric characterisation

As discussed in the Introduction section, the behaviour of compressed earth blocks when in contact with water is one of the most critical issues for their durability and serviceability. For this reason, test of water absorption and capillarity rise were carried out on investigated blocks in order to gain understanding over the water/block interaction. The characterisation was focussed on the blocks with satisfactory strength results. MC5L5 and ML20BCSC5L5 were not retained for the kinetic of the water absorption test whilst ML20BCSC5L5 were not kept for the water absorption tests.

Water absorption tests

Fig. 9 shows the kinetics of water absorption obtained on some formulations of blocks stabilised with cement, lime and clay materials.

It was observed that the water absorption kinetics of the bricks was very fast in the first minutes of the test due to the high porosity of the blocks. In the first 5 min of immersion, about 80 – 90% of the total water absorption was achieved from the investigated samples.

Final values of water absorption were recorded in the range 20.7% to 28%, with no significant difference among the blocks. These values were lower than the values of the respective total porosities discussed in section 3.3. This result seems to suggest that some of the voids cannot be reached by water ('closed bubbles' from the bottom ash), at least within 24 h

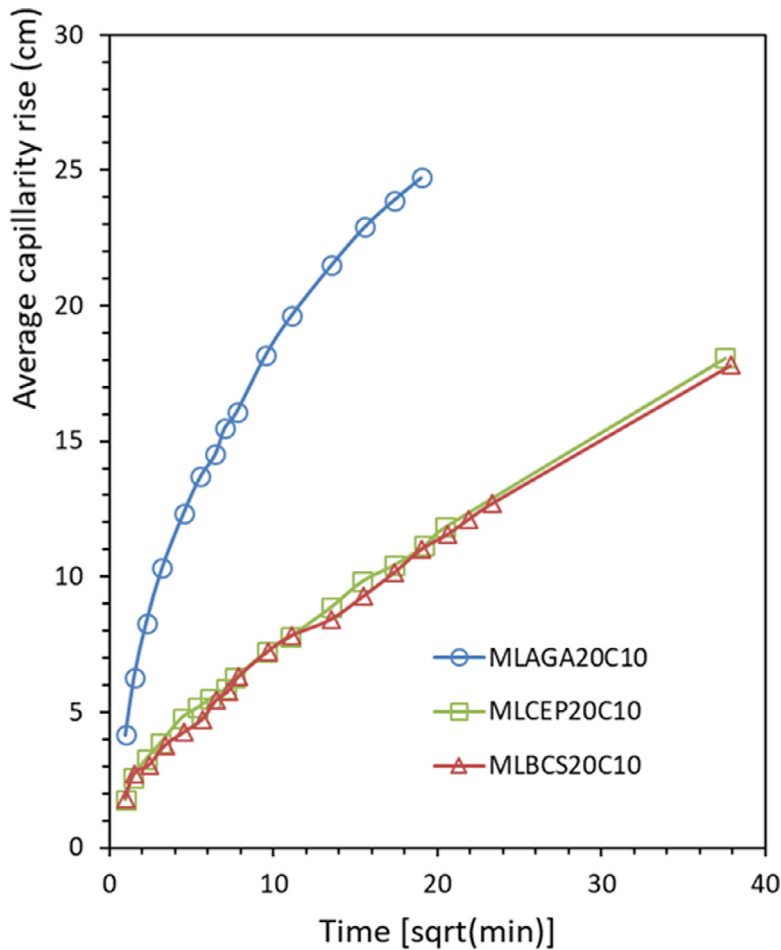


Fig. 10. Measured capillarity rise (average on the four faces of each blocks) vs. time.

of immersion, and therefore the absorption time is very long. It can be observed (see Fig. 8) that the slopes of the experimental curves are still positive at the end of the test, suggesting that the absorption did not reach a stabilised value. These absorption values remain relatively high compared to those found in the literature for soil blocks stabilised with cement and cement/lime blends (15–20% on average) [11,13] and to a common threshold value of 18% for block to be used for masonry applications. This might be due to the nature of BA and its intrinsic porosity (a 'popcorn-like' structure of the grains, which have inclusions of air). The addition of clay materials seemed to decrease the absorption value of blocks compared to those of neat BA blocks stabilised with cement (MC10). Due to the intrinsic porosity of BA, the reduction of BA quantity (MC10 equal to 79% of BA in volume) might have decreased the absorption when lateritic soil was used for the production of the blocks (68% of BA in volume). In addition, lateritic soil used in this study had a significant amount of fine particles that could have reduced the pores in the structure in comparison to the blocks containing only BA. The block production process, i.e. the mechanical hand press used in this study, might also have had a detrimental effect on the porosity of blocks, which could possibly be overcome by the use of hydraulic press.

Capillarity rise test

Capillarity rise tests were carried out only on blocks produced with BA stabilised with cement and lateritic soil from three sources. These formulations were retained because of their good mechanical properties and their low water absorption compared to the others blocks. Fig. 10 shows the obtained results as the average of capillarity rise heights measured on the four faces of each brick. Two blocks per each formulation were tested. The measured values showed significant differences on the four faces of the blocks (Fig. 4). This effect might be due to the block production process, which could not ensure full homogeneity of the blocks in each section. More blocks tested would increase the reproducibility of the capillarity rise tests.

It was observed that the maximum water capillarity rise was measured on sample MLAGA20C10, exceeding 25 cm before 24 h. On the other hand, samples MLBCS20C10 and MLCEP20C10 gave very similar values of approximately 18 cm after 24 h. Since the porosities of mixes MLAGA20C10 and MLCEP20C10 were very similar, the justification for the observed difference

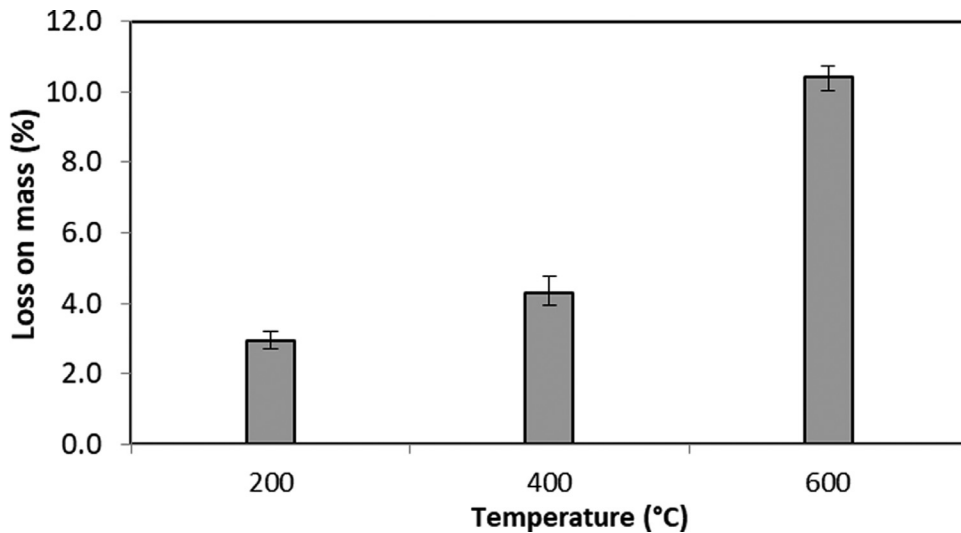


Fig. 11. Mass loss vs. temperature for the blocks MLCEP20C10.

in capillarity rise might be related to the different grain size distribution of the two soils. Soil AGA was found to be the coarser among the pre-selected materials, and this property might have influenced the behaviour of blocks versus the capillarity rise. As the capillarity rise height is inversely proportional to the pore channel dimension, it is to be assumed that pore channels in sample MLAGA20C10 were smaller than the ones in mixes MLCEP20C10 and MLBCS20C10, but presumably more diffused. The structure of the materials was influenced by the quality of the static compaction of blocks during production, which was in turn influenced by the geotechnical properties of the soils used in the mix. Silt fraction in soil LAGA was lower than the in other two soils, whilst the activity index was the highest. These conditions might have led to a block structure with a network of pores that resulted in greater capillarity rise, and this could explain the high porosity and the low compressive strength of blocks produced with LAGA soil. Soil LCEP had the best grain sizes distribution grading and the lowest activity index. Blocks produced with soil LCEP gave the highest compressive strength and the lowest capillarity rise, although the water absorption was measured similar to that of samples produced with soil LAGA.

The high values of capillarity rise would suggest to avoid using these blocks in lower parts of building, such as basement, i.e. in areas with direct contact with groundwater. However, simple surface treatment such as the application of a layer of waterproof paint would reduce water absorption and capillarity rise.

Fire resistance

In order to assess the mechanical behaviour of blocks subjected to high temperature, fire resistance test were carried out on samples produced with the mix MLCEP20C10, being the one that gave the highest compressive strength (4.8 MPa at 90 days cure) compared to mixes MLAGA20C10 and MLBCS20C10. Mass loss and compressive strength were measured on samples exposed to 200 °C (four samples), 400 °C (six samples), and 600 °C (six samples) and results were averaged.

Results of mass loss measurements are shown in Fig. 11.

As expected, the higher mass loss was recorded at 600 °C and it was 10.4%. Mass losses at 200 °C and 400 °C were 2.9% and 4.3%, respectively. Although it was not possible to run a complete thermogravimetric analysis, the following interpretation to the obtained data was proposed:

- Up to 200 °C, the mass loss was mainly due to the evaporation of free water and to some initial chemically bound water from cement reaction products, such as AFm phase.
- From 200 °C to 400 °C, the further dehydroxilation of CSH gel as well as decomposition of unreacted portlandite (i.e. calcium hydroxide) was presumably ongoing.
- From 400 °C to 600 °C, 60% of the total mass loss was recorded. This was presumably due to the completion of chemically bound water removal in CHS, the thermal destruction of clay minerals from lateritic soil, the burning of any residual organic content in the soil and the de-carbonation of CaCO_3 that could be available both in the soil and as reaction product of calcium hydroxide with atmospheric CO_2 .

As a matter of comparison, most of the mass loss of a concrete occurs between temperatures of 150 °C and 300 °C [40]. Strength reduction rate for standard concrete, calculated as the ratio of compressive strength at the target temperature over compressive strength at room temperature of samples at the same age of curing, is estimated to be around 90% at 300 °C. [30].

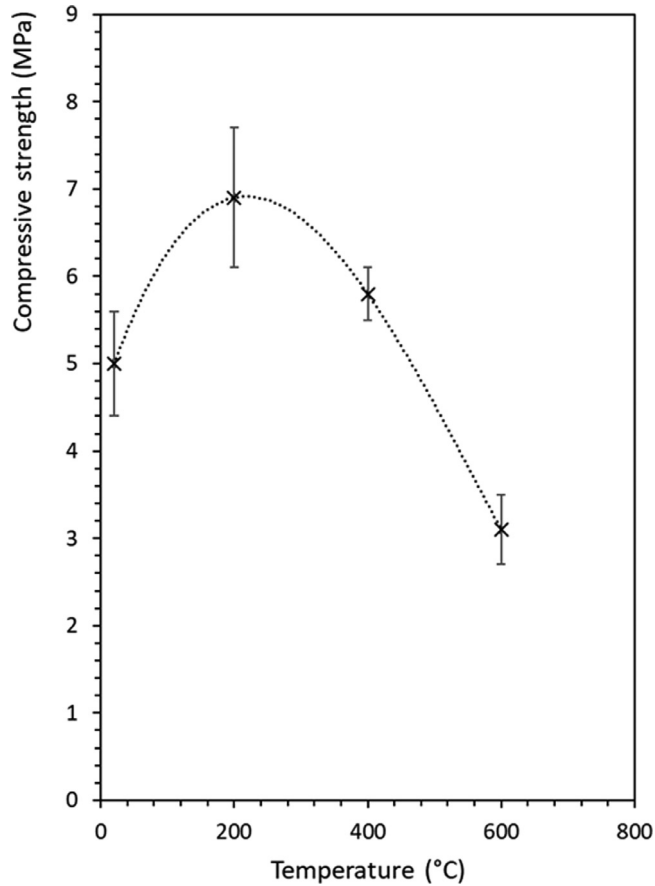


Fig. 12. Compressive strength versus heating temperature. Sample MLCEP20C10.

Compressive strength of blocks was tested at room temperature (20 °C) and after the heating cycles (200 °C, 400 °C and 600 °C), see Fig. 12.

It was observed that after the heating at 200 °C (and to a lesser extent at 400 °C) an increase in the residual compressive strength compared to its value at ambient temperature was measured. Compressive strength increased by 40% and 15% at 200 °C and at 400 °C (although in this latter case a significant overlapping of the error bars was found). The thermal treatment might have resulted in further reactions which involved silicate and aluminate from BA, such as geopolymerisation i.e. the production of amorphous silicoaluminate gel which does not depend from hydration and therefore it is inherently resistant to heating process. After the heating at 600 °C a strength decrease of about 40% was measured. This result is due to the complete decomposition of C-H-S binding gel. It is interesting to observe that the relative residual strength at 600 °C was higher than the average relative residual strength of a concrete at the same temperature [40–42]. This might represent an indirect confirmation that other reaction products due to the chemical nature of BA were present in the blocks.

The visual observation carried out on BA-based blocks after heating cycles (see Fig. 13) suggested that:

- After the heating cycle at 200 °C, no colour change was observed on the samples when compared to the control samples kept at room temperature.
- Progressive blushing was observed after the 400 °C heating cycle, which was more pronounced after the 600 °C heating cycle. The red coloration observed from 400 °C could be related to the dehydroxilation of goethite (iron hydroxide) contained in the laterite [22] which transforms into ferric oxide (Fe_2O_3) at a temperature of 350 °C.
- No macroscopic cracking of the samples was visible after the heating cycles. However, a more detailed study of the microstructure of blocks as a result of different thermal treatments would provide better insights on the presence of cracks in the structure (e.g. observations with scanning electron microscope technique).

The fire resistance of the blocks produced with BA, cement and lateritic soil was considered satisfactory, as strength up to 3 MPa were measured after exposure to 600 °C.

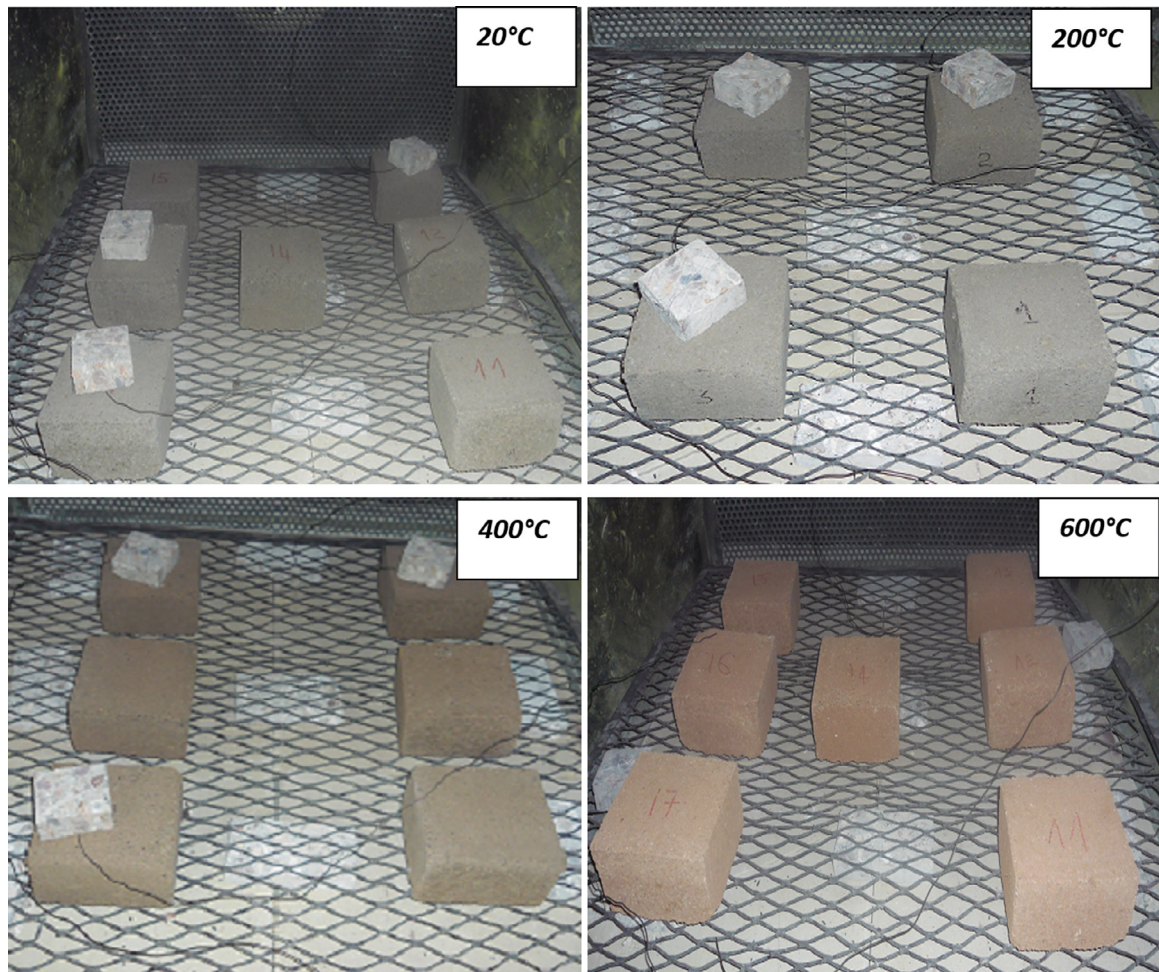


Fig. 13. Blocks MLCEP20C10 after exposure to different temperatures.

Discussion

The range of tests carried out under this investigation allowed to answer the research questions presented in Section 1.2. Several parameters were varied in the mix proportions for the production of compressed blocks made with BA, i.e. the effects of stabilisation with Portland cement, the use of a stabilisation binder blend where Portland cement was partially replaced with lime, the use of locally available lateritic soils, and the combined effect of binder blend and locally sourced soils.

Results from compressive strength tests demonstrated that it is possible to produce compressed blocks made with stabilised BA, either as a sole constituent or blended with locally sourced lateritic soil, with desirable properties. Mixes MC10 (i.e. neat BA stabilised with Portland cement) and mix MLCEP20C10 (i.e. BA blended with a fraction of lateritic soil and stabilised with Portland cement) were fully compliant with the CRATerre recommended threshold of 4 MPa. Samples MLBCS20C10 and MLAGA20C10 (i.e. BA blended with a fraction of different lateritic soils and stabilised with Portland cement) were suitable although with a narrower margin. When the Portland cement was partially substituted with lime, the compressive strength of the blocks decreased, and only the mix MLBCS20C7L3 was able to match the target strength after a curing time of 60 days. Produced blocks also showed excellent thermal insulation properties, with thermal conductivities measured in the range of 0.4 W/m·K, significantly lower than thermal conductivities of common building materials. Although a complete economic and environmental analysis was outside the scope of the study, it can be observed that the local availability of the main constituents (BA and lateritic soil) and their low economic value (BA in particular is a by-product that would possibly have a “negative” cost, if the alternative scenario was to dispose it as a waste) make this a low-cost and low-carbon building material that is appropriate in the local context. Blocks showed mechanical strengths that can compete with traditional building elements (produced with higher Portland cement content), but with superior thermal insulation properties. The possibility of substituting a fraction of Portland cement with lime, although only partially successful, would

further improve the environmental profile of the blocks, and at the same time can reduce the production costs. The use of BA as constituent for the production of compressed blocks can therefore be considered a technically viable solution for the recycling of such industrial by-product when the local context (availability, acceptance and demand) is favourable.

The nature and properties of the lateritic soils used for sample preparation had a significant impact over a number of physical and mechanical characteristics of the blocks. Under the same production conditions in terms of stabilising binder and mix proportions, blocks produced with soil LAGA showed the lower strength, the higher porosity, the higher water absorption and the higher capillarity rise. This seemed to be related to the grain size distribution (the silt fraction in LAGA soil was lower than in other soils) and the activity index of the clay minerals (LAGA clay had the highest activity index among the investigated soils). These factors had repercussions on the quality of the static compaction during block production and therefore on the mechanical properties. On the contrary, soil LCEP showed the best grading in terms of grain size distribution (with a better balance between silt and sand fractions), and the lowest clay activity index, which resulted in high compressive strength and low capillarity rise. Conversely, the water absorption was similar to that of sample LAGA. Soil LBCS, having intermediate geotechnical properties, resulted in blocks of intermediate physical and mechanical quality.

The most promising soil-blended blocks in terms of compressive strength (MLCEP20C10) underwent fire test, being exposed to high temperatures in a furnace. Results suggested that the strength reduction for temperatures up to 600 °C was acceptable, whilst the strength at 200 °C and 400 °C was even higher than the initial one. This might be due to the triggering of other reactions in the binding matrix (presumably the aluminosilicate gel formation) due to the chemistry of the bottom ash, as the test temperatures were lower than the clay sintering point. Electronic microscope and microstructural analysis would be needed to confirm this hypothesis and give further insights on the effect of thermal treatments.

Conclusions

Resource scarcity, population growth, need for recycling industrial by-products and environmental impact of Portland cement-based materials are all drivers for the development of alternative building elements for sub-Saharan countries. This paper focusses on the development of compressed bricks produced with bottom ash from coal combustion and clay materials sourced in northeastern Niger, with the addition of Portland cement and lime. Physical, mechanical, hydric and thermal properties of obtained bricks were assessed, as well as the behaviour of bricks in terms of compressive strength when exposed to high temperatures (200, 400 and 600 °C).

The following conclusions were made:

- Blocks with satisfactory compressive strength (> 4 MPa) were produced mixing BA with lateritic soils locally sourced in northeastern Niger and stabilised with a hydraulic binder.
- Stabilisation with Portland cement proved to be the most effective in terms of strength development. Nonetheless, satisfactory results were obtained blending Portland cement with lime (added in the form of liquid suspension) in mixes with BA and LBCS soil, which matched the target strength of 4 MPa after 60 days of curing.
- Thermal conductivities of blocks were measured in the range 0.31 to 0.48 W/m.K.
- Porosity was found to be very high irrespective of the mix constituents, in the range 33% to 40%.
- Water absorption of the bricks was very fast, reaching about 80 – 90% of its maximum value within 5 min of immersion. Total water absorption was measured in the range 20.6 to 28.7%.
- Capillarity rise was measured on samples produced with a blend of BA and lateritic soils, showing significant rise (between 17 and 25 cm) after 24 h.
- Geotechnical properties of soils used in the mix have significant impact on physical and mechanical properties of the blocks. Clay mineralogy and grain size distribution need to be controlled as these can affect the quality of brick compaction during production, and therefore the void structure, with obvious repercussions on mechanical strength and water-related behaviour of blocks.
- Samples subjected to furnace heating showed promising results in terms of strength and mass loss. Blocks MLCEP20C10 (i.e., the mix leading to the highest compressive strength) showed an increase of compressive strength for temperatures up to 400 °C compared to the room temperature compressive strength. A change in colour of samples (from grey to red) was observed, due to the dehydroxilation of iron hydroxide turning into ferric oxide.
- The inclusion of BA in compressed block production is a viable strategy for the management of coal combustion products in developing countries.

Future research will be needed to investigate the economic and environmental assessment of the developed products, ensuring their viability and social acceptance in the Niger context. Furthermore, the reactions between lime, aluminosilicate in BA and clay, and the effect of blended binder carbonation, need to be investigated.

Declaration of Competing Interest

None

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