

# Optimization of the performance of earth mortars at elevated temperatures

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Beckett, C., Bras, A., Fabbri, A., Keita, E., Perlot, C., Perrot, A. (eds) Second RILEM International Conference on Earthen Construction. ICEC 2024. RILEM Bookseries 52, 387-397. Springer, Cham, Switzerland. [https://doi.org/10.1007/978-3-031-62690-6\\_40](https://doi.org/10.1007/978-3-031-62690-6_40)

**Abstract.** This study investigates the behavior of three earth mortars exposed to elevated temperatures, considering the nature of the aggregates used. Earth mortars made from a combination of a silty-clay earth, silica-calcareous sand (S) and fired bricks waste (W) were investigated. One volumetric fractions of sand/earth and two percentages of sand/fire bricks waste substitution were considered and test specimens were prepared for each mortar. After drying to have constant mass, tests were carried out to determine physical and mechanical properties at room temperature and after being subjected to heating-cooling cycles at temperatures of 200°C, 400°C, 600°C and 800°C. Notable observations were made, in particular the negative effect of the silico-calcareous sand fraction after 400°C. At the highest temperature of 800°C, mortar containing sand showed significant degradation due to decarbonation of the calcareous aggregate. The effect of using fire bricks waste as additional aggregate was very positive. The results obtained can be used to optimize the performance of earth mortars at elevated temperatures.

**Keywords:** Earth mortar, Fire bricks waste, Residual properties, Fire performance.

## 1 Introduction

Awareness of the environmental impact of the construction sector has led to efforts to reduce its carbon footprint. Against this backdrop, there is a growing interest in traditional construction methods, and in particular on earth construction. There is also a growing interest in the use of clay-based plastering mortars for the passive regulation of internal relative humidity and temperature [1]. Earthen mortars are 100% reusable, non-toxic and release no volatile organic compounds (VOCs) in the air. They can be made just mixing a raw earth, extracted from the soil, with water. The clay fraction of the earth is their binder. To control the shrinkage of earth mortars, and depending on the raw earth composition (types and fractions of clay, silt and sand, after removal of coarser particles), often additional sand and/or vegetal fibres can be added. As sand is not an inexhaustible resource, the use of fired brick waste as a substitute for sand could be an advantageous option. This would further improve the environmental performance of earthen mortars. Despite the environmental and other advantages of the use of earth in building, the absence of standards governing the performance of earthen structures and products, namely in terms of fire safety, is a barrier to its use. Most studies of the effect of clayish materials on high temperature are focus on fired ceramic clay bricks [2–5]. In general, the compressive strength of fired clay bricks is greater than that of unfired clay bricks. This increase in strength is due to the dehydration and dehydroxylation of clay minerals between 20°C and 600°C [3,6] and to the vitrification of the clayey matrix between 600°C and 800°C [3,5]. The materials used in clay-based mortars are more diverse and generally contain a higher proportion of aggregate added to the earth (sand or wastes with similar particle size distribution) than the clayish earth itself, also higher than the one used to make fired bricks. The aim of this study is to evaluate the effect of sand and its substitution by fired brick waste on an earth mortar. The earth mortars to test will be based on a clayed silty earth, natural sand and fired bricks waste, and the objective is that it can be used as interior plastering mortars to limit heat transfer in a fire situation. As a first step, various mortars were formulated and their properties in the fresh state and hardened at room temperature were determined. Secondly, the prepared mortar specimens were subjected to various heating-cooling cycles from room temperature to 800°C for residual characterisation.

## 2 Materials and Methods

### 2.1 Materials

The materials used in this study were provided by french brickyard ‘‘DeWulf’’. Earth mortars were made with a silty clay earth, a silica-calcareous sand (S), and fired brick wastes with two granular classes of 0-2 mm ( $W_{(0/2)}$ ) and 0-4 mm ( $W_{(0/4)}$ ). The limits of liquidity, plasticity and the plasticity index of the silty clay earth were 31%, 19% and 12%, respectively, according to standard EN ISO 17892-12[7]. The dry bulk densities of the materials used (Table1) were determined after drying at 110°C in an oven to constant mass, according to standard EN 1097-3[8], and that of the silty clay

earth was determined after drying to constant mass in an oven at 40°C. The water absorption of the aggregates were determined according to standard EN 1097-6[9] and the results are presented in Table 1.

**Table 1.** Raw material characteristics (mean and standard deviation).

	Sand <b>S</b>	Brick waste <b>W_(0/4)</b>	Brick waste <b>W_(0/2)</b>	silty clay earth
Water absorption (%)	1.0 ± 0.02	3.5 ± 0.03	1.85 ± 0.02	-
Dry bulk density (g/cm <sup>3</sup> )	1.74 ± 0.01	1.29 ± 0.01	1.25 ± 0.01	1.24 ± 0.0

## 2.2 Mortars, fresh characterisation and samples

A control mortars (M-2.5S) were prepared with sand and silty clay earth and two mortars were prepared from fired brick waste by substituting a volume of 50% (M-2.5SW) and 100% (M-2.5W) sand for the M-2.5S mortar formulation. (2.5) represents the volume of sand used for (1) volume of silty clay earth. (W) represents the mixture of 50 % (in volume) of W\_(0/2) and 50 % of W\_(0/4) fired brick wastes, allowing to have a similar particle size distribution as the sand (S). (SW) represents the mixture of 50% (in volume) of the sand (S) and 50% of of the two fired bricks waste (W). Preliminary tests were carried out on the different mortars to determine the total quantity of water required to achieve a relatively constant spread (considered workable with 129 ± 5 mm). Table 2 summarizes the different mortars formulations and the quantity of water added when mixing the dry materials.

**Table 2.** Composition of the earth mortars for a volume of earth.

Mortars	Earth	Sand <b>S</b>	Brick waste <b>W</b>	Water (wt %)
M-2.5S	1	2.5	-	17.8
M-2.5SW	1	1.25	1.25	19.3
M-2.5W	1	-	2.5	24.1

**Specimens preparation:** After mixing, two types prismatic specimens were manufactured: 4 x 4 x 16 cm<sup>3</sup> specimens to obtain linear shrinkage, mass loss, bulk density and mechanical strength; and 4 x 7.5 x 9 cm<sup>3</sup> specimens to obtain thermal conductivity. Specimens were stored in the laboratory at a temperature of 22 ± 4°C and a relative humidity of 60 ± 5% until constant mass which was obtained after 19 days.

### 2.3 Tests on mortar specimens

**Physical, thermal and mechanical properties:** mass loss, linear shrinkage and dry bulk density were carried out according standard DIN 18946[10]. Flexural and compressive strength were carried out using an Instron hydraulic press. The loading rate were 0.007mm/s and 0.013mm/s for the flexure and compressive test, respectively. Thermal conductivity was measured using a HOT Disk TPS 1500 system which is controlled by a computer .

**Heating test:** the mortar samples were subjected to various heating - cooling cycles, from room temperature (20°) to temperatures of 200°C, 400°C, 600°C and 800°C; each heating-cooling cycle consisted of a temperature increase phase at a rate of 1°C/min, a constant temperature plateau lasting two hours and a cooling phase down to room temperature at the theoretical rate of -1°C/min.

**TGA/DSC:** thermogravimetric analysis (TGA) was carried out using a Jupr STA 449 F1 instrument developed by Netzsch; each mortar sample (approximately 100 mg) was heated at a rate of 10°C/min from 20°C to 1100°C in an inert atmosphere; differential scanning calorimetry (DSC) was carried out in parallel with the TGA.

## 3 Result and Discussion

### 3.1 TG /DSC analyses of the raw materials

The results of the TGA/DSC test carried out on the raw materials are presented in Figure 11 as curves of mass loss (TG), the derivative of mass loss (DTG) and DSC curve as a function of temperature. Five distinct zones can be distinguished:

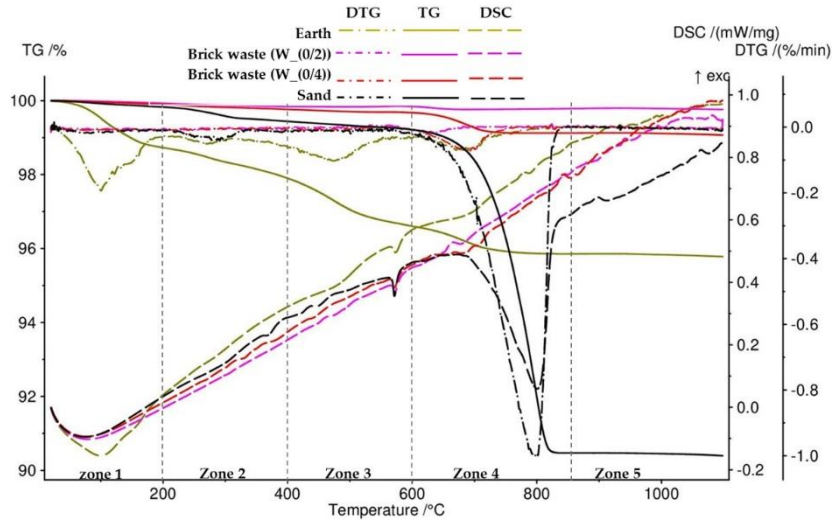
**Zone 1 - [100-200°C]:** In this zone, a mass loss of 1.26% with a DTG peak around 100°C is noticed for the silty clay earth; this mass loss corresponds to the free departure of water around 80°C (free water) and to the removal of adsorbed and interlayer water from clay minerals between 100°C and 200°C [11,12].

**Zone 2 - [200-400°C]:** zone 2 shows a mass loss of 0.82% of the silty clay earth related to a DTG peak around 300°C, which can be attributed to the decomposition of ferrous hydroxides around 300°C into iron oxide such as hematite [13–15].

**Zone 3 - [400- 600°C]:** The third zone, shows a mass loss of 1.30% with a peak on the DTG curve around 500°C. This mass loss can be explained by dehydroxylation between 450°C and 550°C of kaolinite present in the clayey silt to metakaolin [14,15]. A peak on the all DSC curves observed around 572°C is explained by the transformation of quartz- $\alpha$  into quartz- $\beta$  [13].

**Zone 4 - [600- 850°C]:** The fourth zone shows a mass loss of 0.75%, 0.55% and 0.06% for the clayey silt, the fire brick waste W\_(0/4) and W\_(0/2), respectively. The mass loss of the fire brick wastes and silty clay earth, related to a DTG peak around 700°C, can be explained by the dehydroxylation of illite or montmorillonite clay minerals [14–16]. The sand S exhibits a mass loss of 8.75% with a DTG peak around 800°C. This loss of mass can be explained by the decarbonation of calcite (CaCO<sub>3</sub>) [14,15].

**Zone 5 - (beyond 850°C):** In the fifth zone, no transformation was observed.



**Fig. 1.** Thermal behavior of raw materials (silty clay earth, fire brick waste and sand) up to 1200°C during TG/ DSC analysis.

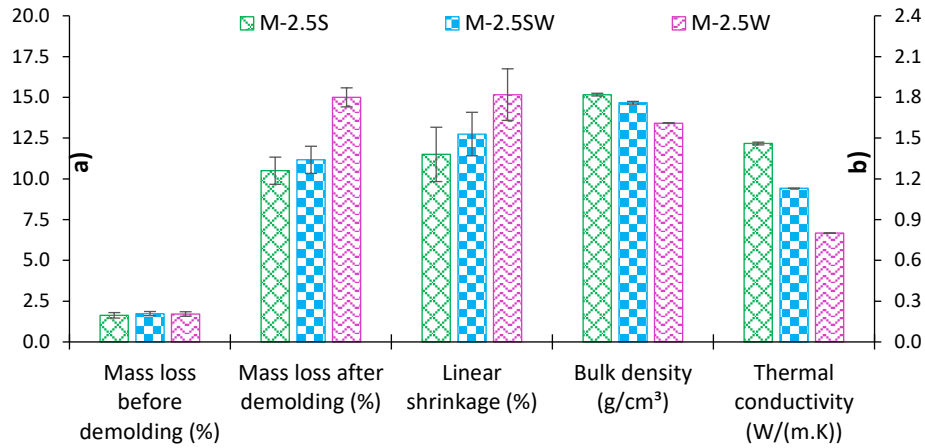
### 3.2 Results at room temperature

Figure 2 below shows the physical and thermal properties at room temperature. The values for mass loss before and after demoulding are shown on axis (a) and the values for linear shrinkage, bulk density and thermal conductivity are shown on axis (b). Before demoulding (after 10 days), the mass losses (axis (a)) are  $2.0 \pm 0.2\%$ ,  $2.1 \pm 0.2\%$  and  $2.1 \pm 0.2\%$  for mortars M-2.5S, M-2.5SW and M-2.5W, respectively. These values are lower than the mass losses after demoulding and stabilization (after 19 days), which are  $12.6 \pm 1\%$ ,  $13.4 \pm 0.9\%$  and  $18.0 \pm 0.7\%$ , respectively. Figure 2 also shows that, the mass losses of the M-2.5SW and M-2.5W (axis (a)) mortar specimens are higher than those of the M-2.5S mortar specimens, probably because their mixing water content is higher.

It can be seen from figure 2 that the final linear shrinkage values (axis (b)) of the M-2.5SW and M-2.5W mortars ( $1.53 \pm 0.16\%$  and  $1.82 \pm 0.19\%$ , respectively) are higher than those of the M-2.5S mortar ( $1.38 \pm 0.20\%$ ). The higher shrinkage of M-2.5W mortar although higher than the other mortars, does not lead to visible cracking. Moreover, this shrinkage is lower than the maximum allowed by the standard DIN 18946[10], which is 2.5% for non-fibre mortars.

The dry bulk densities (axis (b)) shown in figure 2 increase with sand content. This can be attributed to the loose bulk density difference between sand and fired bricks waste. Dry bulk densities, influenced by both mass loss and shrinkage, indicate that M-2.5W mortar is less dense than the others. In fact, fired bricks waste helps to make the mortars lighter. Specifically, M-2.5W mortar is 13% lighter than M-2.5S mortar. This could be an industrial and technical advantage. According to the DIN 18946[10], the

bulk densities class of mortars M-2.5SW and M-2.5W is class 1.8 and for the mortars M-2.5S is class 2.0. Both classes correspond to mortars accepted for the construction of load-bearing masonry. Moreover, the porosity contained in the fire bricks waste leads to a decrease in thermal conductivity (axis (b)), as can be seen in Figure 2.



**Fig. 2.** Physical and thermal properties.

Despite of the lighter nature of mortars containing fired bricks waste, their compressive and flexural strengths are slightly affected as can be seen in Table 3. The mortars with the lowest flexural strengths (M-2.5W) have the greatest shrinkage. It can be hypothesized that drying shrinkage related micro-cracks promote the initiation of failure during the flexural test. Anyway, all three mortars have mechanical strength higher than the minimum values defined in class S-II (compressive strength  $\geq 1.5$  MPa and flexural strength  $\geq 0.7$  MPa) according to DIN 18947[17] for earth plasters, or in class M3 according to DIN 18946[10] for earth masonry bedding mortars, which is clearly positive.

**Table 3.** Mechanical properties.

Mortars	Compressive strength (MPa)	Flexural strength (MPa)
M-2.5S	$3.4 \pm 0.2$	$1.4 \pm 0.1$
M-2.5SW	$3.7 \pm 0.2$	$1.3 \pm 0.3$
M-2.5W	$3.6 \pm 0.3$	$1.2 \pm 0.1$

### 3.3 High temperature behavior and residual properties of mortars

#### 3.3.1 Macroscopic observation.

No cracking was visually observed on the mortar specimens up to 600°C. But at 800°C (Fig. 2) all specimens containing sand were completely cracked in all directions. Except mass measurements, it was not possible to carry out other tests on these specimens. On

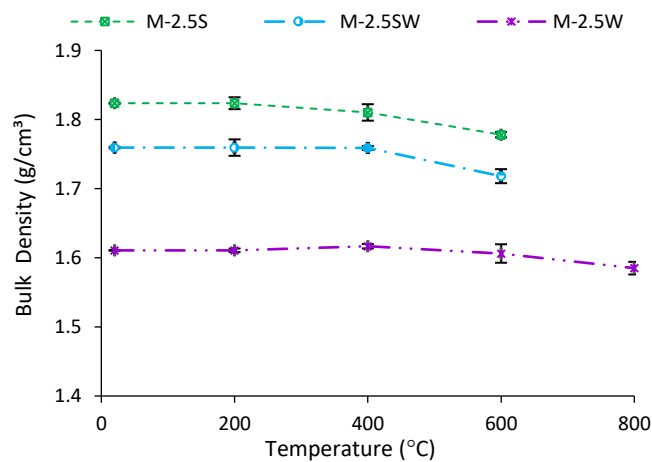
the contrary, M-2.5W mortar specimens showed no visible cracks. M-2.5SW showed finer and fewer cracks than the mortars M-2.5S. The presence of the silica-calcareous sand has a very unfavorable influence on the cracking of the mortar at 800°C.



**Fig. 3.** Mortar specimens after heating-cooling at 800°C.

### 3.3.2 Evolution of Bulk density.

The variation of bulk densities of mortars, assessed after each heating-cooling cycle by the average of three specimens measuring 4 x 4 x 16 cm<sup>3</sup>, is presented in Figure 3. There are no significant changes when all the mortars were submitted to 200°C for mortar M-2.5S, or even to 400°C for M-2.5SW and M-2.5W. After temperature of 400°C all the mortars lose bulk density. The fact that it was possible to measure the density of the M-2.5W mortar after submitted to 800°C is very positive, showing its integrity and resilience in case of fire.

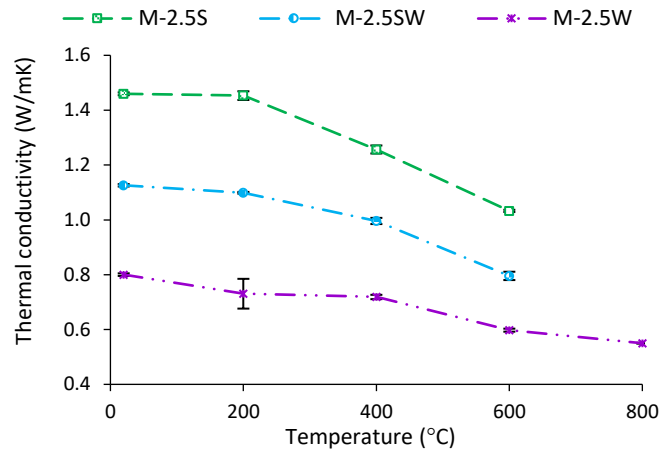


**Fig. 4.** Evolution of bulk densities of mortars after various heating-cooling cycles.

### 3.3.3 Evolution of thermal conductivity.

Figure 4 below shows the evolution of the residual's thermal conductivities of mortars. The decrease in the conductivity is only observed at a temperature of 400 °C at a temperature of 600°C, a decrease of 30%, 29% and 25%, is observed for the mortars M-2.5S, M-2.5SW and M-2.5W, respectively, compared to the values at room

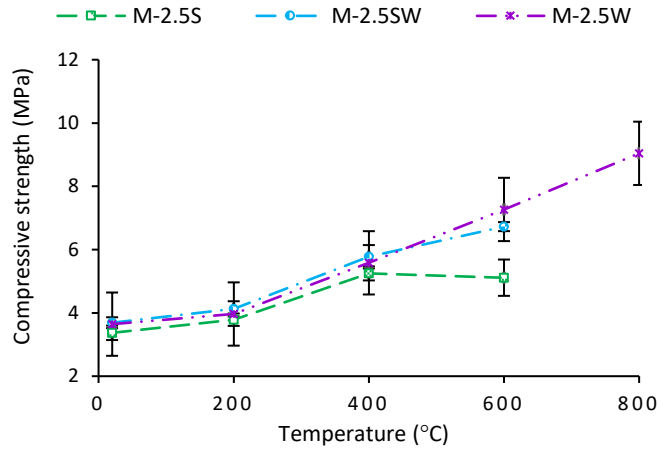
temperature. At 800°C, the thermal conductivity of M-2.5W dropped by 31.26%. The decrease in conductivity could be attributed to the increase in porosity associated with the cracks around the sand aggregates and the microcracks caused by the shrinkage of the clay. These results correlate with the evolution of the bulk densities.



**Fig. 5.** Evolution of residual thermal conductivities of mortars after various heating-cooling cycles.

### 3.3.4 Residual compressive strength.

Figure 5 shows the evolution of the compressive strengths of the different mortars. At a temperature of 600°C, M-2.5S, M-2.5SW and M-2.5W mortars recorded an increase in strength of 52%, 82% and 99% respectively. This increase in residual compressive strength is attributed successively to the dehydration and dehydroxylation of the clay minerals [11,19]. The presence of sand seems to limit the increase in strength with temperature between 400°C and 600°C. This limitation could be attributed to the loss of adhesion between the grains of clayey silt and sand. Indeed, the transformation of quartz- $\alpha$  into quartz- $\beta$  is accompanied by an expansion [13] generating microcracks. At 800°C, the residual compressive strength of M-2.5W mortar increases by 148%.



**Fig. 6.** Evolution of compressive strength after various heating-cooling cycles.

### 3.3.5 Residual flexural strengths.

Figure 9 shows that the flexural strengths of the various mortars decrease from 200°C, except for M-2.5W. At 600°C, M-2.5S and M-2.5SW mortars recorded a reduction in strength of 52% and 36%, respectively. On the other hand, the flexural strength of M-2.5W mortars remains 5% higher at 600°C and decreases by only 7% at 800°C. For the M-2.5S specimens, flexural strength appears to be more affected by sand than compressive strength. This can be explained by the fact that flexural is strongly influenced by the clay matrix/sand interface, as well as by the presence of microcracks after drying. It can also be assumed that the differential thermal deformation between the expanding sand and the contracting clayey silt can generate stresses at the interface during heating, which can weaken the interface bond. It is worth noting that the flexural tensile strength of M-2.5W underwent a slight decrease after 400°C, unlike its compressive strength, which increased continuously between 200°C and 800°C.

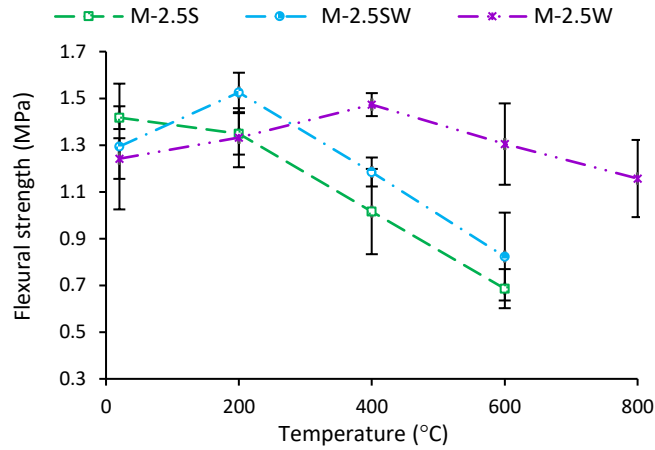


Fig. 7. Evolution of mortars flexural strength after various heating-cooling cycles.

#### 4 conclusion

The effect of the substitution of natural silica-calcareous sand with fired brick waste in three earthen mortars was studied at room temperature and at high temperature.

At room temperature, the study showed that replacing sand with fired brick waste resulted in an increase in linear shrinkage, a decrease and in bulk density without compromising the mechanical performance of earthen mortars. Thermal conductivity was considerably reduced by the use of fired brick waste.

At high temperatures, M-2.5W mortar showed minimal mass loss, while mortars containing sand showed greater mass loss. Thermal conductivity decreased at 400°C for all mortars, and mortar M-2.5SW and M-2.5W always remained lower than mortar M-2.5S. The compressive strength of all samples increased with temperature up to 600°C for mortars containing sand and 800°C for M-2.5W. Flexural strength decreased after 200°C, particularly for M-2.5S. The flexural strength of M-2.5W increased up to 400°C before decreasing slightly up to 800°C.

In short, this study has, on the one hand, shown the limitations of earth mortars formulated with silico-calcareous sand, particularly after 400°C. On the other hand, it has shown that mortars formulated using only waste materials remain stable up to a temperature of 800°C.

#### Acknowledgments

This research was made possible thanks to the EUTOPIA Ph.D. co-tutelle program.

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