



Note

Hardening of clayey soil blocks during freezing and thawing cycles

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ABSTRACT

Considering the lack of references dealing with the behaviour of clayey soil blocks subjected to freezing–thawing cycles, exploratory work has been carried out on such blocks to study the effects of freezing–thawing cycles on their mechanical properties. As the blocks were not stabilized, the procedures conventionally applied for freezing–thawing cycles, in which the samples are immersed, were adapted to this case of study: the specimens were humidified by placing them in a moist environment for 1 week (20 °C and 95% RH (relative humidity)) and were then subjected to freezing–thawing cycles without any re-humidification between cycles. The variations of weight and compressive wave velocity showed that freezing–thawing cycles led to the desiccation of the samples. The consequences of these cycles on the characteristics of the soil blocks were thus those of a conventional desiccation of specimens and, in particular, their hardening. This hardening was highlighted by the study of the mechanical characteristics of the clayey soil blocks (compressive strength and modulus).

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1. Introduction

Earth is one of the oldest building materials and is still the most widely used in many countries in the world. Even today, one third of the human population lives in earthen houses; in developing countries this figure is more than one half (Minke, 2006). In advanced economies, e.g. in south-western France, soil was intensively used for construction until about 1870. After this date, cement was developed and preferred for construction. However, in the last 20 years, soil has been reconsidered as a pertinent material for construction in advanced economies and especially in European countries like Germany, Italy, France and Great Britain or in newly industrialized countries such as India. There are several reasons for this new attraction of earthen building materials in industrialized countries but the most important are the low impact of this material on the environment and the regulation of the hydrothermal conditions of the indoor climate. With the recent keen interest in sustainable development, earthen constructions have become very attractive. The consequence of this has been the appearance of scientific studies on earthen building materials during the last 20 years. However, the number of these studies is relatively small and the main works are summarized in the recent paper by Kouakou and Morel (2009). The most numerous studies concern the mechanical properties of soil blocks (Kouakou and Morel, 2009; Morel and P'kla, 2002; Morel et al., 2007; Reddy and Gupta, 2005, 2006; Walker, 1995, 1999; Walker and Stace,

1997). Other studies deal with the durability of the blocks, the main characteristic studied being their behaviour with respect to water (Guettala et al., 2006; Heathcote, 1995; Ogunye and Bousabaine, 2002a, 2002b; Walker, 1995, 2004). Although earthen building materials are being increasingly used for construction in temperate climates, the behaviour of these materials during freezing and thawing cycles has been studied very rarely. Only Guettala et al. (2006) have studied the behaviour of stabilized blocks in such conditions.

Considering the lack of references dealing with the behaviour of clayey soil blocks subjected to freezing–thawing cycles, an exploratory work was carried out to study the effects of freezing–thawing cycles on their mechanical properties. The blocks under study were not stabilized and this meant that the procedures conventionally applied for freezing–thawing, in which the samples are immersed, had to be adapted. The paper presents the characteristics of soil blocks before and after freezing–thawing cycles adapted to this case of study.

2. Material and methods

2.1. Laboratory analysis

The soil used for this study came from the quarry of a brickworks in southern France. This brickworks produces both bricks and soil blocks but with different compositions, especially for the proportions of clay and sand in the mixtures. These blocks, whether fired or not, are manufactured in the same way: the clay is mixed and crushed with sand and then mixed with 10–15% water. The fresh mixture is extruded to form a long cable of material that is cut into bricks of the desired length. The cut bricks are then hardened by drying for

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nearly 4 days at low temperature (between 50 and 70 °C). For this study, specific specimens were cut at the brickworks with the following dimensions in the fresh state: 5 cm × 5 cm × 10 cm.

The size distribution of the soil determined from pipette analysis for the finer fraction (<80 µm) and from wet sieving for the coarser fraction was: sand (50–2000 µm; 36%), silt (2–50 µm; 28%) and clay (<2 µm; 36%). This soil was considered as a clay loam according to the USDA (United States Department of Agriculture) textural classification system. The chemical composition of the soil determined using Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) was: SiO₂ (64.7%), Al₂O₃ (16.6%), Fe₂O₃ (4.8%), K₂O (4.0%), MgO (1.1%), CaO (1.1%), TiO₂ (0.6%), Na₂O (0.2%), P₂O₅ (0.1%) and MnO (0.1%). The loss on ignition (calcination at 1000 °C) was equal to 5.9%. The main crystalline minerals contained in the soil, determined by XRD analysis on a sample of soil crushed to smaller than 40 µm, were: quartz (SiO₂), muscovite (KAl₂Si₃AlO₁₀(OH)₂), kaolinite (Al₂(Si₂O₅)(OH)₄), orthoclase (KAlSi₃O₈) and goethite (FeO(OH)).

2.2. Freezing–thawing test design

As the tested blocks could lose all their natural cohesion when immersed in water, it was decided to use typically proposed values for freezing–thawing cycles but to thaw the samples in dry conditions. Thus each cycle lasted 12 h and was made up of the following steps: from 20 °C samples were brought to –4 °C in 2 h, the temperature was further reduced to –10 °C in 4 more hours, it was then increased to 20 °C in 3 h, and samples were kept at 20 °C for 3 more hours.

The humidity of the soil blocks played a significant role in their behaviour in freezing–thawing cycles because the damage caused by these cycles was due to the movement of water (in liquid or gaseous form) in the solid or to the transformation of condensed water into ice, with an increase in volume. If the specimens had been dry, as was the case when they left the brickworks, the freezing–thawing cycles would certainly have had no effect. As these blocks were not stabilized, it was not possible to immerse them in water for saturation. Thus it was decided that the specimens be humidified by placing them in a moist environment for 1 week (20 °C and 95% RH (relative humidity)).

2.3. Treatment applied to soil blocks

As shown in Table 1, three series of three specimens were subjected to freezing–thawing cycles. 3 blocks were cured for 1 week at 20 °C and 95% RH and then covered with cellophane to reduce vapour exchanges (“95%*C*”), 3 blocks were cured for 1 week at 20 °C and 95% RH (“95%”) and 3 blocks were “dry” (as they arrived from the brickworks and were kept in the laboratory without specific precautions i.e. in equilibrium with the relative humidity of the laboratory (between 50 and 60%)). After the freezing–thawing cycles, one block of each series was broken to evaluate its compressive strength and its initial tangent modulus. The other two blocks were kept at 20 °C

Table 1
Treatment applied to soil blocks.

Samples	95% HR	Freezing–thawing	95% RH	50 °C
1–95% <i>C</i>	1 week + cellophane	7, 8 and 11 cycles	–	–
2–95% <i>C</i>	1 week + cellophane	7, 8 and 11 cycles	1 week	1 day
3–95% <i>C</i>	1 week + cellophane	7, 8 and 11 cycles	1 week	–
4–95%	1 week	7, 8 and 11 cycles	–	–
5–95%	1 week	7, 8 and 11 cycles	1 week	–
6–95%	1 week	7, 8 and 11 cycles	1 week	1 day
7–“dry”	–	7, 8 and 11 cycles	–	–
8–“dry”	–	7, 8 and 11 cycles	1 week	–
9–“dry”	–	7, 8 and 11 cycles	1 week	1 day
10–“dry”	–	–	–	–
11–95%	1 week	–	–	–
12–95%	1 week	–	–	–

and 95% RH for 1 more week in order to study whether the phenomenon was reversible. It is important to note that, after the freezing–thawing cycles, all the blocks were kept in the same conditions and, in particular, that the “95%*C*” were kept without a cellophane cover. Finally, after this week at 95% RH, one more specimen was broken and the remaining one was placed in an oven at 50 °C for 24 h in order to compare the effects of thermal drying with those of drying observed during the freezing–thawing cycles. At the end of the various treatments, specimens were broken in order to evaluate their compressive strength and initial tangent modulus. The test was run at a constant speed of 0.02 mm.s^{–1}.

2.4. Characterization of damage in the soil blocks

At each step of the treatments applied to the soil blocks, the specimens were weighed, their dimensions were measured and the compression elastic wave velocity (*V_p*) was measured following ASTM standard D 2845–05 (2005).

3. Results and discussion

3.1. The freezing–thawing experiment

Table 2 gives the weight and the compressive wave velocity of the clayey soil blocks.

Fig. 1 presents the variation of the loss of weight (%). The first week of curing at 95% RH led to the same weight gain for the six specimens. It is important to note that, during this phase, the “95%*C*” specimens were not covered with cellophane. The cellophane was only applied during the freezing–thawing cycles.

During the freezing–thawing cycles, the soil blocks had homogeneous behaviour depending on the initial RH with which they were in equilibrium and also on the way they were stored in the climatic enclosure. The group initially wet and not wrapped (“95%”) lost weight very homogeneously and to the greatest extent. Conversely, the group initially wet, but wrapped so as to limit water loss (“95%*C*”) was the least homogeneous group. The difference in weight variation among these samples was greatest in the first series of freezing–thawing cycles and then tended to reduce. These samples lost the least water during the cycles, certainly because their wrapping limited exchanges. For these specimens, the weight at the end of the freezing–thawing cycles was the same as the initial weight.

Finally, the group initially kept in the laboratory without specific drying or humidification (“dry”) also showed only a slight loss of weight and its behaviour was very comparable to that of the “95%*C*” group.

These first results show that the freezing–thawing cycles led to a loss of weight which seemed to correspond to a desiccation of the material. When subjected to re-humidification by being brought into equilibrium with a moist environment (95% RH), all samples gained weight and the mass gain was comparable for all the samples independently of their initial pre-treatment. The behaviour of the “95%” samples was very interesting because their re-humidification under 95% RH led to a weight gain greater than a return to their initial weight. This could indicate that the freezing–thawing cycles (or their drying effect) led to the creation of additional microcracks that could be filled with water.

Finally, when dried at 50 °C for 24 h, all the specimens lost weight. The final weights of samples 6 and 9 were close to the weights they had at the end of the freezing–thawing cycles whereas the loss of water was higher for sample 2.

3.2. *V_p*

Fig. 2 presents the variation of the compression elastic wave velocity (*V_p*) of the clayey soil blocks. As was the case for the variation of

Table 2
Variation of weight and compressive wave velocity.

Samples	0 day		7 days		11 days		15 days		21 days		28 days		29 days	
	m (g)	v_p (m.s ⁻¹)	m (g)	v_p (m.s ⁻¹)	m (g)	v_p (m.s ⁻¹)	m (g)	v_p (m.s ⁻¹)	m (g)	v_p (m.s ⁻¹)	m (g)	v_p (m.s ⁻¹)	m (g)	v_p (m.s ⁻¹)
1–95%C	547.5	1439	550.3	1408	548.0	1535	547.4	1608	547.2	1583	–	–	–	–
2–95%C	550.2	1410	552.9	1410	551.1	1543	550.3	1535	550.2	1522	559.3	1323	546.4	1648
3–95%C	550.9	1372	553.7	1408	553.0	1443	551.3	1544	550.9	1488	561.0	–	–	–
4–95%	538.8	1408	541.5	1658	536.3	1658	534.5	1761	534.9	1769	–	–	–	–
5–95%	556.9	1421	559.8	1666	554.6	1666	553.0	1754	553.2	1727	568.8	1224	–	–
6–95%	548.2	1429	550.9	1736	545.5	1736	543.7	1736	544.2	1755	560.8	1150	543.9	1652
7-“dry”	–	1522	547.9	1676	545.5	1676	544.3	1724	544.9	1714	–	–	–	–
8-“dry”	–	1578	548.3	1690	545.6	1690	544.3	1764	545.2	1737	556.3	1263	–	–
9-“dry”	–	1607	542.8	1705	541.1	1705	539.0	1735	539.2	1740	551.1	1219	539.4	1646
10-“dry”	–	–	–	–	–	–	–	–	–	–	547.2	1448	–	–
11–95%	–	–	–	–	–	–	–	–	–	–	563.6	1202	–	–
12–95%	–	–	–	–	–	–	–	–	–	–	577.0	1180	–	–

weight, the specimens subjected to the same initial conditions behaved in the same way. All samples initially at 95% RH had a very similar initial velocity (between 1400 and 1450 m/s) while the “dried” samples had higher velocities (between 1520 and 1600 m/s).

Moreover, the first set of freezing–thawing cycles led to a larger variation in the 95% RH samples than in those that were kept wrapped. Furthermore, the velocities in these samples after fewer than 20 freezing–thawing cycles (2 sets) were similar to the velocities measured on the “dry” samples that were subjected to the same cycles. The velocities in 95%C samples varied in a way very parallel to those of the “dry” samples. The difference between their initial and final velocities remained about 150 m/s.

When the blocks were subjected to 7 days of 95% RH, all velocities decreased and the amplitude of the decrease was inversely proportional to the velocity value at the end of the freezing–thawing cycles. This means that, the higher the velocity had been, the lower it became after re-humidification. Finally when oven-dried (50 °C), samples presented the same velocity independently of their hydric history.

The last few observations are noteworthy because the elastic wave velocities, and especially that of the elastic compressive wave v_p , provide a non-destructive parameter that gives information on the damage of the sample. It is interesting to compare these results with those of the literature. Numerous studies have been carried out on different sedimentary rocks. Gueguen and Palciauskas (1992) showed that the presence of water (or any other low compressibility fluid) in the pores of the rocks, and thus the degree of saturation, had a strong effect on the value of v_p (Gueguen and Palciauskas, 1992). This effect can be explained by the fact that the compressive wave velocity in

water (about 1500 m/s) is much higher than that in air (about 331 m/s according to Lama and Vutukuri (1978)). It is generally observed that the higher the degree of saturation, the more significant the increase of wave velocity, as shown by the following three examples: Wyllie et al. (1956) quoted by Lama and Vutukuri (1978) measured the wave velocities in three sandstones of various porosities at various degrees of saturation. They observed a significant increase in v_p when the degree of saturation increased from 70 to 100% (between 10% and 70%, v_p was fairly constant). Ramana and Venkatanarayana (1973) studied the effect of the degree of saturation of rocks immersed in water on their weight and on v_p . They showed that the weight and v_p increased with the duration of the immersion. Furthermore, Yu et al. (1991) observed an increase of 23% in the wave velocity on coals immersed in water for 120 h.

These study cases only concerned sedimentary rocks and not clayey ones. Ghorbani et al. (2009) worked on clayey rocks. They showed that, when the degree of saturation decreased, both P and S wave velocities increased. This was quite a new result for clayey rock but some experimental studies (e.g. Santamarina, 2001) have shown that desiccation leads to the consolidation of clayey soils under suction (negative pore water pressure that occurs during desiccation). The samples of clayey soil blocks studied in the present paper behaved in exactly that way.

3.3. σ_c and E

Table 3 presents the results of the uniaxial compressive strength (σ_c) and the initial tangent modulus (E) measurements on the soil

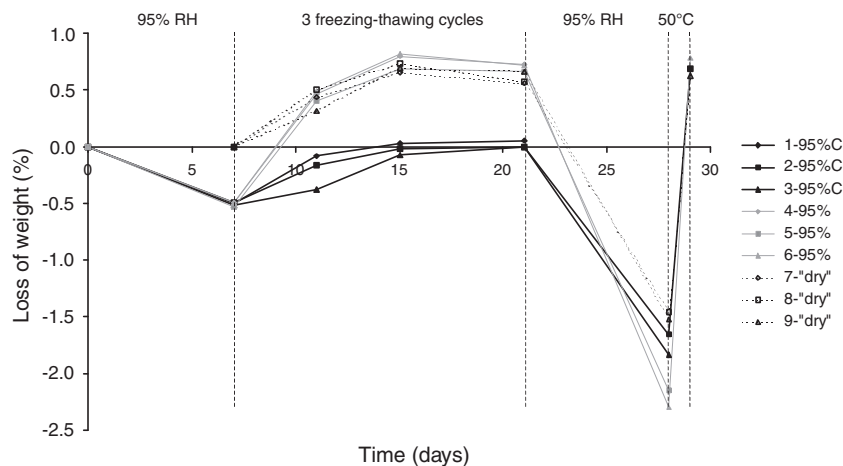


Fig. 1. Loss of weight (%) of the specimens.

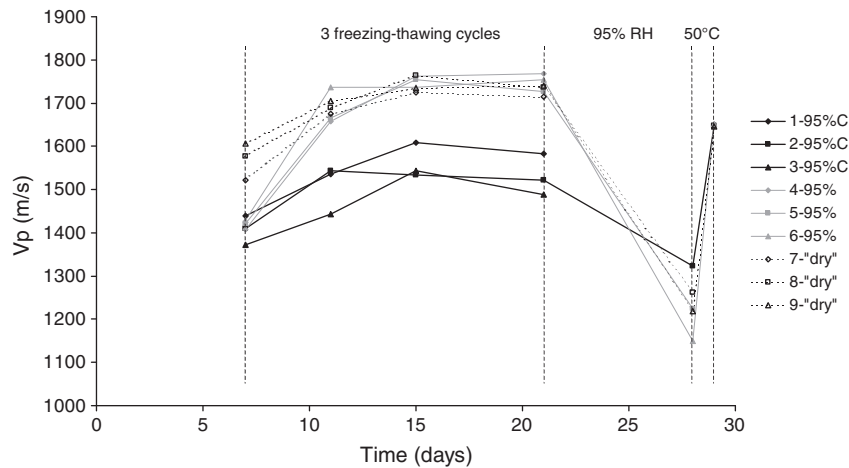


Fig. 2. Compressive wave velocity (V_p) of the specimens.

blocks. The height of the specimens is shown so that the apparent density of the blocks can be calculated, and the water content of the blocks is given (obtained after shrinkage at 105 °C on the pieces of broken blocks). Specimen 3 was broken during V_p measurement. For this reason, no values are shown for σ_c and E for this specimen.

The results show that the freezing–thawing cycles lead to a marked increase in σ_c and E . This increase is accompanied by a decrease in the water content of the specimens subjected to freezing–thawing cycles. These results confirm those presented previously: the freezing–thawing cycles dry the soil blocks and lead to a hardening of the samples. Moreover, the re-humidification of the specimens after the freezing–thawing cycles leads to a strong increase in the water content and, in parallel, a decrease in σ_c and E . It is interesting to note that the mechanical characteristics of the two re-humidified specimens, 5 and 8, are the same. It seems that the re-humidification erases the effect of the previous treatments applied to the soil blocks. Moreover, it is possible to compare the results of specimens 5 and 8 with those of specimens 11 and 12 in order to evaluate the reversibility of the wetting–drying cycle (the first drying being due to the freezing–thawing cycles as shown previously). Once again, the mechanical properties of the specimens re-humidified under 95% RH are the same whatever the previous treatments received.

4. Conclusion

The aim of this paper was to carry out exploratory work on the effects of freezing–thawing cycles on the mechanical properties of clayey soil blocks. As the blocks were not stabilized, the conventional

procedures applied for freezing–thawing cycles were adapted to this case of study (the specimens were humidified by placing them in a moist environment for 1 week (20 °C and 95% RH)).

The results of the study lead to the conclusion that, under the experimental conditions used, freezing–thawing cycles desiccate clayey soil blocks in exactly the same way as thermal desiccation. The loss of weight and the variation of compressive wave velocity in soil blocks show that two opposing phenomena occur during freezing–thawing cycles: on the one hand, the desiccation induced by freezing–thawing cycles leads to the creation of micro cracks in the soil blocks and, on the other hand, water loss leads to a reduction of the pores and thus to a hardening of the soil. Such hardening was also confirmed by the study of the mechanical properties of the blocks (initial tangent modulus and compressive strength).

These first conclusions would merit confirmation on clayey soil blocks composed of other types of clay minerals. The clayey soil blocks studied in this paper were essentially composed of kaolinite, which is not very sensitive to water compared to other clay minerals such as montmorillonite. Moreover, it could be interesting to test more severe conditions of freezing–thawing cycles than those used in this paper. It would also be interesting to bring the water into the specimens in liquid form: one of the surfaces of the specimens could be in contact with a wet compress during a time to be defined. This humidification could be done between series to maintain a quantity of liquid water inside the specimens. In this case, in contrast to the humidification at 95% RH (with the eventual condensation of vapour inside the pores), the water could freeze, which would be very unfavourable for the durability of the clayey soil blocks. Whatever the test chosen for the study of freezing–thawing cycles on clayey soil blocks, it will need to be representative of real risks encountered by these blocks during their service life. Setting up such a test still requires thought and discussion among experts on the topic.

Table 3
Geometrical and mechanical characteristics of soil blocks.

Samples	FT	95%	50 °C	σ_c	E	w	V_p	Weight	Height	Density
				MPa	MPa	%	m/s	g	mm	$\text{g}\cdot\text{cm}^{-3}$
1–95°C	X			3.60	665	1.66	1583	546.9	109.5	2.00
2–95°C	X	X	X	5.03	585	1.07	1648	546.4	109.2	2.00
3–95°C	X	X		–	–	–	–	561.0	109.8	2.04
4–95%	X			2.78	337	1.46	1769	537.4	109.5	1.96
5–95%	X	X		2.50	244	3.50	1224	568.8	109.6	2.08
6–95%	X	X	X	5.20	1150	0.99	1652	543.9	109.4	1.99
7–“dry”	X			4.23	684	1.42	1714	547.1	109.5	2.00
8–“dry”	X	X		2.79	300	2.97	1263	556.3	109.7	2.03
9–“dry”	X	X	X	4.93	852	1.04	1646	539.4	109.6	1.97
10–“dry”				3.36	355	1.42	1448	547.2	109.8	1.99
11–95%				2.20	371	3.12	1202	563.6	109.7	2.06
12–95%				2.50	303	3.30	1180	577.0	109.6	2.11

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