

Chapter 2 Review on the characterization of earth used in earth construction materials

Jean-Emmanuel Aubert¹, Paulina Faria², Pascal Maillard³, Kouka Amed Jérémy Ouedraogo⁴, Claudiane Ouellet-Plamondon⁵, Elodie Prud'homme⁶.

¹ Université de Toulouse, UPS, INSA, LMDC (Laboratoire Matériaux et Durabilité des Constructions), Toulouse, France, jean-emmanuel.aubert@univ-tlse3.fr

² CERIS and Universidade NOVA de Lisboa, Caparica, Portugal

³ CTMNC, Research and Development Department of Ceramic, Ester Technopole, Limoges, France

⁴ Université de Toulouse, UPS, INSA, LMDC (Laboratoire Matériaux et Durabilité des Constructions), Toulouse, France

⁵ École de technologie supérieure, Université du Québec, Montreal, Quebec, Canada

⁶ Université de Lyon, MATEIS, Matériaux: Ingénierie et Science

Abstract

The objective of this chapter is to present the physical, geotechnical, chemical and mineralogical characterization techniques used to characterize the raw material (earth and mineral addition, such as sand and gravel) contained in the earth materials manufactured with different techniques: earth bricks, rammed earth or cob. This chapter will be divided into 6 sections. The first will present the method used to find the references considered in this state of the art and we will carry out a general qualitative analysis of these references. The other sections will deal respectively with granular, geotechnical, chemical and mineralogical characteristics and, finally, the last part will be dedicated to field tests.

Keywords: Particle size distribution, physical and geotechnical characterization, chemical and mineralogical properties, field tests.

2.1. Introduction

Earth has always been used by man to build his habitat. Until the middle of the 20th century, it was the most used building material with stone and wood. After the Second World War, concrete came to replace these materials, especially in western countries, thanks to its properties: speed of curing coupled with ease of mechanization of implementation, low cost, high mechanical performances and good durability. Concrete made from aggregates (mostly from natural origin), cement and admixture is being used at a fast rate worldwide. If cement did not pose any environmental problems (in particular because of its carbon footprint due to the significant CO₂ emission during its production), it is unlikely that researchers will once again be interested in natural materials, such as earth, as an alternative to cementitious composites. But the dramatic ecological situation in which the world is at the beginning of the third millennium forces men to reconsider how they consume, and in particular how they build. Furthermore, cementitious traditional components, especially sand scarcity, more and more highlights the economic effectiveness of using alternative available construction materials.

Thus, materials that have been neglected for several decades, such as earth or biobased materials as the case of vegetable fibres, are finding renewed interest. This is due to their low environmental impact (abundance, renewability in the case of biobased materials, recyclability and low embodied energy) but also to their own properties, especially from the point of view of the comfort of inhabitants (see the chapter on the hygrothermal properties of earth construction materials). Today, it is estimated that over two billion people are still living in earthen buildings and that 10% of the architectural world heritage properties include earth structures (Van Damme and Houben, 2018). Earth construction is part of the solution to ensure men live within the resources and climate planetary boundaries.

Historically, the formulation and the manufacture of earth construction materials have been done in an empirical way based on local constructive cultures, often orally passed down from generation to generation by the builders who were also mostly farmers who exploited earth for agriculture. Those builders learned to adapt to the constraints of local materials and, in particular, to the properties of the local soils. In fact, unlike today, it was not possible to transport materials over long distances and it was not possible to improve an inadequate earth by adding chemical stabilizers. This has led in particular to a regionalization of techniques which France can be used as an example as illustrated in Figure 2-1.

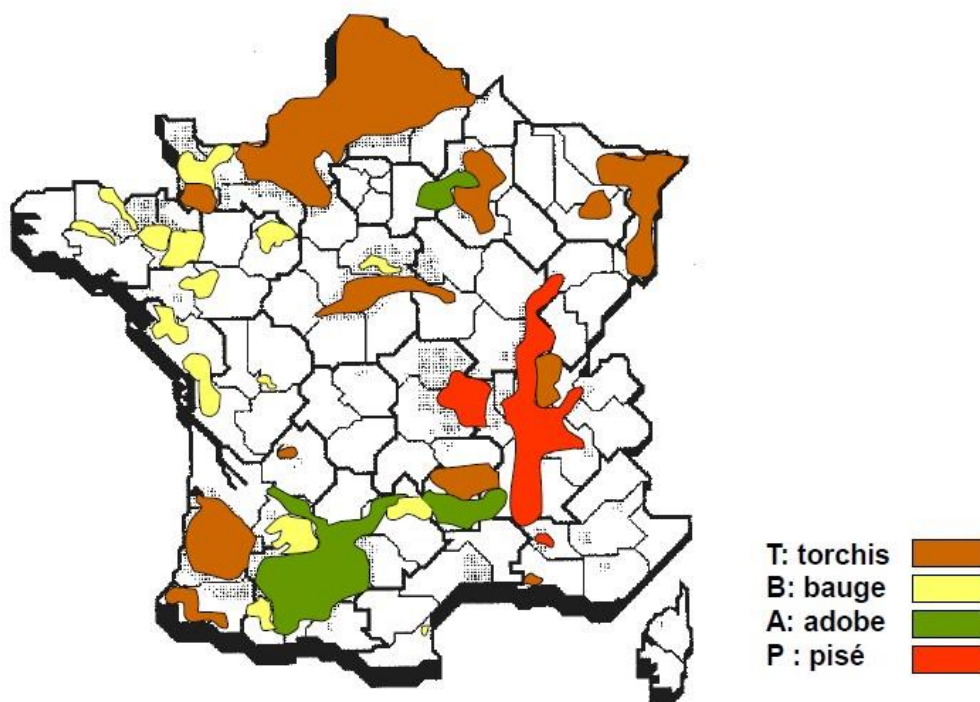


Figure 2-1. Geographical distribution of earthen heritage in France («pisé» = «rammed earth», «bauge» = «cob», «torchis» = «wattle and daub») based on Guillaud (2018)

Indeed, before it became cultural, men adapted to the properties of the soil from his region, which oriented his choices on the technique used. For example, earth from the Rhone Valley contains significant amounts of coarse grains and if the builder of that period had wanted to manufacture adobe with this earth, he would have needed to sift (or to grind) his material which was not possible at that time. Thus, the best adapted technique to this granularity of earth was rammed earth, which explains why almost all the earth buildings in the Rhône Valley were

built with rammed earth technology. There is a similar analysis for the cob in Normandy but this time, one of the reasons for the choice of this technique compared to others is not necessarily related to the characteristics of the earth but more to the meteorological conditions. Even if the earth from Normandy has a granularity usable to manufacture adobe, the state of humidity in place and the difficulties of drying have oriented the builders towards "adobe put directly in place without drying", namely the cob technology. In cases where the earth used was too clayey, the high-water content of cob caused significant cracking of the materials during drying. The masons of the past had found a solution to this problem by adding vegetable fibres to the earthen mixture, which allowed both to limit cracking during drying but also to better structure the fresh pieces of cob. In the south-west, hot and dry summers coupled with Garonne Valley soil with excellent properties have led to the development of adobe in this region. Thus, the vernacular heritage shows that the builders, by experience, have learned to adapt to local materials. The same goes for the formulation of the materials: they used no weighing, no particle size distribution and even less chemistry or mineralogy! The formulas used were the result of the experience gained on the construction site by the builders.

The world of building materials and products has changed and today we talk about standards, control, performance and modelling. In addition, we are able to transport materials over greater distances (even if from an environmental point of view this is not desirable) or to transform soils not adapted to earth construction by using chemical additions especially hydraulic binders. In this logic, researchers are trying to rationalize the formulation of earth building materials or to understand and predict the behaviour of these materials from a thorough characterization of raw materials. The objective of this chapter is to present the physical, geotechnical, chemical and mineralogical characterization techniques used to characterize the raw material (earth and mineral addition, such as sand and gravel) contained in the earth materials manufactured with different techniques: earth bricks, rammed earth or cob. This chapter will be divided into 6 sections. The first will present the method used to find the references considered in this state of the art and we will carry out a general qualitative analysis of these references. The other sections will deal respectively with granular, geotechnical, chemical and mineralogical characteristics and, finally, the last part will be dedicated to field tests.

2.2. Global analysis of references used for this review - methodology

The number of scientific studies on earth construction materials has increased rapidly in the last ten years, as shown in Figure 2-2. This curve was obtained in 2017 after a search using the key following keywords: adobe, cob, wattle and daub, compressed earth bricks, rammed earth, earthen material and earth bricks. Then a sorting was done, in particular to eliminate the duplicates. We find a total of 422 references whose distribution by years is shown in Figure 2-2.

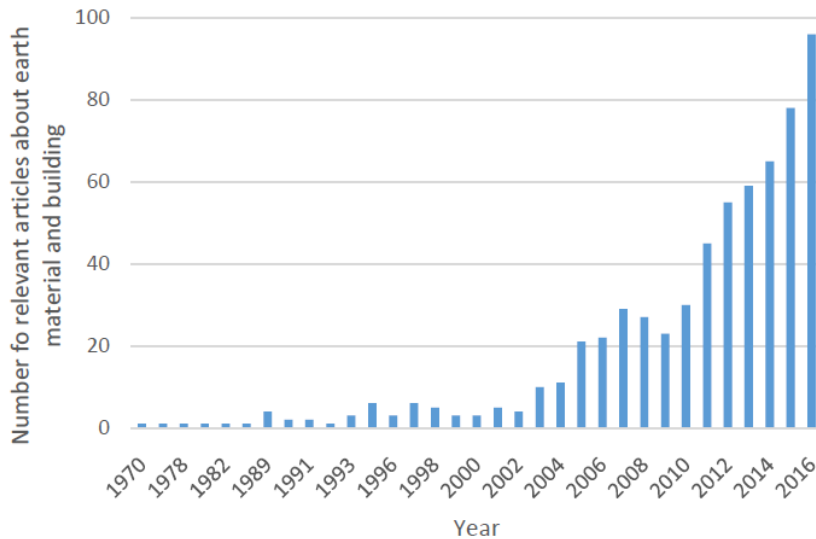


Figure 2-2. Number of relevant articles about earth materials and buildings found in 2017

To facilitate the research of our working group, we did not want to analyse the data in a systematic way or to make an exhaustive bibliography. The review should be sufficiently representative of what exists in the literature. In the vast majority of cases, we have limited our research to scientific articles published in reference journals. Many articles about earth construction materials are published in Elsevier's journals, so we did our article research using the Scencedirect search engine. Another important source of articles was Rilem's Materials and Structures journal; so we have completed the data found in Elsevier by those found in this journal. To limit the number of articles, we used in Scencedirect the following combinations:

- 1 - Earth bricks (in abstract, title, keywords): 146 results,
- 2 - Adobe alone does not work (too many references); so adobe (in abstract, title, keywords) and earth (in all fields): 143
- 3 - Compressed earth blocks (in abstract, title, keywords): 58
- 4 - Rammed earth (in abstract, title, keywords): 176
- 5 - Cob alone does not work (too many references); so cob (in abstract, title, keywords) and earth (in all fields): 138
- 6 - Wattle (in abstract, title, keywords) and earth (in all fields): 24
- 7 - Daub (in abstract, title, keywords) and earth (in all fields): 12

Search by keywords gives many results and sometimes some are not relevant. It was therefore necessary to perform an important manual sorting of these results by removing irrelevant references. Then, we kept only the references in which a minimum of raw materials characterization was done. To facilitate the reading and the comparison of the results, we separated these articles by technique (Extruded Earth Bricks (EEB), Compressed Earth Bricks (CEB), adobe, cob and rammed earth) and according to whether they were chemically stabilized or not. The results are shown in Table 2-1.

As shown in Table 2-1, the reference number for some materials is very low but this corresponds to a reality. Moreover, in the case of the cob, we had to add references that are not papers from international journals to supplement the data that were too scarce. The most studied materials in the literature are earth bricks (adobe and CEB) and rammed earth. It is important

to remember that this research is not exhaustive and that the number of articles on earth construction materials in which raw materials data are found is more important. But, with the partial research that we have done, we reach a relatively high reference number (71 articles) which therefore begins to be representative. In addition, some authors have written several articles using the same earth: in these cases, either we keep only the article in which the characterization is presented, or we keep the most recent reference if the data presented are the same.

Table 2-1 Number of articles dealing with earth construction materials where raw materials are characterised

Technology	Unstabilized	Stabilized	Total
Extruded earth bricks	5	1	6
Compressed earth bricks	4	12	16
Adobe	18	7	25
Cob	5	0	4
Rammed earth	8	11	19
Total	40	31	71

In recent years, it has been usual to add chemical stabilizers to the earth. The reasons given are the improvement of the mechanical performance of the construction products (see chapter 3) as well as the improvement of its resistance to liquid water (see chapter 5). It is interesting to study the differences in the use or not of stabilization depending on the techniques used. For example, we note that the CEB are almost systematically stabilized. This can be explained by the fact that CEB are modern materials that emerged recently (mainly after the 1980s) in the history of earth construction. The objective is to accelerate the drying of the bricks by producing them with less water than in the traditional adobes: the consistency thus obtained no longer allows the material to be applied by moulding and must then be pressed (or compacted) in a mould. Since mould release is immediate, the CEB manufacturers quickly decided to add a hydraulic binder (lime with hydraulic properties classified based on EN 459-1 (2015) or cement) in their material to improve its performances in a very short term to facilitate handling and storage of these bricks. Conversely, the earth of all the articles on cob is unstabilised. A large majority of articles are about unstabilised adobes probably because the materials from the vernacular heritage often studied in these articles are rarely stabilized. However, when the local earth had low clay content, air lime could be added for vernacular adobe production (Parracha et al., 2019a). EEB are singular because, like CEB, they are modern materials, yet few studies focus on stabilization (only 1 out of 6). This can partly be explained by the method of manufacture of these bricks (extrusion after evacuation of the air under vacuum) which gives the bricks at the extruder outlet exceptional holding that allows them to be handled and stored easily while waiting for them drying. In fact, EEB are often similar to the ones that will be fired, but do not embody the firing energy of the latter. Finally, there is no strong trend for rammed earth where there are generally as many studies on stabilized or unstabilised materials.

Data was collected from these articles and separated into 4 types of material characterization:

- particle size distribution ;
- physical and geotechnical characterization ;
- chemical characterization ;

- mineralogical characterization.

The accuracy of earth characterization in the papers dealing with earth building materials is highly variable. In almost every paper, the particle size distribution of the earth is presented: this characteristic can be considered as the basic characteristic of earth. Nevertheless, the particle size distribution is not always complete: sometimes, the fine fraction is missing, and in some cases it is not performed by wet method and, therefore, the fine fraction can be considered agglutinated in clods. However, the measurement of other characteristics is not systematic. To quantify this, we used three levels as follows:

- the number "3" corresponds to the most basic characterization, that means that only the particle size distribution of the earth is given;
- the number "2" is used when, in addition to particle size distribution, at least one other characteristic is given (physical and geotechnical characterization or chemical or mineralogical characterization);
- finally, the number "1" qualifies the articles in which we find: particle size distribution + physical and geotechnical characterization + chemical and / or mineralogical composition: this corresponds to the most thorough characterization.

Table 2-2 presents the distribution of these levels of deepening of the characterization according to the type of materials.

Table 2-2. Deepening levels of the characterization of raw earth

Technology	"1" Deepest	"2" Average	"3" The most basic	Total
Extruded earth bricks	3	2	1	6
Compressed earth bricks	4	11	1	16
Adobe	8	12	5	25
Cob	1	1	3	5
Rammed earth	3	10	6	19
Total	19	36	16	71

Generally, the majority of the items we analysed are in the middle level "2". It is also interesting to note that it depends a lot on the type of material studied. For example, in the case of the cob, the majority of references are level "3" whereas in the case of the EEB, it is the opposite: the studies present thorough characterization of the earth. However, for both technologies, the total of references is very low. This could be explained by the fact that the cob is a more traditional material whereas the EEB are more modern and more technological materials. For these latter, a better knowledge of the characteristics of the components is needed to efficiently optimize them.

Table 2-3 presents these results in a different and more precise way. In this table, the proportions of the different characteristics found in the articles are presented.

Table 2-3. Proportions of the main characteristics of raw earth found in the papers dealing with earth construction materials

Technology	Particle size distribution	Physical and geotechnical characterization	Chemical characterization	Mineralogical characterization
Extruded earth bricks	6 (100%)	2 (33%)	4 (67%)	4 (67%)
Compressed earth bricks	16 (100%)	14 (88%)	5 (31%)	4 (25%)
Adobe	24 (96%)	16 (64%)	7 (28%)	9 (36%)
Cob	5 (100%)	2 (50%)	0 (0%)	2 (50%)
Rammed earth	19 (100%)	8 (42%)	3 (16%)	7 (37%)
Average value	70 (99%)	42 (71%)	19 (27%)	26 (37%)

As previously noted, when the level was established, all selected items (except one) present the particle size distribution of the earth. For the other characteristics, the proportions are much more variable. Physical and geotechnical characteristics come in second place with an average of 71% appearance. As a general rule, most of the paper dealing with earth bricks contains the Atterberg limits and the particle size distribution of earth. For rammed earth articles, the Atterberg limits are often replaced by the Proctor tests, which is consistent because of the differences in the use of these materials, namely the use of humid compacted earth for rammed earth instead of plastic moulded earth for adobe. The mineralogical characterization of the earth appears in about a third of the papers, which is still relatively high. However, we will analyse these data in section 2.6.2 of this chapter and we will see that most of the data presented are qualitative and that many questions raise about the procedures used to obtain some results and about the accuracy of these results. Finally, the least studied characteristics are the chemical characteristics. No data are available for the cob and, for rammed earth, there is very little data: there are measurements of earth pH in two articles and measurements of organic matter content in two others. Considering the 19 articles dealing with rammed earth, no basic chemical analysis of earth is given what may seem surprising. One can try to explain this by a different cultural approach of the researchers working on rammed earth and those working on earth bricks and namely on EEB. Indeed, for rammed earth, one often uses the earth which is directly available on the building site and which showed by experiment that is suitable. The researchers working on these materials are essentially specialists in mechanics or more recently in hygrothermal behaviour of materials, and are probably less sensitive to the chemical composition of earth. In addition, the studies of the rammed earth are often made on a macroscopic scale because of the specific implementation of these materials (a wall is directly built and not elements of a wall as in the case of bricks), and the heterogeneous granular material, with presence of coarse aggregates. The presence of those coarse aggregates turns low the clay content of the earth. Therefore, rammed earth is not as much influenced by the characteristics of the clay as other more clayey materials used for brick production. In the field of research on bricks, the analyses are often done on a finer scale and the researchers are often specialists in physico-chemistry of materials.

After this first chapter of global analysis of the elements of the bibliography that we collected for this state of the art, we will present different sections corresponding to the different families

of characteristics presented previously. Each of these sections will have the same structure and will be divided into two parts. In the first part, we will present the test procedures found in the literature based on some international standards, when they exist. The aim here is not to make a comprehensive review of existing international standards but to provide examples of the most used standards. Thus, and also in relation to the origin of the co-authors of this chapter, we will use the following norms: American, Quebec, English and European. The second part of each section will be devoted to the analysis of the results extracted from the bibliographical references in order to see if trends exist according to the type of materials or the type of technique used. Something that is very important for earth characterization is the representativeness of the samples. A representative earth sample depends on the tests to perform and, therefore, the type of earth building technology that is intended. However, the earthen sample size is often not presented in scientific articles. Furthermore, in case of earth architectural heritage characterization, the sampling is frequently limited due to restrictions of the heritage property (Parracha et al. 2019b). The first of the characteristic studied that is most present and certainly the most important for earth construction materials is the particle size distribution that is the focus of the next section.

In this review we have used the term earth for the building material. However, namely for geotechnical characterization, frequently the term soil is used not only for the non-extracted earth. Therefore in the following sections both terms are used to follow the original authors' terminology.

2.3. Particle size distribution

As commented before, the particle size distribution is one of the most important physical characteristics of soil. Classification of soils is mainly based on the particle size distribution. Many geotechnical and geohydrological properties of soil are related to the particle size distribution. The particle size distribution provides a description of soil based on a subdivision in discrete classes of particle sizes.

2.3.1 Procedures and standards

Standards:

Several standards for soil classification and particle size distribution exist. It is possible to separate them in two types: the wet sieving particle size for the coarser particles ($> 80 \mu\text{m}$) and the sedimentometry for the fine fraction (1 to $80 \mu\text{m}$). It is important to specify that the laser granulometry is not suitable for the measurements of the granularity on clay soils, mainly because of the difficulties of dispersion of the particles. To be applied the previous dissolution of the soil in water and a wet method should be used.

North American standards ASTM C136 (2014) deal with wet sieving and ASTM D422 (2011) with sedimentometry. The British BS 1377 Part 2.9 (1990) and Quebec BNQ-2501-025 (2013) standards include procedures for wet sieving and sedimentometry. It is the same for the European standard EN ISO 17892-4 (2018). Whether they are North American, English or European, they are very close or even similar in particular in characterization methods (sieving and sedimentation). The principle of these test procedures is described later in this section.

Procedures:

Coarse soils are usually tested by sieving, but fine and mixed soils are usually tested by a combination of sieving and sedimentation, depending on the composition of the soil. The sieving method described is applicable to all non-cemented soils with particle sizes less than 125 mm. Two sedimentation methods are described: the hydrometer method and the pipette method.

The test method or combination of methods should be specified prior to testing or be selected on the following basis. If a sample has less than about 10% of particles smaller than 0.063 mm, sedimentation test is not normally required. If all particles of the sample are smaller than 2 mm and the sample has less than about 10% of particles larger than 0.063 mm, a full-sieve test is not normally required. For all other samples, a combination of a sieve test and a sedimentation should be performed in order to determine the full-particle size distribution.

Sieving method: The test consists of separating the agglomerated grains from a known mass of soil by fractionating it under water with a series of sieves and weighing the cumulative and dried rejection on each sieve (dried usually at 105 °C). The mass of the cumulative rejection for each sieve is related to the total dry mass of the soil sample submitted for analysis. Either a moist or a dry sample may be tested. The sieve test consists in the determination of the masses of material retained on the various sieves with decreasing diameter sizes. The number of sieves used and their aperture sizes shall be sufficient to ensure that any discontinuities in the grading curve are detected. In the standard EN ISO 17892-4 (2018), it is recommended (but not imposed) to use the sieves of 63 mm, 20 mm, 6.3 mm, 2.0 mm, 0.63 mm, 0.20 mm, 0.0063 mm because these values represent the size limits for coarse materials as defined in EN ISO 14688-1 (2018).

Dry sieving is not appropriate particularly for clayey earths/soils because grains that result from the agglomeration of particles are sieved without separation.

Sedimentation: Based on the Stokes' law, the method is based on the measurement of the sedimentation time of solid particles in suspension in a solution of water mixed with sodium hexametaphosphate as a deflocculating agent. The sedimentation analysis is an analysis completing the sieving analysis for particles usually with a diameter of less than 80 µm. The test is based on the fact that in a liquid in which a deflocculating agent has been added (sodium hexametaphosphate), the decantation rate of the fine particles depends on their size. The principle follows Stokes' law linking the diameter of the grains and their sedimentation rate. By convention, this law is applied to the elements of a soil to determine the equivalent diameters of the particles. The test can be carried out using two different methods:

- Hydrometer method: A part of the soil is dried then mixed with water containing the dispersing agent, and then the hydrometer is introduced into the graduated cylinder. The density of the mixture is measured with the hydrometer at various time intervals (e.g.: 30 s, 1 min, 2 min, 4 min, 8 min, 30 min, 1 h, 2 h and 24 h). From the density measured at a given time, the size of the suspended particles can be determined. The hydrometer shall be torpedo-shaped, made of glass, as free as possible from visible defects and preferably manufactured to a national standard. The hydrometer stem and bulb shall be circular in cross section and symmetrical around the main axis, without abrupt change in cross section.
- Pipette method: Based on the same principle and theory, the pipette method consists of taking a fraction of the mixture (soil dispersed in water containing a dispersant) at different times and depths, and then drying and weighing the residue. It is also possible to initially define the particle sizes in order to know their quantity, and then calculate the corresponding sampling times. The pipette shall have a nominal volume of 2 % of the volume of the soil suspension and shall be mounted in a pipette configuration.

This sedimentation measurement method has also been automated and modernized with the use of a sedigraph. An X-ray beam measures the concentration of suspended particles at a sedimentation height that decreases with time. The particle diameters are obtained instantly corresponding to the elapsed time and sedimentation height.

A source of error in these different procedures could be linked to the incomplete dispersion of soil clays. If clay particles are not separated correctly, they form aggregates with a larger size. It results in low values for clay and high values for silt and sand. The rate of sedimentation is also affected by temperature, the density of the dispersing solution and by a too abrupt introduction of the hygrometer or of the pipette.

Soil classification:

As defined in the standard EN ISO 14688-1 (2018), Table 2-4 shows the terms to be used for each size fraction, together with the corresponding range of particle sizes. Clay can be defined from a granular point of view (particle size) and also from a geological point of view (mineral composition). But, in most publications, clay is defined as a particle with a diameter of less than 2 μm . According to the standards and their origin, the limits between the particle size and their names can vary, especially the limit silt - sand. In the standards EN ISO 14688-1 (2018), USDA (1987) and ASTM-D2487 (2017), this limit is fixed respectively to 0.063 mm, 0.05 mm and 0.075 mm.

Table 2-4. Particle size fractions according to the EN ISO 14688-1 (2018)

Soil group	Particle size fractions	Range of particle sizes (mm)
Very coarse soil	Large boulder	>630
	Boulder	>200 to \leq 630
	Cobble	>63 to \leq 200
Coarse soil	Gravel	>2.0 to \leq 63
	Coarse gravel	>20 to \leq 63
	Medium gravel	>6.3 to \leq 20
	Fine gravel	>2.0 to \leq 6.3
	Sand	>0.063 to \leq 2.0
	Coarse sand	>0.63 to \leq 2.0
	Medium sand	>0.20 to \leq 0.63
	Fine sand	>0.063 to \leq 0.20
Fine soil	Silt	>0.002 to \leq 0.063
	Coarse silt	>0.02 to \leq 0.063
	Medium silt	>0.0063 to \leq 0.02
	Fine silt	>0.002 to \leq 0.0063
	Clay	\leq 0.002

2.3.2 Study of data from literature

The particle size distributions of the soils studied in the literature are presented in Tables 2-18 to 2-22 of Appendix 1. Some cells were deliberately grayed out: this corresponds to the data that we were unable to use in our study because the granular classes used do not correspond to the conventional classes.

2.3.2.1. Earth bricks

Figure 2-3 and Figure 2-4 show the particle size distributions of the earth bricks studied in different papers, all techniques being considered (moulding, compression and extrusion) (see details in Tables 2-18 to 2-20 given in Appendix). Although the ternary diagram is not the most used in the literature, it allows here to represent the different sizes. No clear trend appears but earth used for EEB seems to be thinner than those used for adobe and CEB, and the silt and clay quantities are generally higher, which makes sense because of the particular manufacturing process of EEB. For adobes and CEB, there is no significant difference observed. However, one technique moulds a plastic earth mixture while the latter compresses a humid mixture (with much lower water content), and the composition (mineralogical but also in terms of particle size distribution) could be different. Moreover, the ternary diagram shows specific earth as for example earth containing little or no clay. That seems to be strange because clay, like cement for concrete, is the binder of earth building materials. As mentioned above, certain values may be due to handling errors (insufficient dispersion, etc.). Moreover, these “special” values also highlight the role of a binder (lime or cement) added to the soil to ensure better cohesion. Although stabilization can improve the mechanical performance of bricks, it is important that the earth alone already has good cohesion as indicated in French standard XP P13-901 (2001).

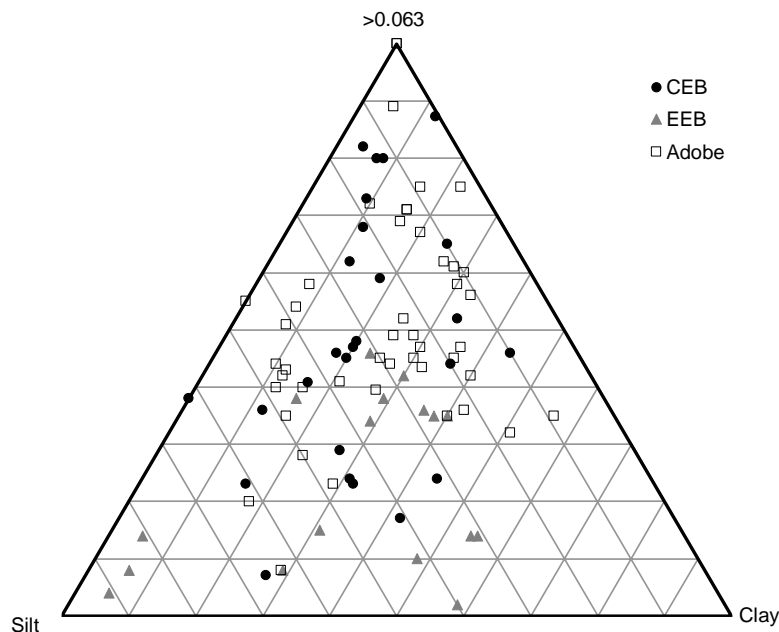


Figure 2-3. Particle size distribution or raw earth used for earth bricks in a ternary diagram

On Figure 2-4, the limits recommended by XP 13-901 for CEB are also presented. Many soils studied in the literature do not respect these limits. Moreover, Houben and Guillau (2006) gave other limits for adobes (clay: 10-30%, silt: 15-33%, sand + gravel: 37-75%) while specifying that the recommended area is only approximate. Other specific cases of study have been observed particularly in the French heritage: an example in south-western France with adobes

of the nineteenth century which contain pebbles of several centimetres (Figure 2-5) questions the relevance of these limits.

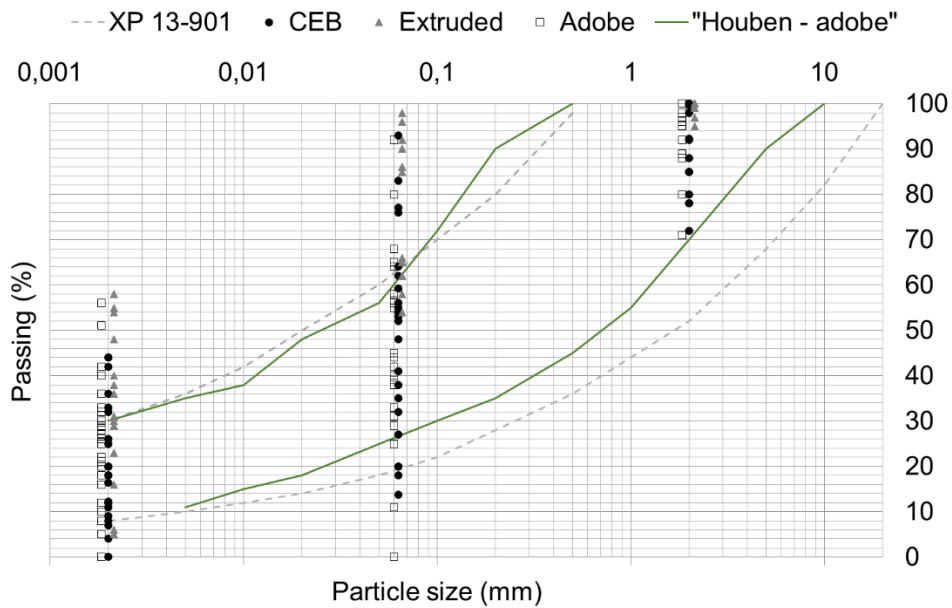


Figure 2-4. Particle size distribution of raw earth used for earth bricks



Figure 2-5. Adobes from southern France (Montricoux) with coarse aggregates (XIXth century) ((c) Pays Midi-Quercy ; Conseil départemental de Tarn-et-Garonne ; Inventaire général Région Occitanie)

2.3.2.2. Cob

A significant review was done by Hamard et al. (2016). The authors indicate that the cob is less studied, unlike other techniques, the number of results on the soils used for this technique is therefore rather low and it is difficult to generalize the few data found in the literature. In contrast to other techniques, it is not necessary to differentiate between stabilized and non-stabilized materials, since all references only deal on earthen materials that are not chemically stabilized. Moreover, the straw content is only very seldom specified: only one reference (Coventry, 2004) provides the straw content of the cob (1% wt.). Fibres that are generally largely present in this type of material make particle size analysis difficult. Indeed, for these types of materials including plant fibres, it is necessary to separate the mineral phase from the

plant phase to characterize the particle size distribution the more precisely as possible. This separation is often difficult.

Table 2-21 of Appendix A presents the particle size distribution of soils found in the literature. Some data are special as that of Quagliarini et al. (2010) because the soil used for the renovation of the studied buildings is a soil rich in clay intended for the manufacture of clay bricks. Other older data indicate only two lots of grains (clay + silt and sand + gravel). In Coventry (2004), two of the five soils studied have very few fine particles (clay and silt contents of between 5 and 7%) whereas for one earth, the content of fine particles is very high (clay + silt equal to 94%). The researcher specifies that the five soils tested in compression (cylindrical sample) have good mechanical properties, which shows that it is possible to produce cob having good mechanical performance with very different particle size distributions of soil. Nevertheless, Saxton (1995) recommends an ideal particle size distribution (clay + silt = 35%, sand = 35% and gravel = 30%) with a wider acceptable zone (clay + silt = 25-45%, sand = 25-45% and gravel = 20-40%). Same limits (1/3 clay + silt, 1/3 sand, 1/3 gravel) are indicated by Harries et al. (1995). It can be seen that none of the nine soils used for cobs in the literature and presented in Table 2-21 meet these recommendations.

Finally, Hamard et al. (2018) have developed a new methodology to identify and quantify material resources at a large scale for earth construction especially applied to the cob in Brittany. This methodology is based on the cross-referencing of spatialized pedological and heritage data. The methodology applied at the regional scale in France (for a given area of 27,200 km² in Brittany) enabled to specify five new texture classes (balance between clay, silt, sand and gravel content) of suitability for cob soils. For a further discussion on the identification and quantification of soils for construction, the researchers propose to apply the same methodology to other regions with different earth construction techniques.

2.3.2.3. Rammed earth

Figure 2-6 presents the particle size distributions of 19 bibliographic references dealing with rammed earth. Compared to the bricks described in the previous paragraph, a trend is clearly apparent: the earth generally contains more than 60% of grains with a diameter greater than 0.063 mm and less than 20% of clay ($\varnothing < 2 \mu\text{m}$). These values follow the recommendations of the Walker and Australian standard HP195 (2001) which indicates the following limits: clay up to 20%, silt between 10 and 30%, sand and gravel between 45 and 75%. Some points highlight clay-rich soils, up to 40% of clay, that do not respect the recommended limits (Ciancio et al., 2013; Serrano et al., 2013).

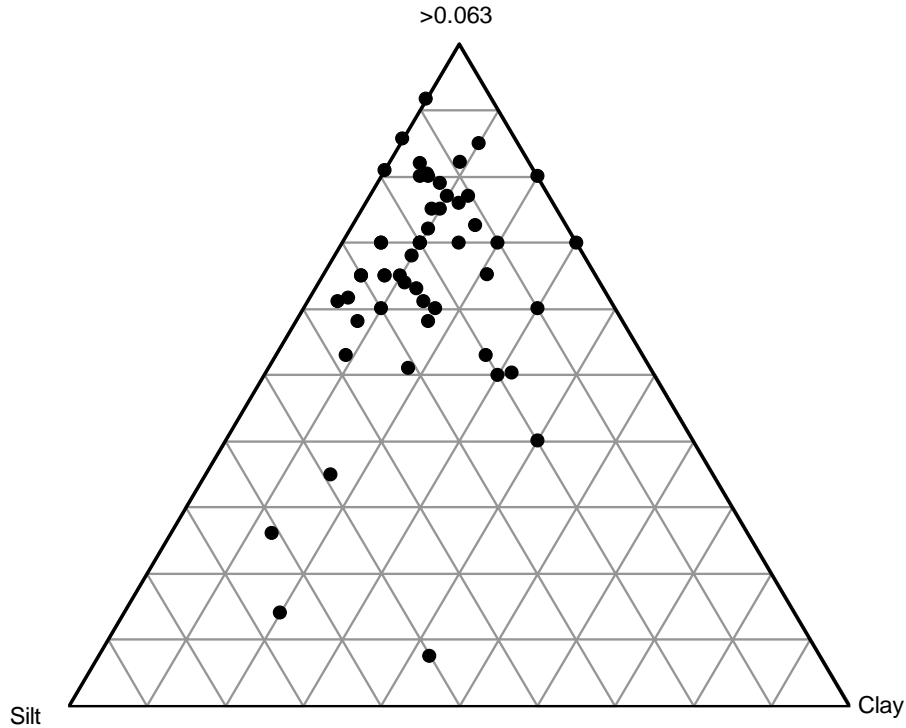


Figure 2-6. Particle size distribution or raw earth used for rammed earth in a ternary diagram

Nevertheless, no soil is likely to be ideal; therefore researchers usually published in the past upper and lower limits for each of the main soil elements (Table 2-5) (Maniatidis and Walker, 2003; Gomes et al., 2013). As it was the case for bricks, the particle size distribution of earth materials often comes out of these limits, which was found by Gomes et al. (2014) on the six earth materials from existent constructions they have studied.

Table 2-5. Recommendations concerning the particle size distribution of soils for rammed earth construction (adapted from Gomes et al., 2014)

Reference	Clay (%)	Silt (%)	Sand + gravel (%)
(Keable, 1996)	5-15	15-30	50-70
(Norton, 1997)	10-25	15-30	45-75
(SAZS 724, 2001)	5-15	15-30	50-70
(Keefe, 2005)	7-15	10-18	75
(Walker <i>et al.</i> , 2005)	5-20	10-30	45-80
(Houben and Guillaud, 2006)	0-20	10-30	45-75

2.4. Physical and geotechnical characterization

2.4.1 Procedures and standards

Atterberg limits

The United Kingdom (UK) and North American standards on how to measure the Atterberg limits are ASTM D4318 (2017), BNQ 2501-090 (2005), BS 1377-2 (1990) and BS 5930 (2015). In Europe, the test method is defined by EN ISO 17892-12 (2018).

The liquid limit (LL) and the plastic limit (PL) classify fine-grained earth and the fine fraction of mixed soil. They are commonly required for geotechnical engineering tests, both in industry and in academic research (O'Kelly et al., 2018). In our literature review, it is the most frequent geotechnical test performed to characterize the earth for construction. According to the standard BS 1377-2 (1990), LL is the empirical moisture content at which a soil passes from a liquid state to a plastic state. The LL is measured either with the cone penetrometer or the Casagrande method. The definitions of PL slightly differ depending of the standard considered. In BS 1377-2 (1990), the PL is the empirical moisture content at which a soil is too dry to be plastic, which is the transition from a ductile to a brittle behaviour (O'Kelly et al., 2018). In ASTM D4318 (2017), the PL is the percentage of water content of a soil at the boundary between the plastic and the semi-solid state. The PL is measured by rolling a thread of soil. The plasticity index (PI) is calculated as the numerical difference between the LL and the PL. The graphical representation of the PI allows to classify cohesive soils (BS 1377-2, 1990) and to determine boundaries between consistency states of plastic soil, which means the relative ease in which soil can be deformed (ASTM D4318, 2017). These tests are not much precise and the standard BS 1377-2 (1990) specifies that the results remain variable with the judgment of the operator. These tests originate from the work of Atterberg, which was then standardized. The PL and LL are often collectively referred as the Atterberg limits. Actually Atterberg defined 7 limits (Vardanega and Haigh, 2014). Many countries have their own version of the standard, which means that the variability according to the testing method has been a subject of discussion. As a start point, the BS 5930 (2015) and ASTM D4318 (2017) specify to measure the plasticity on the fraction finer than 0.425 mm, while finer than 0.400 mm in the standards from Quebec (BNQ 2501-090,2019) and in Europe (EN ISO 17892-12, 2018) or Internationally (ISO 17892-12, 2018). Increasing the sand content decreases plasticity, while the fine organic content increases plasticity.

To determine the LL limit, the fall cone penetrometer is preferred in the UK (BS 5930, 2015) and by the International (ISO17892-12, 2018) and European (EN ISO 17892-12, 2018) standards because it is a static test (O'Kelly et al., 2018). The Casagrande test can introduce a dynamic effect and is susceptible to variability between operators (BS 1377-2, 1990). The cone penetrometer gives results that are more reproducible than the Casagrande method (Sherwood and Ryley, 1970). The fall-cone test assesses the soil shear strength by relating the soil's undrained shear strength to the fall-cone weight divided by the square of the penetration depth (O'Kelly et al., 2018). The LL is measured with the Casagrande percussion cup in ASTM and Canadian standards. A portion of the earth specimen is spread in a brass cup, divided in two by a grooving tool, and subsequently allowed to flow together from the shocks caused by dropping the cup in a standard mechanical device (ASTM D4318, 2017). According to the number of drops, the test follows a one-point method or a multipoint method. The number of drops to decide on the method may vary according to standards. The multipoint method is generally more precise. The water content is determined on the soil in the cup at the end of the test.

The plastic limit is measured internationally by pressing and manually rolling a thread of plastic soil on a glass plate until the water content is reduced to the point the thread crumbles and can

no longer be pressed again and rerolled (ASTM D4318, 2017; O'Kelly et al., 2018). The soil is rolled to a thread diameter of 3.0 mm in the UK and Quebec standard, while it is 3.2 mm in the ASTM standard. The soil water content is determined at the breaking point. The repeatability of the thread rolling tests varies with the number of operators. For example, the standard deviation was less than 1% when one operator was considered and up to 3% when considering 41 operators from different laboratories (Sherwood, 1970).

Test methods for laboratory compaction characteristics of soil using standard effort (Proctor tests)

The standard test methods for laboratory compaction characteristics of soil using standard effort is commonly known as the Proctor test in reference to the equipment and procedure proposed by R.R. Proctor in 1933 (ASTM D698, 2012; BNQ 2501-250, 2013; BS 1377,1990; NF P94-093, 2014). This test determines the relationship between moulding water content and dry unit weight of soils compacted in mould with a 24.5 N rammer dropped in a free fall from a height of 305 mm producing a compacting volumetric energy of 600 kN-m/m³. The diameter of the cylindrical mould is 101.6 mm or 152.4 mm and the height is 116.4 mm. In the original Proctor test, the rammer blows were applied as firm strokes, producing variable compaction effort with the operator. Compactability is the ability of earth to be compacted by static pressure or dynamic compaction so that its volume is reduced. To attain maximum compaction, the earth must have a specific water content, the so-called “optimum water content,” which allows particles to be moved into a denser configuration without too much friction.

Moisture content test

The moisture content is determined on soil samples and the mass of the sample depends on the maximum grain size. In BNQ 2501-170 (2014), it varies from 10 g for soil with maximum particle size of 400 µm to 1000 g for soil with maximum particle size of 56 mm. The sample is heated to 110°C ± 5°C until a constant mass is obtained. These parameters vary between various international standards (ASTM D2216, 2010; BS 1377, 1990; ISO 17892-1, 2014). For example, for the ISO standard the drying temperature is equal to 105°C ± 5°C and the mass of the sample according to the particle size is different (ISO 17892-1, 2014). The standards specify that this method has interferences for soils containing gypsum, hydrated minerals and organic matter. The amount of water can also vary for soil with significant content in salt and other dissolved materials.

Specific gravity test

The specific density is a relative density, the ratio of mass of an aggregate to the mass of a volume of water equal to the volume of the aggregate particles, also known as the absolute volume of the aggregates. This relative density is needed to calculate the volume occupied by the components of earth mixes. The specific gravity can be determined with a pycnometer for the gravimetric procedure. A Le Chatelier flask is used in the volumetric procedure. The sample is dried at 110°C until the mass is constant, or the natural conditions of the aggregates. Several international standards exist for the measurement of this characteristic: ASTM C128-15 (2015), BS 1377 (1990) and EN ISO 17892-3 (2015).

Methylene blue value and the activity of clay minerals

The methylene blue test aims to detect clay minerals in aggregates fines. It is based on an ion exchange phenomenon between methylene blue cations and clay ions that is possible thanks to the large surface area and negative charge of clays. The amount of absorbed methylene solution

varies according to the amount of clay minerals and clay type, cation exchange capacity and specific surface area. Based on this test the specific surface area of soils can be determined. Generally, the sieve at 400 μm is used for this test. There are two main test methods for the methylene blue test: titration method and “spot-test” method.

The first method is described in ASTM C1777 (2015). The test specimen in a methylene blue solution is shaken twice for 60 s, with a rest of 180 s between the two shakings. The mixture is filtered and a sample from the filtered solution is diluted for the measurement with a colorimeter. The concentration adsorbed is calculated from the initial concentration and the final concentration.

The “spot-test” is described in NF P 94-068 (1998), EN 933-9+A1 (2013) and ASTM C837 (2009). Depending on the soil, a mass of 30 to 60 g is taken for high clayey soil and 60 to 120 g for less clayey soil. The soil sample is then dissolved in 500 ml of distilled water, each time 5 ml of methylene blue solution (10 g/L) is added to the soil solution. One drop of the mixture is placed onto a paper filter after 1 min. The test ends when the dye forms a second lighter coloured blue halo around the aggregate dye spot and stays stable over five consecutive spots without addition of methylene blue to the soil solution. NF EN 933-9+A1 (2013) follows the same procedure but with a soil sample mass higher than 200 g depending on the sample moisture. ASTM C837 (2009) follows the same procedure but involves the use of 2 g of soil samples and of acidic solutions (pH value from 2.5 and 3.8). However due to the small amount of investigated soil, ATSM C837 (2009) is more suitable for homogeneous and fine materials.

The methylene blue value (MBV or V_{BS}) is reported in mg/g. A high methylene blue value indicates the presence of clays and allows the definition of six categories of soil as described in Table 2-6 (NF P11-300, 1992).

Table 2-6. Definition of soil categories according to Methylene Blue Value (V_{BS}) (NF P11-300, 1992)

V_{BS}	Soil categories
$0.1 \leq V_{BS} < 0.2$	Water insensitive
$0.2 \leq V_{BS} < 1.5$	Sandy and silty
$1.5 \leq V_{BS} < 2.5$	Sandy-clay
$2.5 \leq V_{BS} < 6$	Silty moderately plastic
$6 \leq V_{BS} < 8$	Clayey
$8 \leq V_{BS}$	Heavy clayey

2.4.2 Study of data from literature

For physical and geotechnical properties, the most numerous results in the literature concern the Atterberg limits. Tables 2-23 to 2-27 in appendix B show the Atterberg limit values for various techniques of earth construction. The most numerous results concern CEB (16 in 10 papers) and adobes (55 in 14 papers). For the other techniques, the number of values available in the literature is too low to be meaningful. We will therefore focus our analysis of the Atterberg limits on soils used for CEB and adobes. Figure 2-7 shows the frequencies of occurrence of these limits for these two techniques.

Figure 2-7 also shows that the liquid limits are relatively dispersed. For CEB, 75% of the soils studied have liquid limits ranging between 25 and 45% that is slightly higher than for adobes. For adobes, 67% of soils have liquid limits ranging between 20 and 40%. However, if we compare the averages obtained for the values of the two techniques, they are exactly the same for the two materials (37%).

For the plastic limit, the results are much less dispersed and quite similar for the two techniques: the majority of the plastic limit values ranges between 15 and 29% (94% and 76% of the values for the CEB and the adobes respectively).

Finally, the liquid limits being different, it is therefore normal to observe marked differences for the plasticity index between the two techniques. For CEB, the frequency distribution is very centred: 50% of the values range between 30 and 34%. For adobes, the results are much more dispersed between 20 and 39%. A hypothesis to explain this difference could be the existence of a standard (French but used in many other countries) on the CEB which indicates a zone recommended for the Atterberg limits (XP P13-901, 2001). For the manufacture of adobes, such normative recommendation does not exist and researchers working on the subject often use the soil on the site directly without seeking to modify their granularity or their Atterberg limits, what leads to a greater variability of characteristics. Furthermore, the majority of the characterization studies on adobe are on vernacular constructions, built when normative requirements were not established.

The results of Figure 2-7 are placed in the Casagrande plasticity chart on Figure 8. According to the standards ASTM D2487 (2017) and ASTM D2488 (2017), the signification of the acronyms is:

- CL = Lean Clay: inorganic clays, sandy clays, silty clays, lean clays, low to medium plasticity, no or slow dilatancy;
- ML = Silt: inorganic silts and very fine sands, rock flour, silty or clayey fine sands, slight plasticity to non-plastic, slow to rapid dilatancy;
- CH = Fat Clay: inorganic clays, fat clays, high plasticity, no dilatancy;
- MH = Elastic Silt: micaceous or diatomaceous fine sandy and silty soils, elastic silts, low to medium plasticity, no to slow dilatancy;
- CL-ML = Silty Clay: mixed zone where both CL and ML soils plot.

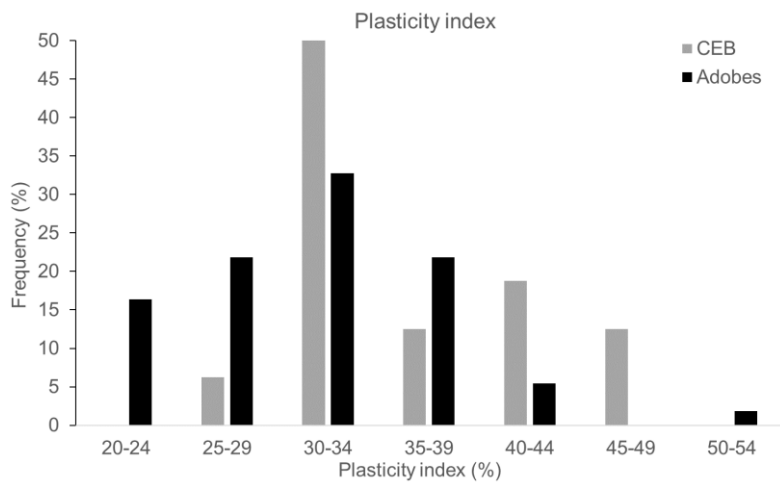
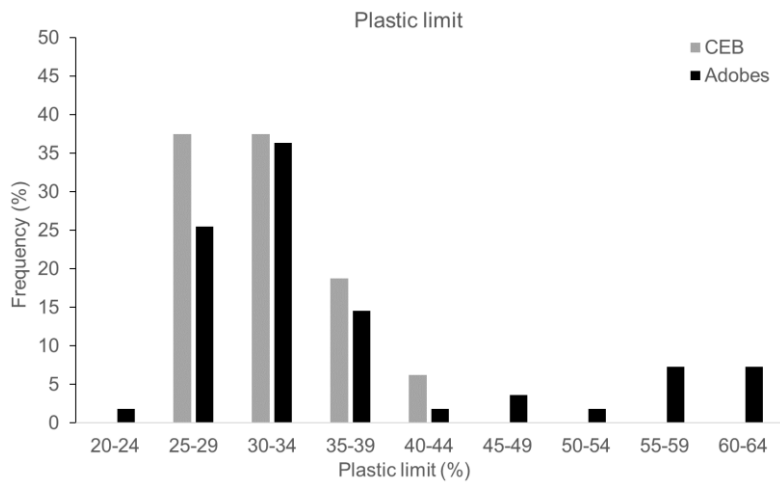
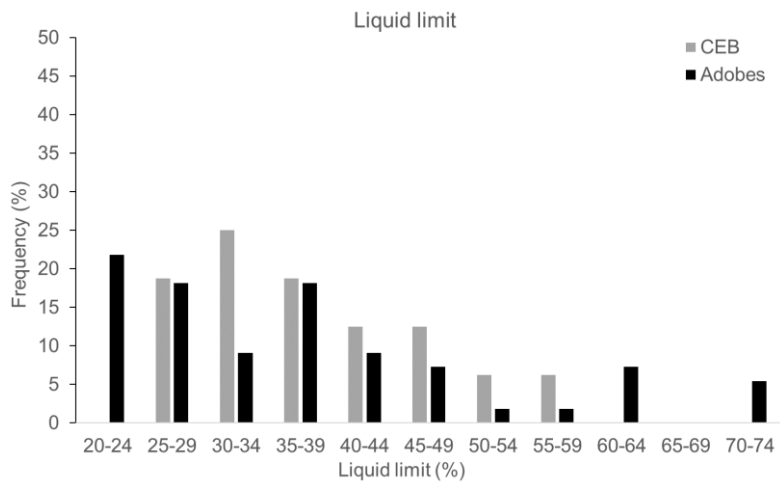


Figure 2-7. Frequency of occurrence of Atterberg limits for CEB and adobes

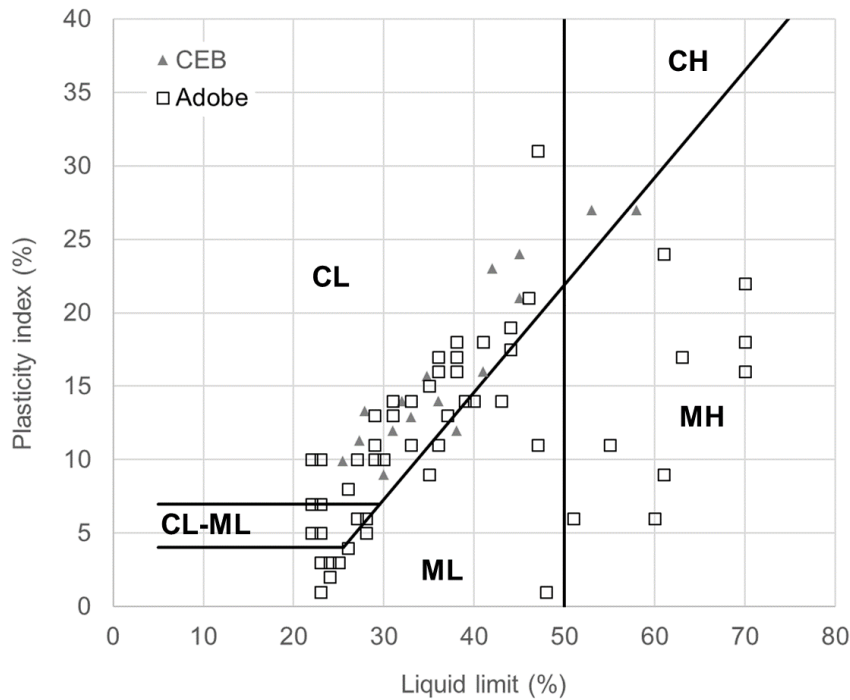


Figure 2-8. Casagrande plasticity chart of CEB and adobes

The results of Figure 2-8 show that the vast majority of results are in the CL category corresponding to lean clay. For adobes, few analyses are in the MH category corresponding to elastic silt but this concerns a minority of the values (around 16%). These analyses correspond to the higher values of plastic limits (higher than 40%).

The zones for the Atterberg limits of CEB and adobes recommended by Houben and Guillaud (2006) are possible to add on this chart. It is interesting to note that for the CEB, these are the same limits that have been included in the French standard (XP P13-901, 2001). The results with the recommended limits are shown in Figure 2-9.

Figure 2-9 shows that the majority of the results obtained for the CEB are within the recommended limits. But, for adobes, this is not really the case and the Atterberg limits of adobes are more within the limits recommended for CEB with the exception of the few analyses in the MH category with high plastic limits. As we have seen for particle size distribution, these recommended areas are essentially indicative and many soils used with success in earth construction materials do not respect them.

For the other geotechnical characteristics, the studies on adobes give some values of MBV (13 in 4 articles). These values are between 0.16 and 0.60 mg/g with an average value equal to 0.34 mg/g, which would correspond to the soil category "sandy and silty" in comparison with the values in Table 2-6. However, the number of Methylene Blue values (V_{BS}) available is too limited to enable draw generalizable conclusions. For the other techniques, the number of results is much lower (3 in 2 articles for CEB, 0 for rammed earth and cob and 3 in one article for extruded earth bricks).

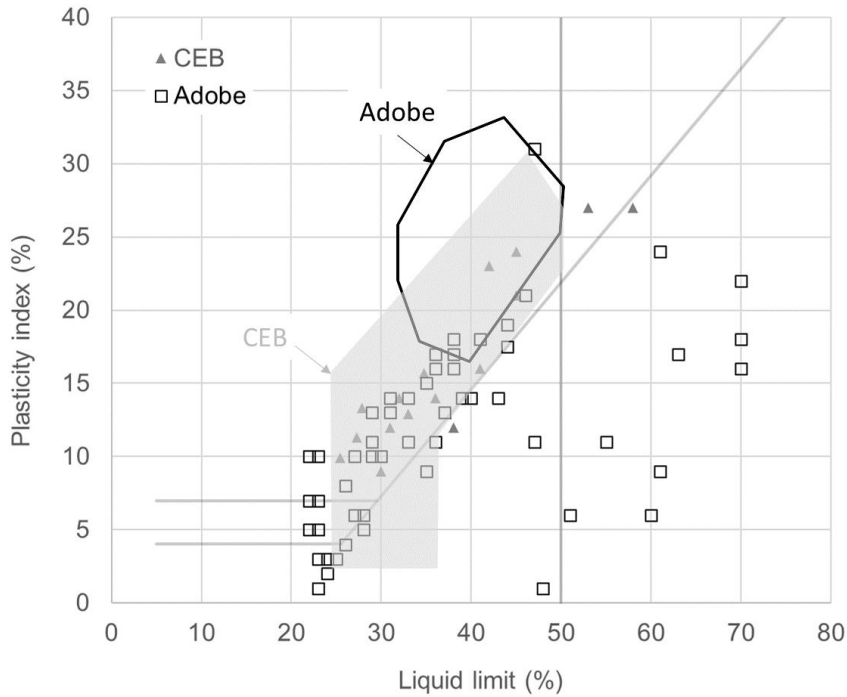


Figure 2-9. Recommendations on Atterberg limits for CEB and adobes

Concerning the optimal compaction characteristics of soil using the Proctor test, the trend is a little opposite because these data are more available for CEB (11 in 6 articles) and rammed earth (19 in 7 articles) in comparison to adobes (1 in 1 article), extruded bricks (0) and cob (4 in an article). This could be easily explained by the methods used to manufacture CEB and rammed earth, which is based on optimal compaction of the earth and which therefore uses the results of Proctor tests as formulation parameters. The values of optimum moisture content and dry density of earth used for CEB and rammed earth are given in Tables 2-28 and 2-29. The Proctor test results are reported for rammed earth, CEB and cob in Figure 2-10.

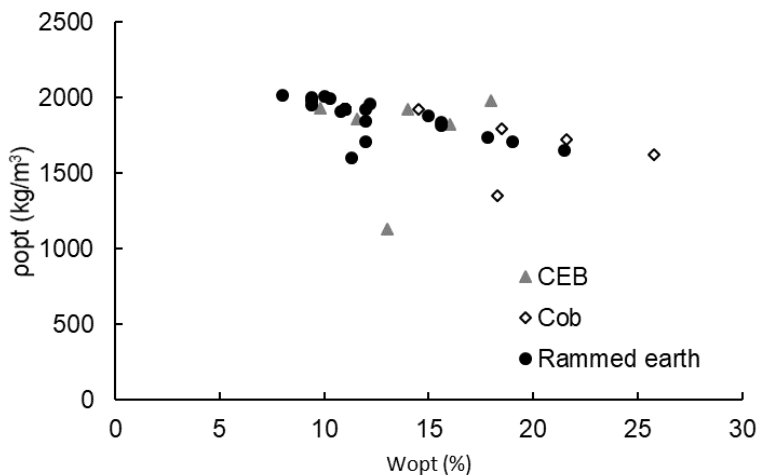


Figure 2-10. Results from the Proctor tests (optimum density (ρ_{opt}) and optimum water content (w_{opt}) for CEB, cob and rammed earth

The earth optimum water content is lower for rammed earth, while it is higher for the cob. The higher densities are found at lower water content. For rammed earth, the optimum water content varies between 8 and 21.5% and most values are below 12%. For CEB, the optimum water

content is found between 9.8% and 18%. For cob, the optimum water content is between 14.5% and 25.8%. More characterisation is needed on the earth properties that allow engineering design, such as compressive strength, soil friction angle, California bearing ratio (CBR) and vane strength. The German standard on earth blocks and earth masonry mortar specify strength classes (Schroeder, 2018). More data is needed to correlate the geotechnical properties of earth used in construction to engineering design properties.

2.5. Chemical characterisation

The chemical characterization aims at determining the chemical properties of the soils used for earth construction material. Many chemical properties could be measured but the most important is then the elemental chemical composition since it permits, in combination with the mineralogical qualitative analysis, to calculate the mineral composition of the material (see section 2.6). For physico-chemical analysis, the samples must be prepared based on ISO 11464 (2006). Other chemical parameters, such as the amount of organic matters, the soluble salts or the pH, are important especially in the case of stabilization using mineral binders because they could affect the reaction with the binders.

2.5.1 Procedures and standards

Chemical composition (major elements)

Two main techniques are used in the literature: the Inductively Coupled Plasma - Atomic Emission Spectrometry (ICP-AES) (or Inductively Coupled Plasma - Optical Emission Spectrometry (ICP-OES)) and the X-ray fluorescence spectroscopy.

Analysis by the ICP-AES (with the exception of laser ablation systems), requires samples to be completely dissolved (digested) into a solution. Hence, a sample of the material in powder ($\leq 80 \mu\text{m}$) is melted in combination with lithium metaborate (LiBO_2) and/or lithium tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$) to form beads. These beads are dissolved in one or more acids as hydrofluoric (HF), nitric (HNO_3) or hydrochloric (HCl) acids. The obtained solution is then used for the ICP analysis.

The X-ray fluorescence spectroscopy test is carried out either on beads prepared with the same procedure as for the ICP test, or on pressed tablets of the material in powder ($\leq 80 \mu\text{m}$), aggregated with an organic waxy binder or not. The principle of X-ray fluorescence is the analysis of the X-ray emitted by the matter excited by an incident X-ray source.

Two other techniques could be used too and are based on the same principle: it consists in the analysis of the X-ray emitted by a beam of electrons. The interactions between the electron beam and the matter are used in two techniques: the Electron Dispersive Spectrometer (EDS) analysis coupled with Scanning Electron Microscope (SEM) or microprobe analysis. The main inconvenient of these techniques is that the result may not be representative of the entire material since the analysis is carried out on a small zone of the sample but they allow having an image of this zone.

Organic matter content

The organic matter is rarely a problem because earth used for construction is often extracted from the pit and in this case, the amount of organic matter is negligible. But, in some specific cases, e.g. some Canadian soils rich in organic matter, it could be necessary to measure the organic matter content of the soils (Ouellet-Plamondon et al., 2017). Van Damme and Houben (2017) also reviewed the soil classification systems and, preventing organic soil for construction, refer that subsoil is preferred. Gomes et al. (2014) have extracted the requirements for organic matter from the literature and their results are presented in Table 2-7.

This content can be determined either by the calcination method (XP P94-047, 1998) or the chemical method (XP P94-055, 1993). For the calcination method, several dried samples are respectively weighed before and after a 3h (or more if necessary to achieve a constant mass) heat at 450°C to 550°C. The mass loss is assumed to represent the organic matter. The materials' organic matter content is then computed as the mean value of the mass loss percentages of the respective samples. The chemical method consists in determining the carbon content of a soil sample by mixing it with an oxidizing solution (potassium dichromate with sulphuric acid). Once the oxidation has been completed, the quantity of products, which has reacted with the carbon of the soil, is measured.

Table 2-7. Requirements for organic matter (adapted from Gomes et al. (2014))

Reference	Requirements for organic matter
(Keable, 1996)	“Soil shall be free of organic materials” (p. 18)
(NZS 4298, 1998)	“Soil shall not be used if contains organic matter prone to rot or breakdown within the wall” (p. 15)
(SAZS 724, 2001)	“Soil should be free from organic material” (p. 6)
(HB 195, 2001)	“A musty aroma indicates an unacceptable quantity of organic matter and the soil should, therefore, be rejected” (p. 131)
(Walker et al., 2005)	< 2% by mass (p. 37)
(Houben and Guillaud, 2006)	< 2 to 4% by mass (p. 34)
(New México Code, 2006)	“The soil shall be free of all organic matter” (p. 5)
(Lehmbau Regeln, 2009)	“The smell test is sufficient for rejection of a soil: organic soil is identifiable by its strong smell of humus. The smell test should be performed immediately after extraction of the soil” (p. 8)

Calcite content

Free carbonates in soils, such as calcite, aragonite and dolomite, affect their physical and chemical properties. The determination of calcite content or equivalent CO₂ is an important point. The evaluation of calcite content can be realised by various techniques: titration (BS 1377-3, 1990), gravimetric (BS 1881-124, 2015) or volumetric measurement (EN ISO 10693, 2014 and NF P94-048, 1996). In all cases, the measurement principle is based on the determination of the volume of CO₂ released by the soil sample under the action of excess hydrochloric acid. A quantity of soil (10 g for soils estimated to be low in carbonates and 0.25 g for chalky soils) is mixed with a solution of hydrochloric acid. Carbonates being unstable at a pH value lower than 7, dissociate leading to the formation of CO₂.

According to EN ISO 10693 (2014), ASTM D4373 (2014) and NF P94-048 (1996), the CO₂ is recovered by the intermediary of a calcimeter. The volume of gas produced allow the evaluation of CO₂ content in the sample.

According to BS 1377-3 (1990), the excess hydrochloric acid is dosed from a NaOH solution and an indicator solution. Thus, evaluating the amount of acid consumed by the reaction, it is possible to calculate the amount of CO₂ initially present in the sample.

Loss on ignition

The loss on ignition (LOI) is the mass fraction lost by a dried sample by ignition at a specified temperature. The LOI is related to the organic content of certain soils (sandy, clayey, chalky). The procedure for soil is specified in BS 1377-3 (1990) and XP P94-047 (1998). A dried soil sample passing the 2 mm test sieve is heated to a constant mass at 550°C, not less than 3 hours. The LOI is determined by the ratio between the mass loss of samples during heat treatment and the initial dried mass of the sample.

When the purpose is to determine all volatile species (hydroxyls, carbonates), the test is then conducted on samples at the temperature of 1000°C during two hours. The change in mass after 1000°C heating represents the total mass loss of the sample including organic matter, hydroxyl for clay minerals and carbonate. Usually the loss on ignition is also determined during the chemical composition test by ICP.

pH measurement

The soils' pH measurement is useful to know its minerals solubility and its ion mobility. The test is performed on air dried samples of the soil without coarse grains (≤ 2 mm). A given amount of the material is mixed with either a pure water, a 0.01 mol/L solution of chloride calcium (CaCl₂) or 1 mol/L solution of potassium chloride. The suspension is stirred for a few minutes, covered with a cover glass and allowed to stand for a couple of hours before pH value measurement. The suspension needs to be stirred right before the pH value measurement. The European standard (EN 15933, 2012) and International standard (EN ISO 10390, 2020) recommend a volumetric ratio of 1:5 and a rest at most 3 hours, while the British standard (BS 1377-3, 1990) recommend a volumetric ratio of 1:2.5 and a rest of at least 8 hours, and the American one (ASTM D 4972 (2001) states a mass concentration (10 g of air-dried soil for 10 mL of solution) and a rest of 1 hour. This last procedure can induce a bias in the case of lightweight soil. Generally, standards recommend performing a test with pure water and one with chloride solution for the determination of the soils' pH value. Both preparations are required to fully assess the soils' pH. A pH meter or a pH paper (low accuracy) can be used for the measurement.

Cation exchange capacity test

The clay minerals in fine soils have a negative surface charge that is balanced by bound cations at the mineral surface. These bound cations can be exchanged by other cations in the pore water. Cation-exchange capacity (CEC) is defined as the amount of positive charge, generally calcium (Ca), sodium (Na), magnesium (Mg) and potassium (K), that can be exchanged per mass of soil. This test makes it possible to estimate the behaviour of the soil during stabilisation with inorganic binder and, therefore, the cationic exchanges between inorganic binder and clays. It has two origins: the isomorphic substitution in the tetrahedral and/or octahedral sheet of clays, which is not dependent of the system pH value, and the dissociation of aluminium groups on the edge of the sheet of clays, which is pH value dependant. CEC is usually measured in centimoles of positive electric charge (cmol_c/kg). Numerous techniques were developed for the CEC measurement of soil. Commonly CEC is measured by displacing all the bound cation with a concentrated solution of another cation, and then measuring either the displaced cations or the amount of added cations that is retained.

Different solutions are used based on salt (ammonium acetate (Thomas, 1982), sodium acetate (Bower et al., 1952), barium chloride (Gillman, 1979), cationic surfactants (Janek and Lagaly, 2003), metal-organic complex (cobalt hexamine (Morel, 1958; Mantin and Glaeser, 1960; Orsini and Remy, 1976), silver-thiourea (Chhabra et al., 1975), copper bisethylenediamine (Bergaya and Vayer, 1997) or copper triethylenetetramine (Meier and Kahr, 1999). Standards and literature focused mostly on three procedures using ammonium acetate (NF X31-130, 1999 and ASTM 7503-18, 2018), cobaltihexammine trichloride (NF X31-130, 1999; ISO 23470, 2018) and barium chloride (ISO 11260, 2018).

Cobalt hexamine chloride method (ISO 23470, 2018) - The exchange is carried out by simply shaking the test portion in the reagent. For a given volume of reagent (100 mL), the quantity of samples weighed (2.5, 5 or 10 g) is such that a sufficient concentration of cobalt hexamine ions remains in solution. This concentration is determined by spectro-colorimetry without chemical pretreatment of the solution. The loss of cobalt hexamine from solution gives the CEC of the sample. Exchangeable cation contents are measured either by flame atomic emission spectrometry for K or by flame atomic absorption spectrometry for Ca and Mg. This procedure is recommended for soil with a natural pH value lower than 6.5.

Barium chloride method (ISO 11260, 2018) - A soil test portion of 2.5 g (< 2 mm) is shaken for 1 h with 30 mL of 0.1 mol.L⁻¹ BaCl₂ solution. The solid and liquid phases are separated by centrifugation. This operation is repeated twice and the three supernatants are collected for the determination of exchanged cations. After equilibrating under shaken overnight the soil with 30 mL of 0.0025 mol.L⁻¹ BaCl₂, the solid phase is shaken once again, but this time with 30 mL of 0.02 mol.L⁻¹ magnesium sulphate (MgSO₄) solution overnight. The adsorbed barium exchanges with magnesium and precipitates in the form of BaSO₄. The residual content of magnesium in leaching solution is measured by flame atomic absorption spectrometry and subtracted from the initial content. The difference gives the CEC value.

Ammonium acetate method (NF X31-130, 1999) - Widely used throughout the world, the ammonium acetate method was proposed by Metson (1956). The saturation of the exchange sites by ammonium is carried out by percolating a 1 mol.L⁻¹ ammonium acetate solution (75 mL) through a test portion of 2.5 g of soil. The excess reagent is eliminated with several rinses with ethanol (75 mL). After drying in air, the solid phase is agitated in 50 mL of a 1 mol.L⁻¹ solution of sodium chloride. The exchanged ammonium is measured by spectrophotometry, which permits the measurement of CEC. However, the obtained solution must be used with caution. In fact, the use of ammonium acetate induces a measurement carried out at a pH value of 7, due to the large excess of sodium acetate, and the dissolution of a part of carbonate species.

Ideally, the CEC measurement should be performed at the natural soil pH value in order to avoid the modification of electrical charges (Charlet and Schlegel, 1999). This induces a dissolution of carbonate and a modification of CEC value. Most of the literature agree that the use of cobalt hexamine trichloride procedure gives reliable and accurate value of the effective CEC, that is to say the CEC value at soil natural pH value (Ciesielski et al., 1997).

Soluble salt content (nitrate, sulfate, chloride)

The most common soluble salts in soils are the cation calcium (Ca⁺²), magnesium (Mg⁺²) and sodium (Na⁺), and the anion chloride (Cl⁻), sulphate (SO₄²⁻) and bicarbonate (HCO₃⁻). Potassium (K⁺), ammonium (NH₄⁺), nitrate (NO₃⁻) and carbonate (CO₃²⁻) can also be found in most soils in a lower quantity. The soluble salt content of the soil is an important element that determines the quality of the soil for its use in construction (Table 2-8).

The determination of the soluble salt content can be approached in different ways. It is indeed possible to assess this content from a qualitative point of view. In this case, standard ISO 11265 (1994) is used. The soil sample is extracted with water with an extraction ratio of 1: 5 (m/V). The specific electrical conductivity of the extract is then measured. The higher the concentration of salt in a solution, the higher will be the electrical conductance (the reciprocal of resistance) (Table 2-9).

Table 2-8. Requirements for salt content (adapted from Gomes et al. (2014))

Reference	Requirements for salt content
(NZS 4298, 1998)	“Shall not be used soils containing water soluble salts to an extent which will impair the strength or durability of the wall” (p. 15)
(SAZS 724, 2001)	“Soil should be free from salts such as sulphates” (p. 6)
(Walker et al., 2005)	< 2% (p. 37)
(Houben and Guillaud, 2006)	“Sulphates of sodium, magnesium and calcium are dangerous to soils used in earth construction, since they crystallize, making it easily broken” (p. 23)
(New México Code, 2006)	< 2% (p. 5)

Table 2-9. Classification of salinity according to soil electrical conductivity (ECe in $\mu\text{S}/\text{cm}$ at 25°C) (Rhoades, 1982)

Grade	CEe ($\mu\text{S}/\text{cm}$)	Soil quality
I	0 to 500	Unsalty
II	500 to 1000	Slitghly salty
III	1000 to 2000	Salty
IV	2000 to 4000	Very salty
V	> 4000	Extremely salty

Another possibility is to try to quantify the amount of each salt, especially sulphate, nitrate and chloride. Sulphate quantification is described in BS 1377-3 (1990) and ISO 11048 (1995). The sulphate is extracted from the dried soil samples using dilute hydrochloric acid or water in a soil/added water ratio of 1:2 or 1:5 (m/V). The sulphate content of these extracts is determined by a gravimetric method according to which barium chloride is added to the aqueous or acid extract. The precipitate of barium sulphate is dried and weighed and the sulphate content is then calculated from the mass of the soil used in the analysis and the mass of precipitated barium sulphate.

Chloride quantification is described in BS 1377-3 (1990) based on Charpentier-Volhard’s method. The chloride is extracted from the dried soil samples water. Silver nitrate (AgNO_3) is added to the aqueous soil extract. The solution is then diluted and then titrated using a solution of potassium thiocyanate (KSCN) in the presence of ammonium ferric sulphate as a coloured indicator. The soluble chloride is calculated based on the volume of silver nitrate added and on the mass of the soil used in the analysis.

Nitrate, nitrite and ammonium quantification are described in ISO 14256-2 (2005) using automatic measurement by spectrophotometry. The homogenized soil samples are extracted using a potassium chloride solution (1 mol/L). The concentrations of mineral nitrogen compounds, namely nitrate, nitrite and ammonium, are determined in the extracts by automated spectrophotometric methods.

2.5.2 Study of data from literature

Table 2-10 shows the chemical composition of the soils used in the studies of the literature for Extruded Earth Bricks (EEB), Compressed Earth Bricks (CEB) and adobes.

The study of this table shows that these results are difficult to exploit and that it is difficult to compare materials with each other. Nevertheless, we can extract some information from Table 10. First of all, we note that the following elements can be considered, in all the soils analysed, as “minor”, that means that their concentration is always lower than 2% (expressed as oxides): Na_2O , TiO_2 , P_2O_5 and MnO . If we consider the average concentration, we can classify the other oxides in ascending order: K_2O , MgO , Fe_2O_3 , Al_2O_3 , CaO and SiO_2 . The cases of iron and calcium are interesting because depending on the type of soils, the contents could be very low or very high. For iron, the concentrations range from 1.7% to 15.0%. This element will play a relatively small role in the behaviour of earth materials. The iron oxides, being often in the form of goethite ($\text{FeO}(\text{OH})$), play the role of inert raw earth materials. However, the nature and the content of iron oxides will have a very important influence on the colour of the earth, especially in the field of fired clay bricks. In the case of calcium, some soils may not contain it at all (0.03% for soil B studied by Ammari et al. (2017)) and others contain very large amounts: 31.8% for soil M studied by Ammari et al. (2017) or 35.3% for the soil studied by Laborel-Préneron et al. (2017). In these two cases, the loss on ignition is also very high (32.9% and 31.9% respectively), which leads to the conclusion that calcite (CaCO_3) is present (approximately 60% in both cases), which shows that these soils are strongly calcareous.

If we do not consider the calcium present in calcareous soils, the two major elements of the soils are silicon and aluminium, which seems to be logical for clayey soils: clays being phyllosilicates are rich in these two elements. The results of Castrillo et al. (2017) were obtained by X-ray fluorescence spectroscopy and do not take into account the loss on ignition (LOI). The researcher normalized the concentrations of the oxides to 100%, which does not allow a direct comparison with the results of the other studies that integrate the loss on ignition. Finally, the measurement of total sulphates and chlorides contained in soils is relatively rare (only two out of nine studies). In some studies, the contents of soluble chlorides and sulphates are measured but this is very rare too. We can cite the study of Galan-Marin et al. (2010) who measured soluble chloride contents equal to 0.03% or the study of Hakimi et al. (1996) with content of sulphates and soluble chlorides equal to 0.64 and 0.07% respectively. Generally, these soluble salt contents are extremely low and rarely measured by the researcher.

In addition to the chemical composition, the two most frequently encountered chemical characteristics in the literature are the measurements of pH value and organic content. The values found in the literature are given in Table 2-11. It is important to note that in all the references studied almost none give CEC values in exception to Dove et al. (2016) and Hamard et al. (2018).

Table 2-10. Chemical composition of the soils used in the studies of the literature (LOI: Loss On Ignition)

Reference	Name	Type	Major elements (%)												
			SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	K ₂ O	MgO	Na ₂ O	TiO ₂	P ₂ O ₅	MnO	SO ₃	Cl	LOI
(Fgaier et al., 2016)	A	EEB	64.22	14.59	1.04	5.66	3.08	1.51	0.35	0.9	n.m.	n.m.	n.m.	n.m.	8.75
	B		50.72	12.83	1.61	15.03	2.63	2.27	0.28	0.8	n.m.	n.m.	n.m.	n.m.	14.11
	C		73.97	8.82	4.18	3.22	2.0	0.73	1.01	0.77	n.m.	n.m.	n.m.	n.m.	6.03
(Aubert et al., 2012)			64.7	16.6	1.1	4.8	4.0	1.1	0.2	0.6	0.1	0.1	n.m.	n.m.	5.9
(Ammari et al., 2017)	M	CEB	22.3	6.0	31.8	1.7	0.5	2.1	0.3	0.2	0.8	0.1	1.29	n.m.	32.9
	B		79.4	11.9	0.03	1.8	0.3	tr	0.0	1.3	-	0.0	0.0	n.m.	6.3
(Galan-Marin et al., 2010)			54.7	19.7	0.9	8.6	3.9	3.6	1.8	1.0	0.2	0.1	n.m.	n.m.	5.0
(Laborel et al., 2017)			18.73	7.47	35.3	3.39	0.9	1.27	0.09	0.39	0.09	0.03	n.m.	n.m.	31.92
(Aubert et al. 2015)			77.81	10.06	0.33	3.23	1.6	0.39	0.24	1.08	0.08	0.04	n.m.	n.m.	
(Castrillo et al., 2017)	PrA1	Adobe	29.7	6.38	41.0	11.9	2.32	6.3	0.48	1.11	-	0.12	0.12	-	-
	PrA2		10.7	2.4	81.0	2.95	0.72	1.39	-	0.39	-	-	-	-	-
	PrA3		10.8	1.72	48.3	1.66	0.47	1.72	1.31	-	0.35	-	33.2	-	-
	PrA4		22.6	4.26	46.6	5.43	1.44	2.55	0.75	0.69	0.21	-	14.9	-	-
	PrA5		13.9	2.59	50.1	3.05	0.82	1.81	1.02	0.29	0.32	-	25.6	-	-
	PrA6		22.5	3.6	54.4	3.35	1.97	2.51	1.09	0.74	0.71	0.11	1.99	0.46	-
	PrA8		18.8	2.72	59.5	2.81	1.59	1.69	0.71	0.66	0.97	0.10	2.82	0.30	-
	OA1		41.6	7.53	28.4	11.4	1.74	5.26	1.83	1.3	-	-	0.32	0.24	-
	OA2		28.8	5.45	46.8	9.01	1.74	4.2	1.2	0.91	-	-	0.18	1.21	-
	OA3		24.1	4.46	61.0	4.99	1.32	2.42	0.17	0.72	-	-	0.12	0.26	-
	OA4		44.6	8.96	21.0	13.4	2.21	6.87	1.0	1.15	-	-	0.12	0.18	-
	OA5		30.4	6.04	48.2	7.84	1.85	3.42	0.43	0.97	-	-	0.22	0.25	-
	OA6		40.4	8.18	27.3	12.8	1.72	6.05	1.46	1.32	-	-	0.20	0.10	-
	LyB		40.7	8.1	26.9	12.3	1.56	6.02	1.85	1.26	-	-	0.35	0.59	-
AthB	26.1	4.5	55.6	5.84	1.6	3.29	0.61	0.73	0.43	-	0.70	0.23	-		
(Millogo et al., 2014)			50.6	23.44	2.48	5.81	1.69	0.02	0.05	0.65	0.17	0.02	n.m.	n.m.	14.91
(Millogo et al., 2016)			49.88	25.95	0.05	9.69	0.39	0.17	0.03	1.12	0.04	0.05	n.m.	n.m.	11.78
(Dao et al., 2018)			66.13	14.38	0.41	6.68	1.0	0.45	0.24	1.09	0.06	0.15	n.m.	n.m.	8.93
Maximum (%)			79.40	25.95	81.00	15.03	4.00	6.87	1.85	1.32	0.97	0.15	33.20	1.21	32.90
Average (%)			39.96	9.21	28.71	6.61	1.67	2.56	0.71	0.85	0.32	0.08	5.13	0.38	12.61
Minimum (%)			10.70	1.72	0.03	1.66	0.30	0.00	0.00	0.20	0.04	0.00	0.00	0.10	4.77

Table 2-11. pH values, organic and CaCO₃ contents of the soils used in the studies of the literature

Reference	Name	Type	pH	Organic content (%)	CaCO ₃ (%)	
(Dove et al., 2016)	U	CEB	4.8			
	V		6.8			
	W		6.9			
(Galan-Marin et al., 2010)			8	0	12.4	
(Hakimi et al., 1996)	1			0.78	16	
(Venkatarama Reddy, 2002)	BCS		8.95	0.67		
	TB1		8.05	2.32		
	TB2		7.22	1.40		
	TB3		6.58	1.26		
(Laborel et al., 2017)					60	
(Duarte et al., 2017)	CLG-1S				0.73	
	CTL-2S				1.89	
	CTL-2A				1.58	
	CHG-3SS				1.44	
	CHG-3SP				0.78	
	CHG-3AR				1.25	
	CHG-3AA				1.48	
	CGR-4S				0.58	
	CHJJ-1S				0.44	
	CHJJ-5CA				1.47	
	LMD-6S			1.42		
	MNG-7A			2.66		
	NDD-8A			1.27		
	NPK-9S			0.76		
(Turanli and Saritas, 2011)				2.1		
(Uguryol and Kulakoglu, 2013)			7.4	1.7	22	
(Gomes et al. 2014)	Av	Rammed earth		0.9		
	PD			4.5		
	VC			3.5		
	CZ			1.8		
	Cl			3.6		
	Ar			5.4		
(Araki et al., 2016)	H2			5.8		
(Venkatarama Reddy et al., 2011)	S1			7.7	0.9	
	S2			7.8	0.6	
	S3			8	0.5	
	S4			8.1	0.4	
	S5			8.3	0.3	

Although the measurement of the CaCO_3 content is relatively simple, this content is rarely given in the studies. This is no doubt explained by the fact that calcite could be considered as an inert in earth construction materials and the knowledge of its content in the soil is therefore not essential. The measurement of calcite content could be important in some specific cases of study as for instance for lime stabilised earth constructions. There are in Portugal examples of « military » rammed earth that was used since the XII century mainly for fortresses and that was stabilised with air lime (Parracha et al., 2019b). For these specific cases of study, the knowledge of calcite content is useful because it permits to determine the amount of lime used for the stabilisation of these materials.

The pH values presented in Table 2-11 on soils reported in the literature vary from 4.8 to 9. It is difficult to compare these values directly because we have seen during the presentation of the procedures that the methods used for the measurement of pH value strongly vary (in particular the volumetric ratio), which has consequences on the pH value. Despite this, except the soil “U” studied by Dove et al. (2016) which has a pH of 4.8, the pH values measured on the different soils are relatively close (between 6.6 and 9).

Finally, the organic contents of the soils studied in the literature are often less than 2% as recommended in Table 2-5 but in some studies, the contents may be higher. This is the case in the study of Gomes et al. (2014) who worked on unstabilised rammed earth collected from old constructions in south Portugal. In this study, some samples (Ar, Cl and particularly PD) contained large sized organic matter particles that could explain the high organic content of these materials.

2.6. Mineralogical characterisation

Chemical analysis provides important data on the chemical compounds contained in soils but they do not permit to know under which forms these elements are in the material. The behaviour of clay materials will essentially depend on the mineral form in which these elements are. For example, silicon will not react at all if it is under the form of quartz (SiO_2), clay, feldspar or mica. The mineralogical characterization therefore permits to complete the chemical analysis by determining the nature and, under certain conditions, the quantity of the minerals contained in a sample. Many techniques exist but the most used for the characterization of clay materials are X-Ray Diffraction (XRD), thermal analyses, infrared spectroscopy and microscopic observations.

2.6.1 Procedures and standards

X-ray diffraction

XRD is an analytical technique used for phase identification of a crystalline material and can provide information on unit cell dimensions. The principle consists on placing the crystallized material in an intense beam of X-rays, usually of a single wavelength (monochromatic X-rays). This beam of X-rays is diffracted by the materials: the angles and intensities of diffracted X-rays are measured, with each compound having a unique diffraction pattern. By comparison with standards obtained on reference minerals, it is possible to determine the nature of the crystallized phases contained in the sample.

Conventionally, the tests are carried out on the sample crushed $< 80 \mu\text{m}$. This technique is sufficient for materials without clay minerals or materials containing illite and kaolinite. But, if the sample contains clay minerals with a basal reflection (001) at 14 \AA (typically chlorite, vermiculite or smectite), it is necessary to complete this first test by another one carried out on oriented aggregates using three preparations: air-dried or natural, after glycolation and after heat treatment at 500°C (Thorez, 1975; Moore and Reynolds, 1997).

Thermal Analysis

Three types of thermal analysis exist and could be used to complete the XRD analysis: Differential Thermal Analysis (DTA), Differential Scanning Calorimetry (DSC) and Thermal Gravimetric Analysis (TGA). The principle of TGA consists in weighing the sample over time as the temperature increases. DTA and DSC are relatively similar techniques. In DTA, the material under study and an inert reference are submitted to identical thermal cycles while recording any temperature difference between the two samples. Changes in the sample, either exothermic or endothermic, are thus detected by comparison to the inert reference. In DSC, the difference in the amount of heat required to increase the temperature of a sample and a reference is measured as a function of temperature (generally, the temperature increases linearly as a function of time).

Concerning the mineralogical characterization of earth construction materials, DTA could be used in addition to the XRD to determine the nature of the minerals contained in soil. However, it is relatively rare that it brings new results and it often confirms the qualitative characterization performed by XRD. The results of DSC are less used for the characterization of soil but could be used for the determination of the thermal properties of earth construction materials such as the heat capacity. TGA is very useful because it permits to calculate the content of some minerals contained in soil such as goethite (FeO(OH)) or gibbsite (Al(OH)₃) (loss of weight around 300°C for both minerals), clay minerals (dehydroxylation around 500°C) and calcite (decarbonation around 700°C).

Infrared spectroscopy

Infrared spectroscopy is based on the absorption phenomenon occurring when infrared radiation passes through a material. When a molecule is excited to its own energy of vibration, it absorbs the incident energy, thus allowing the study of the various bonds present in the material. The soils are mainly composed of silicon, aluminium, calcium, iron, alkaline elements and metals. The presence of these elements will induce a large number of possible atomic bonds (Si-O-Si, Si-O-Al, ...), each one having different vibrational modes. The signal processing by Fourier transform allows to highlight vibration bands linked to the covalent bonds of the materials. Certain characteristic bands allow the identification of materials. Analysis can be done in transmission or Attenuated Total Reflection (ATR). In all cases, the range of analysis is in the mid-infrared between 4000 cm⁻¹ and 600 cm⁻¹ (or if possible up to 250 cm⁻¹). Clay minerals are identified from the signal of chemical groups in specific regions (Van Olphen, 1979; Madejova, 2003).

Transmission analysis requires the use of potassium bromide (KBr) for the production of pellets. The preparation is performed by mixing 0.5 to 1.5 mg of prepared soil with 100 mg of KBr in a mortar. The mixture is very finely ground. The mixture is then put into a 13 mm diameter steel pressing die and pressed at 10 tons for 2 minutes. At the end of this time, the pellet is removed from the press and placed in a support suitable for the spectrometer.

The analysis in attenuated total reflection (ATR) allows the study of the soil, without addition of chemical products. The sample is placed on a crystal, usually diamond type, during the analysis. The infrared beam penetrates into the material over a thin thickness of the order of 5 μm. This rapid type of analysis leads to the identification of crystalline or amorphous mineralogical phases, but not to their quantitative evaluation.

Microscopy

Some researchers use SEM (Scanning Electron Microscopy) to complete the mineralogical characterization of soil. That permits to show some pictures of the microstructure of the soil. These observations could be completed by a very useful isolated chemical analysis using Energy Dispersive X-ray Spectroscopy (EDS).

2.6.2 Study of data from literature

Table 2-12 presents the numbers of qualitative and quantitative mineralogical compositions found in the literature. The number of articles where these compositions are presented is given too.

Table 2-12. Numbers of mineralogical studies in the literature (with the number of articles where this qualitative or quantitative mineralogical composition is found).

Technology	“Qualitative” (articles)	“Quantitative” (articles)
EEB	9 (4)	1 (1)
Adobes	46 (10)	6 (6)
CEB	10 (4)	1 (1)
Rammed Earth	13 (5)	1 (1)
Cob	4 (1)	0
Total	82 (24)	9 (9)

Table 2-12 shows that qualitative mineralogical analyses are relatively numerous even if they are found only in 24 articles out of 71 (Table 2-1). All these analysis use X-ray diffractions. Figure 2-10 and Figure 2-11 illustrate a complex case of mineralogical characterization by XRD of clay soil from the study by Ouedraogo et al. (2020). This study focuses on the effects of stabilization by cement or lime of two soils of different mineralogy. XRD patterns of the two soils measured on crushed powder are presented on Figure 2-11.

The XRD pattern on crushed powder leads to the identification of the main constituents of the two soils: quartz (SiO_2), calcite (CaCO_3), feldspar (albite ($\text{NaSi}_3\text{AlO}_8$) and orthoclase (KSi_3AlO_8) and goethite ($\text{FeO}(\text{OH})$). The most interesting result of this analysis is the differences in the nature of clay minerals. Soil B contained illite/muscovite (it is not possible to distinguish these two phases using XRD) and kaolinite. For soil N, it is not possible to determine the nature of clays with the diagram of Figure 2-10 because the peaks at 14 Å can correspond to various types of clay. It is necessary in this case of study to use the oriented aggregate technique, the XRD patterns for which are presented on Figure 2-12.

The analysis of the evolution of the first four peaks of the pattern during the various preparations permits to conclude that the soil N contains three types of clay minerals: illite, chlorite and montmorillonite.

Quantitative mineralogical studies are much rarer (9 in 9 papers). In some papers, the authors provide semi-quantitative studies based on the intensity of the diffraction peaks of X-ray powder diagrams. In these papers, some tables present the minerals present with semi-quantitative criteria, as for example in the study by Gomes et al. (2014): “+++” corresponds to high proportion, “++” to intermediate proportion and “+” to low proportion. These semi-quantitative analyses are only indicative because all minerals do not diffract X-rays with the

same intensity. These semi-quantitative studies are not considered in the quantitative studies listed in Table 2-12.

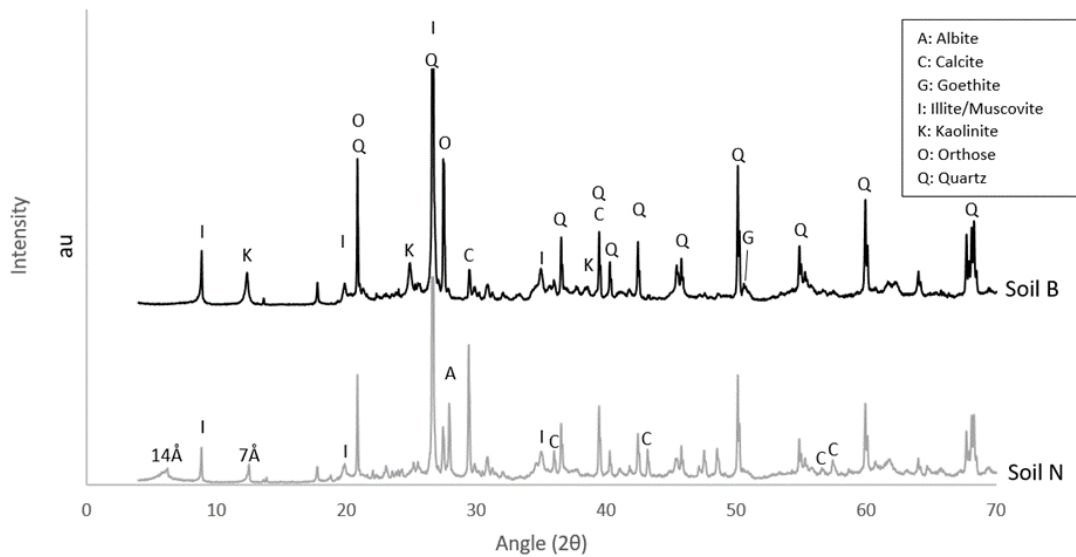


Figure 2-11. X-ray diffraction patterns of two soils studied by Ouedraogo et al. (2020)

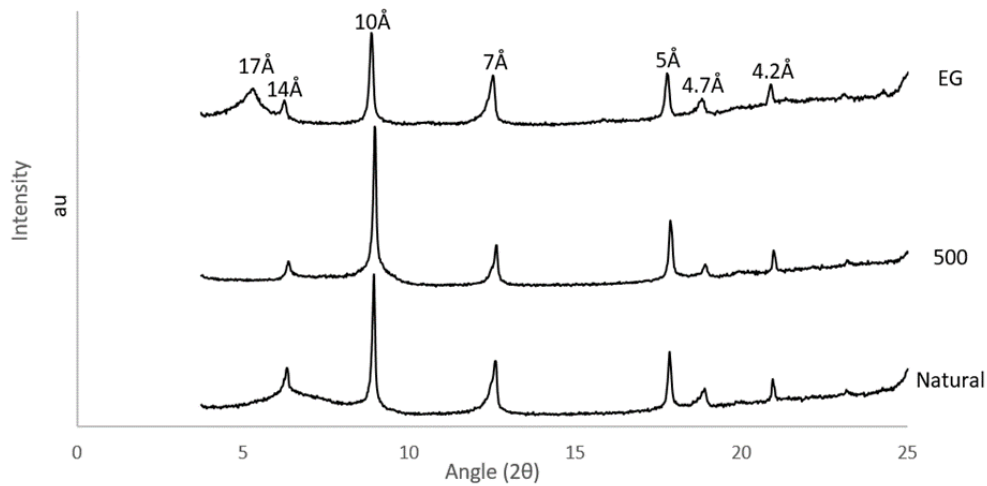


Figure 2-12. X-ray diffractograms of oriented aggregates (EG: ethylene glycol, 500: heated at 50 °C and natural) (Ouedraogo et al., 2020)

In several papers presenting quantitative mineralogical compositions, the methodologies used are questionable. For example, Duarte et al. (2017) have determined the semi-quantitative abundance of the minerals using the Reference Intensity Ratio (RIR) method by XRD. In their article, no detail on this technique was given and the chemical composition of the studied samples was not presented too. It is then impossible to verify the correctness of the compositions given by this method by comparing the calculated mineralogical compositions with the measured chemical compositions. In other articles, Maskel et al. (2010) or Wouatong et al. (2017) give accurate mineralogical compositions but both groups of researchers do not present the method they used to measure these compositions and they do not give any chemical composition that could allow to check their results. The most serious example is undoubtedly

that of the use of the Rietveld method for the calculation of the mineralogical compositions of earth building materials. Rietveld's method consists in calculating a X-ray diagram from crystallographic characteristics of reference minerals and in deducing the proportions of these phases by comparison to the real X-ray diagram of the studied sample. This quantification method is both very powerful and very robust, but also very dangerous for inexperienced users. Indeed, there are a lot of quantification software using Rietveld's method, which is very easy to use, automated and which will always give a final result to the user even if this result is perfectly aberrant. This quantification method is based on many hypotheses, which are not always verified and can lead to aberrant results. To illustrate this, it is possible to refer to the results obtained by Costi de Castrillo et al. (2017) but other studies have certainly used this method as the previously mentioned studies in which the quantification method was not specified. Costi de Castrillo et al. (2017) have compared adobes from pre-history to date. They characterized in depth 15 samples assessing in particular the chemical composition using XRF (Table 2 in the article) and the mineralogical composition with very high precision using the Rietveld method (Table 3 in the article). A quick comparison of the results shows that there is no consistency between the values of these two tables. It is likely that the chemical composition was correctly carried out, which means that the results of the quantification using Rietveld's method are false. To illustrate this, we will take a very simple example: the adobe "OA4" contains 1.00% of Na₂O. If we consider that all the sodium is in the form of albite (which is probably not true because these soils contain clay minerals and it is possible that a part of the sodium is present in some of these clays), this amount of sodium would correspond to 8.00% of albite (Si₃O₈AlNa) while the computer using the Rietveld's software finds 41.53% of albite! The same observations could be done with all the results of these two tables. It is possible that the Rietveld's method can strongly help for the quantification of the mineral phases contained in an earth material but the use of this tool requires significant know-how and skills in crystallography and mineralogy and it is therefore advisable to be very careful about its use.

There are examples in other articles where the researchers use robust methods of quantification based on the qualitative characterization by XRD coupled with a calculation using the chemical composition of the soils studied (Dao et al., 2018; Laborel-Préneron et al., 2017; Millogo et al., 2014; Millogo et al., 2016). A simple example of this calculation is given in the study of Millogo et al. (2016) on earth blocks stabilized by cow dung. The XRD spectrum of the soil used in this study showed that it only contained kaolinite (Al₂(Si₂O₅)(OH)₄), quartz (SiO₂) and goethite (FeO(OH)). By using the chemical composition of this soil, it is very easy to calculate the amount of kaolinite (by using the content of Al₂O₃), of quartz (content of SiO₂ corrected by the amount of SiO₂ contained in kaolinite) and of goethite (amount of Fe₂O₃). To make this calculation, it is nevertheless necessary to know the chemical formula of the minerals present in the soils and in particular the clay minerals. For the quoted studies, the calculations are relatively simple because the studies relate to soils which mainly contain kaolinite whose chemical formula is simple. However, in the case of soils containing other clay minerals such as illite, montmorillonite or chlorite for example, the chemical compositions of these minerals are complex and variable, which extremely complicates the calculation of the mineralogical composition (Deer et al., 2013).

In such complex cases of study, thermal gravimetric analysis (TGA) is a complementary tool that can help to improve the accuracy of the calculation or to check the correctness of the results obtained by the calculation. As an example, the differential thermal gravimetric analyses (DTGA) of the two soils studied by Ouedraogo et al. (2020) are presented on Figure 2-13.

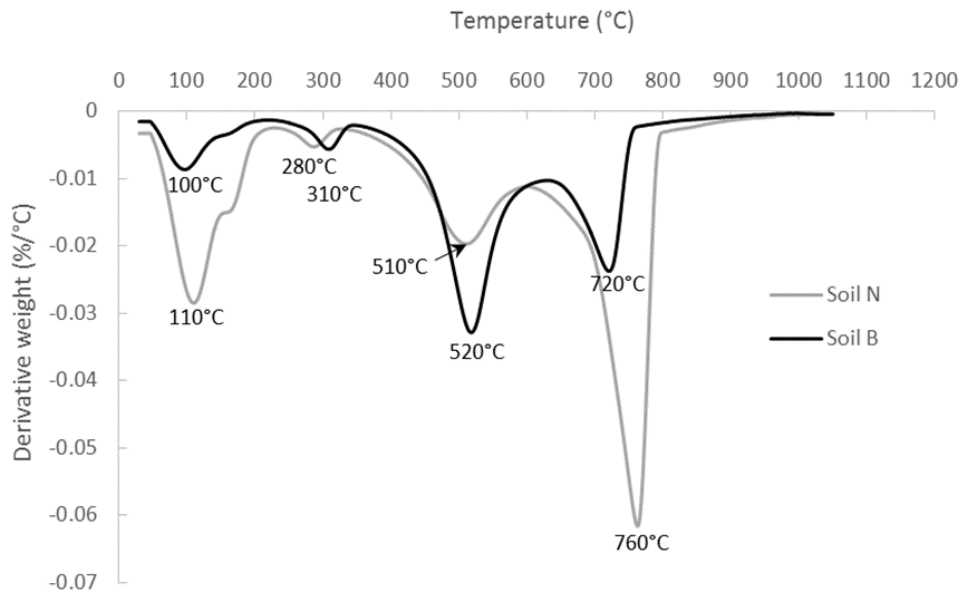


Figure 2-13. Differential thermal gravimetric analyses of the two soils studied by Ouedraogo et al. (2020)

The DTGA presented in Figure 2-13 shows the presence of the same four main peaks for the two soils even if their qualitative mineralogical compositions are different (especially the nature of clay minerals):

- 100-200°C - loss of hygroscopic water (water strongly linked to the material);
- 300°C - dehydroxylation of goethite (FeO(OH));
- 500-550°C - dehydroxylation of clay minerals;
- 700-800°C - decarbonation of calcite (CaCO₃).

Some differences of intensity between the peaks of the two soils exist and especially for the first peak, corresponding to hygroscopic water. This peak is much higher for soil N that is consistent with the nature of the clay minerals it contains (chlorite and essentially montmorillonite are able to “stock” a lot of water in opposition of kaolinite contained in soil B). Finally, the main interest of DTGA is the possibility to quantify the numbers of some minerals contained in the soils. In this case of study, it is possible to quantify calcite and goethite for the two soils: they are equal to 11.6% (respectively 1.3%) for soil N and 5.3% (respectively 2.2%) for soil B.

Finally, examples of the use of IR spectroscopy to characterize earth materials are rare. We can quote the studies of Dao et al. (2018) and Ouedraogo et al. (2020). In these studies, the analysis of the IR spectra carried out on the soils confirms the qualitative analyses obtained by XRD and TGA but it does not bring any new results. This technique is still underused for the moment but it is likely that it could be very useful to improve the understanding of the phenomena that occur during the chemical stabilization of earth materials.

2.7. Field tests

2.7.1 Procedures and standards

Several field-test procedures are used. They have been developed since long, based on the expertise of professionals. To allow comparison between qualitative tests, in some cases each sample is assigned a score based on the soils' performance. Some examples of score assignments for the qualitative tests are presented with the test itself. In other cases, a table is presented. They can provide information namely about particle size and shape, presence of organic compounds, clay, silt and sand content and adequability to a determined building technique.

Visual inspection of soils

Visual inspection of a soil can be carried out based in ASTM D2488 (2017) and other simple test procedures, evaluating more objectively properties such as colour, texture (angularity, shape of particles) and odour of a soil.

The visual particle size test: a thin layer of soil is placed on a surface and particles are pressed with fingers to be sure that clay granules are scrapped. The grain particles that are visible are the sand and gravel; the rest is clay and silt. If the volume of clay and silt is bigger than the rest, the soil is considered as not sandy (Neves et al., 2010). The observation of the sand and gravel allows to assess angularity.

The touch test: a small portion of dried soil is picked between two fingers. Sand is detected because the grains can be felt. If the touch is silky, there is clay or silt.

The wash test: a portion of soil is placed in the hand and washed with water. A content of fines is washed and coarser particles (namely sand) can be observed, allowing to assess their particle size and shape.

The exudation test: the test assesses the soil plasticity function of water retention. It consists in adding water to a soil sample, moulding and placing it in the palm of the hand. With the other hand, the sample is banged so that water gets out and the sample presents a shiny bright surface. Qualitative classification can be assessed by Table 2-13.

Table 2-13. Exudation test assessment (Neves et al., 2010)

Reaction	Number of bangs	Effect	Classification
Rapid	5-10	Water appear at the surface; it disappears with finger pression; higher pression disintegrates the sample	Low plasticity; fine inorganic sand or coarse silt; sandy or silty soil
Slow	20-30	Water appears and slowly disappears; finger pressure deforms the sample	Slightly plastic silt or clayey-silt soil
Very slow	>30	No significative change	High plasticity clayey soil

The colour and odour test: a portion of dry soil is observed: light and bright colours are characteristics of inorganic soils while dark colours are characteristics of organic ones. A strong smell when the soil is moistened is also characteristic of organic soil (Neves et al., 2010).

Ball and stick soil tests

The ball test: a portion of soil is moistened and moulded to form a ball. The content of clay is directly related with the easiness to mould a ball. The ball is dried and if the form is maintained, the soil can be considered apt to construction without need of stabilization (Neves et al., 2010). If it disintegrates, it has too low clay content and is not apt to earth construction.

The dropping ball test: a ball similar to the previous one is moulded with about 3 cm diameter and let fall from 1 m high. The disintegration of the ball is observed: for sandy soils, the ball disintegrates in small portions while for clayey soils the ball just deforms by the impact with the floor (NZS 4298, 1998; Neves et al., 2010). This test is considered adequate to evaluate the optimum moisture content for non-plastic earthen techniques, such as rammed earth or compressed earth blocks:

- if the ball disintegrates in small portions, the soil is too dry;
- if it breaks in 4-5 pieces, the moisture content is optimized;
- if it just deforms and stays in one portion, it is too wet (Jayasinghe and Kamaladasa, 2007).

The stick test: a sample of non-sandy soil is added water to form a paste that is rolled into a compact ball by hand, and stabbed with a knife. The amount of soil that clings to the knife when it is removed is observed. Soil sticking to the knife indicates high clay content. In their study, because they used artificial neural networks, Sitton et al. (2017) need values to compute statistics in the method they developed for rapid soil classification for use in constructing compressed earth blocks. The qualitative classification they proposed is the following:

- 1: soil is adhered to the blade when the knife is pulled out of the sample;
- 0: there are streaks of soil residues on the blade when the knife is pulled out;
- -1: there are no soil residues when the knife is pulled out of the sample.

The shine test: a sample of soil is added water to form a paste that is rolled into a compact ball hand sized, that is cut in half with a knife. The cross section of the ball is observed. A glossy/shine cross section indicates high clay content, a dull cross section indicates higher silt or sand content. As it was the case for the stick test, Sitton et al. (2017) have proposed a qualitative classification:

- 1: the ball cross-section appears glossy and reflects light: it is a clayey soil,
- 0: the cross-section appears somewhat glossy but is not very glossy and does not reflect much light: it is a silty soil,
- -1: the cross-section is not glossy and does not reflect light: the soil is sandy.

Cord and ribbon soil tests

The cord test: the test assesses soil cohesion and plasticity with a determined moisture content to classify the type of soil. A soil sample is moistened so that, moulding by hand on a flat surface, it is possible to form a soil cord that brakes when it has about 3 mm diameter. A ball is moulded immediately with that 3 mm cord and is pressed between thumb and forefinger (Neves et al., 2010). The force to achieve that and the classification obtained are presented in Table 2-14.

Table 2-14. Assessment of the cord test and soil classification (Neves et al., 2010)

Qualitative ball rupture	Soil classification
Hard to press and does not disintegrate	High clay content; high plasticity
Low resistance; cracks and disintegrate easily	Clay-silty, sandy or sandy-silty soil; medium plasticity
Brakes when pressed and cannot be moulded again	High silt or sand content and low clay content; low plasticity
Resilient when pressed	Organic soil, not adequate for construction

The ribbon or cigar test: This test is correlated to the plasticity of the soil (HB 195, 2001; ASTM E2392/E2392M-10, 2016). Water is mixed with a soil sample to form a paste, as for the cord test. The paste is rolled on a flat surface and moulded manually into a long cigarette shape (approximately 1 cm diameter and 8 cm long; if longer, it should be cut). The soil is classified with low plasticity if it breaks before it can reach the diameter of the cigarette. With the thumb and forefinger, the cylinder is pressed to form a stripe with 3-6 mm thickness and as long as possible (Neves et al., 2010). For that, part of the soil stripe is in the vertical position (draped over the side of the hand) while the rest of the cylinder is being pressed. If it can be rolled to the diameter of a cigarette and can support its own weight when draped over the side of the hand, the soil has high plasticity. The following qualitative classification is adapted from Neves et al. (2010) and Sitton et al. (2017):

- 2: the ribbon does not break when draped over the side of the hand up to 25-30 cm long
- high clay content and classified as high plasticity soil;
- 1: the ribbon does not break when draped over the side of the hand up to 5-10 cm long
- clay-silty or sand-clayey soil, classified as medium plasticity soil;
- 0: the cylinder holds together and can be rolled out; however, it breaks before it reaches approximately the diameter of the cigar or the ribbon breaks when draped over the side of the hand - silty or sandy soil, with low clay content, classified as low plasticity soil;
- -1: the cylinder does not hold together or cannot be rolled out without crumbling.

The pen or roll test: this test can be performed to assess if a clayey sample of soil is adequate for ramming. A paste of soil sample, already kneaded with water, is moulded on a plane surface with one rounded section to provide a 20 cm long soil cord with 2.5 cm diameter, similar to a pen. The soil pen is placed perpendicular to the round section of the plane surface and is slowly pulled forward so that the first centimetres of the soil pen get in the vertical position, while the rest is still horizontal, maintained by the hand, until a rupture occurs (Neves et al., 2010). The following qualitative classification is proposed by Neves et al. (2010):

- 1: too high clay content for ramming is obtained with a vertical segment with more than 12 cm;
- 0: a vertical segment between 8 cm and 12 cm indicate an ideal clay content for ramming;
- -1: the soil has not enough clay content for ramming if the vertical segment has less than 8 cm.

Sedimentation field tests

The tube particle gradation test: a dry soil sample is dispersed in water in a test tube marked “sand”. The sample will start to slowly settle out of suspension at the bottom of the test tube. After different time increments, the portion of the sample remaining in suspension is

transferred to a “silt” test tube and then to a “clay” test tube. Essentially, the test uses gravity to separate the soil sample into different groups based on particle size and can give an approximation of the soils’ particle gradation and fine content (Sitton et al., 2017).

The Jar test: based on HB 195 (2001), a soil sample is placed inside a glass jar with a flat base. Water is added up to 2/3 of the jar height and a low amount of sodium chloride can be added to act as deflocculant agent (Neves et al., 2010). The jar is capped, vigorously agitated, rested during 1 h and agitated again. Each soil component type, with different loose bulk density, settles out with different velocities. Therefore, once all of the samples have settled out, different strata can be observed and measured. If there are organic compounds, they will float at the surface. Nevertheless, sometimes it is difficult to locate and measure the strata between finer sands and coarser silts, finer silts and clays using this method. Furthermore, Silva et al. (2013) refers the need of considering that flocculated clays occupy a much larger volume than the same clay in deflocculated state.

Linear and volumetric shrinkage test

The shrinkage tests can be easily performed in situ and allow assessing the viability of an earth or/and earthen mix to a defined technique. An earthen material, mixed with a water content that is suitable for the building technique (only moistened and compressed if it is for rammed earth or compressed earth blocks, in plastic state and not as much compressed if it is for adobe or cob) fills a longitudinal mould and is let to dry.

The linear shrinkage test can be visually evaluated by the cracking and space between the sample and the moulds. It can be quantified by the perceptual metric difference of the sample to the mould. Some recommendations for the moulds’ dimensions, the moisture content, sampling and drying were gathered by Gomes et al. (2014) and presented in Table 2-15.

In some cases of earth construction, the volumetric shrinkage should also be considered. The same samples used to assess linear shrinkage can be used and the difference on samples high is also considered to evaluate volume change (Gomes et al., 2014).

Table 2-15 Recommendations and requirements for linear shrinkage test (adapted from Gomes et al. (2014))

Reference	Box dimensions	Water content	Material	Drying	Maximum shrinkage (%)
(Keable, 1996)	60x4x4 cm ³	Optimum moisture content	Same material than in the wall	3 days in the sun	2 ^a
(NZS 4298, 1998)	60x5x5 cm ³	Same moisture content than in the wall	Same material than in the wall	7 days with the sample covered by a plastic sheet. 21 days in the air out of direct sun light	0.05
(HB 195, 2001)	60x4x4 cm ³	Optimum moisture content	Size fractions 6.00 mm sample with 2-2.5 kg	3-7 days in the sun	2.5 ^b
(Keefe, 2005)	60x5x5 cm ³	Optimum moisture content	Same material than in the wall	Until complete drying	0.25
(Lehmbau Regeln, 2009)	60x5x5 cm ³	Not mentioned	Remove the coarse fraction (quantitative values are not specified)	Until complete drying	2

^a For higher shrinkage values, the reference recommends adding a certain percentage of cement or of low clay content soil (sand/aggregate).

^b For stabilized rammed earth with 4%-6% cement content; the document provides threshold values for cement contents from 4%-6% to 10%; the threshold value increases with the cement content.

The dry resistance tests

A soil sample is moistened and mixed to produce a planar specimen with 1 cm thick. Cylindrical samples with 3 cm diameter are cut from the specimen and let to dry. Each dry sample is firstly pressed by hand between the thumb and forefinger and, afterwards, if it has strength enough, it is broken using thumbs and forefingers of both hands (Houben and Guillaud, 2006). A qualitative classification was proposed by Neves et al. (2010) as presented in Table 2-16.

Table 2-16. Dry resistance test assessment and soil classification (adapted from Neves et al., 2010)

Dry resistance	Rupture	Behaviour	Classification
High	Resistant	Does not turn to dust	Inorganic high plasticity earth; clayey soil
Medium	Medium resistant	Pieces can be turn to dust	Clay-silty soil, clayey-sand soil or clayey sand; do not use if an organic clay
Low	Does not resist	Easily disintegrates	Lack of cohesion; sandy soil, inorganic silt or other soil type with low clay content

2.7.2 Study of data from literature

Silva et al. (2013) analysed four superficial soils collected in North Portugal. By visual field tests, the soils were characterized by colour: light tones such as grey and yellow. In terms of angularity, the soils present subangular particles and one also presents sub-rounded particles. The soil particles were not elongated nor flat in terms of shape, whereby researchers expected that destabilised rammed earth walls built with those soils were expected to have lower mechanical properties than those built with schist residual soils from southern Portugal. Odour was not identified which indicates absence of organic matter.

The same soils were characterized by the Jar test. Three of them present very low clay content and researchers suggest they are not suitable to destabilised earth construction, compared to recommended values from literature (Houben and Guillaud, 2006; HB 195, 2001). Only one tested soil present clay content within recommended values for rammed earth. When the soils were tested by the ribbon test, results agreed with the jar test: the clay content of the three soils was very low because it was not possible to make a cylinder with them. The fourth soil had ribbons with an average of 45 mm. Therefore, in accordance with HB 195 (2001) it was suitable for rammed earth or stabilized compressed earth blocks. For the drop ball test, moulding the ball was difficult for the three soils, confirming the low clay content determined by previous tests. For the dry strength test, samples of 4 cm diameter and 1 cm thick were made. The three soils presented low strength when compared with the fourth, evidencing the higher clay content of the latter. Therefore, based on field-test results, this soil was the only considered adequate for ramming.

Neves et al. (2010) recommend combined percentages of sand, silt and clay of soils to be used for earth blocks and rammed earth, based on the jar test results (Figure 2-14). Similar combinations, mostly presented by a triangular diagram, are presented by other researchers (Sitton et al., 2017).

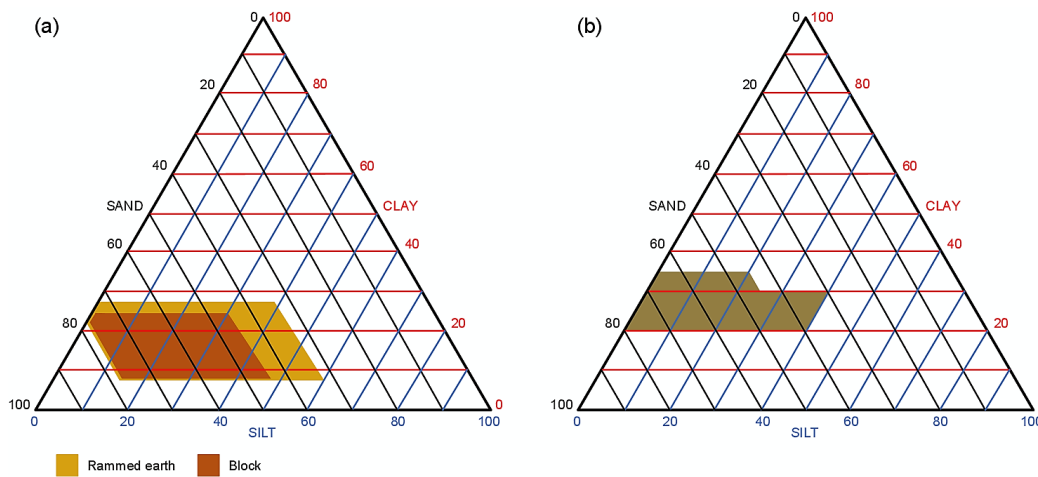


Figure 2-14. Combined percentages of sand, silt and clay of earth material for blocks and rammed earth: (a) for stabilization; (b) without need of stabilization (adapted from Neves et al., 2010)

Sitton et al. (2017) considers that the Jar test has proven to be most valuable when used to assess the expansiveness of a soil, that is generally linked to the type of clay. Therefore, the test can be an easily indirect way to assess the existence in significant contents of expansive clays, such as montmorillonite. A soil that expands significantly after the addition of water in the jar is expected to cause problems with shrinkage cracks if used to produce for instance CEBs. Sitton et al. (2017) consider that may be controlled by stabilization, although problematic.

Based on several field tests (cord, cigarette, exudation, dry resistance tests), Neves et al. (2010) presents a classification of the type of soil and the earth construction techniques that are more adequate (Table 2-17).

Table 2-17. Field-test-based classification of soils and more adequate earth building techniques (adapted from Neves et al., 2010)

Cord test	Cigarette and ribbon test	Exudation test	Dry resistance	Soil type	Building technique
Fragile cord and very low strength	Short cigarette and ribbon not produced	Fast reaction	Very low	Sandy, sand-silty, sand-clayey, silt-clayey	CEB, adobe, rammed earth
Fragile and soft cord	Short ribbon	Slow reaction	Low	Silty	Low content stabilized CEB, rammed earth, adobe
Soft cord	Medium ribbon	Very slow reaction	High	Clayey with gravel, clay-sandy, clay-silty	Stabilized rammed earth or CEB, adobe with fibres
Hard cord	Long ribbon	Without reaction	Very high	Clayey	Adobe with fibres

2.8. Conclusion

This first chapter of this book on earth construction materials focused on the characterization of raw materials. We have seen that there are many characterization techniques in many fields: physics, geotechnics, chemistry and mineralogy. Deep knowledge of characteristics of raw materials is essential for optimizing the performance of earth materials but also for improving understanding of the phenomena. We have seen that the most measured characteristic is the particle size distribution of the earth materials. This test is the most common certainly because it is the simplest to perform and also because it plays a significant role in the behaviour of earth materials for building technologies but it is not essential. Indeed, for all the properties that we will study in the following chapters (hygrothermal and acoustic properties - chapter 2, mechanical behaviour - chapters 3 and 4, and durability - chapter 5), the most important is to know the nature and proportion of the active phase (namely the clay minerals) of earth materials. This is often known indirectly by the measurement of the particle size distribution (which notably caused a confusion in the use of the term "clay") or by the measurement of geotechnical and chemical properties such as the Atterberg limits, the methylene blue value or the cation-exchange capacity. The objective of these techniques is to assess the reactivity of the clayey active phase, which depends on the type of clay and its proportion in the material. A thorough mineralogical characterization would allow measuring these parameters but, as we have seen, this characterization is complex and still relatively rare in the studies of the literature. The chemical composition is a necessary tool to correctly carry out this mineralogical characterization. Finally, we have seen that there is also an even more global scale of analysis: the field tests. Field tests, although only qualitative, have the advantages of being easily performed, without significant costs. Therefore, results are obtained within a short period of time, turning them very useful in the working site. Nevertheless, they should be performed and results analysed by experienced professionals because they are mainly gathered by comparison.

Although characterization tests of soils are numerous, this literature review noted that only a handful of tests are commonly used, and few studies present a complete characterization of soils. This is due to the cost of the tests (in time and money) but also to the need. In fact, a complete characterization is not always necessary depending on the objective of the study.

Based on a set of tests, the local earth mix can be optimized for a defined building technique or the building technique can be adjusted to the local earth. The influence of sieving to decrease the content on a particle size fraction of the earth (and increase in others), the effect of additions such as plant fibres or a low binder content, can be assessed so that the material is optimized for the construction. We have just proposed that the understanding and the optimization of the characteristics of earth materials requires knowledge of the nature and the proportions of the clayey active phase. It is important to note that this comment is only valid for destabilized earth materials. Indeed, the addition of plant aggregates or fibres or low binder content will completely modify the properties of earth materials. In these cases, the physico-chemical interactions will be numerous and complex and they will require further characterization.

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Appendix A Particle size distribution of earth construction materials

Table 2-18. Particle size distribution of earth used for compressed earth bricks

Reference	Origin	Stabilizer	<0.002mm	0.063mm-0.002mm	2mm-0.063mm	>2mm
			Clay (%)	Silt (%)	Sand (%)	Gravel (%)
(Ammari et al., 2017)	M (Morocco)	cement	8	12	52	28
	B (Burkina Faso)	cement	12	26	60	2
	A (Algeria)	cement	0	62	30	8
(Bruno et al., 2017)	France	none	16	43	40	0
(Donkor and Obonyo, 2015)	USA	none	12	1	87	0
(Dove et al., 2016)	U (UK)	alginate	31	45	24	
	V (UK)	alginate	27	44	29	
	W (UK)	alginate	16	61	23	
(Eko et al., 2012)	Cameroon	cement	44	10	46	0
(Galan-Marin et al., 2010)	UK	alginate, lignum	32	45	23	0
(Hakimi et al., 1996)	1 (France)	cement	18	36	34	12
	2 (France)	cement	20	32	33	15
(Hakimi et al., 1999)	TEO (Algeria)	cement	7	13	60	20
	TMA (Algeria)	cement	20	35	30	15
	TRM (Algeria)	cement	25	10	65	0
(Laborel-Préneron et al., 2017)	France	none	27	66	7	0
(Leitao et al., 2017)	Portugal	alkaline activation	4	14	60	22
(Mc Gregor et al., 2014)	UK	cement, lime, NaOH	18	23	59	0
(Mesbah et al., 1999)	France	none	12	52	36	0
(Muntohar, 2011)	Indonesia	lime, RHA	20	33	47	0
(Venkatarama Reddy and Gupta, 2005)	India	cement	9	18	73	0
(Venkatarama Reddy and Hubli, 2002)	BCS (India)	lime	36	20	36	8
	TB1 (India)	lime	44	32	24	0
	TB2 (India)	lime	33	15	52	0

	TB3 (India)	lime	42	41	17	0
(Zine-dine et al., 2000) et al.	Maroc	cement	11	21	46	22

Table 2-19. Particle size distribution of earth used for adobe

Reference	Origin	Stabilizer	<0.002 mm	0.063mm- 0.002mm	2mm- 0.063mm	>2mm
			Clay (%)	Silt (%)	Sand (%)	Gravel (%)
(Achenza and Fennu, 2005)	Italy	tomato, beetroot	0	45	47	8
(Ashour et al., 2015)	Np	cement, gypsum	29	63	5	3
(Aubert et al., 2015)	France	none	18	62	38	0
(Balkis, 2017)	Has (Turkey)	gypsum, lime	0	0	95	5
	Tas (Turkey)	gypsum, lime	0	0	88	12
(Binici et al., 2005)	Turkey	ciment, pozzolana, gypse, chaux	32	25	43	0
(Costi de Castrillo et al., 2017)	PrA1 (Cyprus)	none		64	25	10
	PrA2 (Cyprus)	none		46	42	12
	PrA3 (Cyprus)	none		66	26	9
	PrA4 (Cyprus)	none		67	31	2
	PrA5 (Cyprus)	none		57	35	8
	PrA6 (Cyprus)	none		46	22	32
	PrA8 (Cyprus)	none		56	25	19
	OA1 (Cyprus)	none		74	26	0
	OA2 (Cyprus)	none		72	24	5
	OA3 (Cyprus)	none		79	20	1
	OA4 (Cyprus)	none		79	21	0
	OA5 (Cyprus)	none		57	30	13
	OA6 (Cyprus)	none		76	24	0
	LyB (Cyprus)	none		91	8	1
	AthB (Cyprus)	none		61	26	13
(Dao et al., 2018)	Burkina Faso	cement	30	23	42	5
(Dhandhukia et al., 2013)	EB1 (India)	none		49	51	0
	EB2 (India)	none		47	53	0
	EB6 (India)	none		84	16	0
	EB7 (India)	none		62	35	3
	EB8 (India)	none		68	32	0
	EB9 (India)	none		63	36	1
	EB13 (India)	none		46	54	0

(Duarte et al., 2017)	CLG-1S (Angola)	none	30	25	16	29
	CTL-2S (Angola)	none	16	15	49	20
	CTL-2A (Angola)	none	16	13	67	4
	CHG-3SS (Angola)	none	30	10	60	0
	CHG-3SP (Angola)	none	56	9	33	2
	CHG-3AR (Angola)	none	33	11	56	0
	CHG-3AA (Angola)	none	26	12	62	0
	CGR-4S (Angola)	none	28	11	61	0
(Duarte et al., 2017)	CHJJ-1S (Angola)	none	5	6	89	0
	CHJJ-5CA (Angola)	none	51	17	32	0
	LMD-6S (Angola)	none	30	12	47	11
	MNG-7A (Angola)	none	42	22	36	0
	NDD-8A (Angola)	none	16	9	75	0
	NPK-9S (Angola)	none	36	19	45	0
(Fratini et al., 2011)	Brick 1 (Italy)	none	40	18	42	0
	Brick 2 (Italy)	none	16	13	71	0
	Brick 3 (Italy)	none	22	3	75	0
	Brick 4 (Italy)	none	20	13	67	0
	Brick 5 (Italy)	none	27	29	44	0
	Brick 7 (Italy)	none	25	23	52	0
	Local earth (Italy)	none	28	23	49	0
(Illampas et al., 2014)	Ly (Cyprus)	none	78–91		8–18	1–4
	Ath (Cyprus)	none	61–86		11–26	3–13
(Micoli et al., 2014)	np	none	12	45	43	0
(Millogo et al., 2014)	France	none	25	30	45	0
(Millogo et al., 2016)	Burkina Faso	cow dung	36	17	43	4
(Parisi et al., 2015)	Italy	none	10	18	64	8
(Piattoni et al., 2011)	Italy	none	22	50	25	3

(Quagliarini and Lenci, 2010)	Italy	none	29	48	13	10
(Turanli and Saritas, 2011)	np	fly ash	40	25	35	0
(Uguryol and Kulakoglu, 2013)	Turkey	none	27	33	37	2
(Wouatong et al., 2017)	MW4 (Cameroon)	none	16	49	33	2
	MW3 (Cameroon)	none	10	46	44	0
	MW2 (Cameroon)	none	8	41	49	2
	MW1 (Cameroon)	none	8	38	50	4
	HC3 (Cameroon)	none	21	38	36	5
	HC2 (Cameroon)	none	8	34	56	2
	HC1 (Cameroon)	none	16	44	38	2
	ME2 (Cameroon)	none	12	46	42	0
	BE1 (Cameroon)	none	12	48	40	0
	B (Cameroon)	none	25	26	28	21
(Wu et al., 2013)	China	none	89		11	0

Table 2-20. Particle size distribution of earth used for extruded earth bricks

Reference	Origin	Stabilizer	<0.002mm	0.063mm- 0.002mm	2mm- 0.063mm	>2mm
			Clay (%)	Silt (%)	Sand (%)	Gravel (%)
(Aubert and Gasc-Barbier, 2012)	France	none	36	28	36	0
(Cagnon et al., 2014)	1 (France)	none	30	28	42	0
	2 (France)	none	29	37	33	1
	3 (France)	none	23	31	43	3
	4 (France)	none	29	33	37	1
	5 (France)	none	38	27	35	0
(Fgaier et al., 2016)	A (France)	none	6	86	8	0
	B (France)	none	5	91	4	0
	C (France)	none	5	81	14	0
(Fouchal et al., 2015)	France	none	29	63	8	0
	France	none	54	32	14	0
(Maillard and Aubert, 2014)	B1 (France)	none	40	25	35	0
	B2 (France)	none	55	31	14	0
	B3 (France)	lime	31	54	15	0
	B4 (France)	none	58	40	2	0
	B5 (France)	none	48	42	10	0
(Maskell et al., 2014)	UK	cement or lime	16	46	33	5*

Table 2-21. Particle size distribution of earth used for cob

Reference	Origin	Stabilizer	<0.002mm	0.063mm-0.002mm	2mm-0.063mm	>2mm
			Clay (%)	Silt (%)	Sand (%)	Gravel (%)
(Coventry, 2004)	Crediton (UK)	none	3	2	48	42
	Tedburn (UK)	none	26	25	13	36
	Halstow (UK)	none	49	45	4	1
	Bridgnorth (UK)	none	3	5	67	27
(Harries et al., 1995)	Carboniferous (UK)	none	28	34	16	22
	Permian (UK)	none	18	42	38	2
(Miccoli et al., 2014)	"Cob" (Germany)	none	21	61	18	
(Quagliarini et al., 2010)	Original soil (Italy)	none	34	49	17	0
	Yellow Soil (Italy)	none	36	50.5	13.5	0

Table 2-22. Particle size distribution of earth used for rammed earth

Reference	Origin	Stabilizer	<0.002mm	0.063mm-0.002mm	2mm-0.063mm	>2mm
			Clay (%)	Silt (%)	Sand (%)	Gravel (%)
(Araki et al., 2016)	H2 (Japan)	MgCl ₂	38		56	6
	M (Japan)	CaO	28		70	2
(Arrigoni et al., 2017)	P (Australia)	none	20	66	14	0
	ELS (Australia)	cement, fly ash or calcium carbide	20	9	60	10
(Bui et al., 2009)	FRE (France)	none	16	49	35	
	MRE (France)	none	8	27	49	16
	MRES (France)	lime	8	27	49	16
(Bui et al., 2014a)	A (France)	none	5	30	49	16
	B (France)	none	4	35	59	2
	C (France)	none	9	38	50	3
	D (France)	none	10	30	12	48
	E (France)	none	10	22	43	25
(Bui et al., 2014b)	A (France)	none	10	25	18	47
	B (France)	none	5	30	49	16

	C (France)	none	8	34	8	50
(Ciancio et al., 2013)	1 (Australia)	none	5	25	50	20
	2 (Australia)	none	30	0	50	20
	3 (Australia)	none	15	15	50	20
	4 (Australia)	none	30	20	40	10
	5 (Australia)	none	40	20	20	20
	6 (Australia)	cement	10	15	50	25
	7 (Australia)	cement	10	5	40	45
	8 (Australia)	cement, lime	20	0	60	20
	9 (Australia)	cement, lime	30	10	20	40
	10 (Australia)	cement	5	25	50	20
(Cristelo et al., 2012)	Portugal	sodium silicate, sodium hydroxyde	6	14	46	34
(da Rocha et al., 2014)	BRS (Brazil)	lime, fly ash	5	33	62	0
(Gerard et al., 2015)	Belgium	none	13	64	26	
(Gomes et al., 2014)	Av (Portugal)	none	9	16	67	8
	PD (Portugal)	none	27	20	23	30
	VC (Portugal)	none	18	31	17	34
	CZ (Portugal)	none	10	18	47	25
	CI (Portugal)	none	13	24	29	34
	Ar (Portugal)	none	17	23	33	27
(Hall, 2007)	433 (UK)	cement	10	20	31	39
	613 (UK)	cement	10	20	46	24
	703 (UK)	cement	10	20	55	15
(Hamard et al., 2016)	CRA (France)	none	12	33	53	2
(Jayasinghe and Kamaladasa, 2007)	S (Sri Lanka)	cement	8		59	32
	HL (Sri Lanka)	cement	14		30	56
	C (Sri Lanka)	cement	19		30	51
(Miccoli et al., 2014)	Germany	none	11	25	64	
(Michiels et al., 2017)	USA	none	15	24	61	
(Serrano et al., 2013)	Spain	lime, alabaster	42	50		8

(Silva et al., 2013)	S1 (Portugal)	none	6	14	45	35
	S2 (Portugal)	none	5	15	59	21
	S3 (Portugal)	none	4	14	60	22
	S4 (Portugal)	none	12	12	53	23
(Tripura and Singh, 2015)	India	cement	8	13	79	
(Venkatarama Reddy and Prasanna Kumar, 2011)	S1 (India)	cement	32	18	50	
	S2 (India)	cement	21	14	65	
	S3 (India)	cement	16	12	73	
	S4 (India)	cement	13	10	77	
	S5 (India)	cement	9	9	82	

Appendix B Atterberg limits of earth construction materials

Table 2-23. Atterberg limits of earth used for compressed earth bricks

Reference	Origin	Stabilizer	w _l (%)	w _p (%)	Ip
(Bruno et al., 2017)	France	none	33.0	20.1	12.9
(Dove et al., 2016)	U (UK)	alginate	27.3	16.0	11.3
	V (UK)	alginate	27.9	14.6	13.3
	W (UK)	alginate	25.4	15.3	9.9
(Galan-Marín et al., 2010)	UK	alginate, lignum	34.8	19.1	15.7
(Hakimi et al., 1996)	1 (France)	cement	38	26	12
	2 (France)	cement	36	22	14
(Laborel-Préneron et al., 2017)	France	none	30	21	9
(Mesbah et al., 1999)	France	none	31	21	12
(Muntohar, 2011)	Indonesia	lime, RHA	41	25	16
(Venkatarama Reddy and Gupta, 2005)	India	cement	45	21	24
(Venkatarama Reddy and Hubli, 2002)	BCS (India)	lime	53	26	27
	TB1 (India)	lime	58	31	27
	TB2 (India)	lime	42	19	23
	TB3 (India)	lime	45	24	21
(Zine-dine et al., 2000)	Maroc	cement	32	18	14

Table 2-24. Atterberg limits of earth used for adobe

Reference	Origin	Stabilizer	w _l (%)	w _p (%)	Ip
(Achenza and Fenu, 2005)	Italy	tomato, beetroot	24	21	3
(Aubert et al., 2015)	France	none	23	20	3
(Balkis, 2017)	Has (Turkey)	gypsum, lime	23	13	10
	Tas (Turkey)	gypsum, lime	33	22	11
(Costi de Castrillo et al., 2017)	PrA2 (Cyprus)	none	27	17	10
	PrA4 (Cyprus)	none	28	22	6
	PrA5 (Cyprus)	none	28	22	5
	PrA6 (Cyprus)	none	41	23	18
	PrA8 (Cyprus)	none	38	21	17

	OA1 (Cyprus)	none	35	25	9
	OA2 (Cyprus)	none	33	19	14
	OA3 (Cyprus)	none	38	22	16
	OA4 (Cyprus)	none	40	25	14
	OA5 (Cyprus)	none	30	20	10
	OA6 (Cyprus)	none	39	25	14
	LyB (Cyprus)	none	46	25	21
	AthB (Cyprus)	none	37	24	13
(Dao et al., 2018)	Burkina Faso	cement	31	17	14
(Dhandhukia et al., 2013)	EB1 (India)	none	23	22	1
	EB2 (India)	none	23	22	1
	EB6 (India)	none	24	22	2
	EB7 (India)	none	25	22	3
	EB8 (India)	none	27	21	6
	EB9 (India)	none	26	22	4
	EB13 (India)	none	24	21	3
(Duarte et al., 2017)	CLG-1S (Angola)	none	31	18	13
	CTL-2S (Angola)	none	22	17	5
	CTL-2A (Angola)	none	23	18	5
	CHG-3SS (Angola)	none	29	18	11
	CHG-3SP (Angola)	none	61	37	24
	CHG-3AR (Angola)	none	23	16	7
	CHG-3AA (Angola)	none	29	16	13
	CGR-4S (Angola)	none	36	20	16
	CHJJ-5CA (Angola)	none	43	29	14
	LMD-6S (Angola)	none	29	19	10
	MNG-7A (Angola)	none	35	20	15
	NDD-8A (Angola)	none	22	15	7
	NPK-9S (Angola)	none	44	25	19
(Illampas et al., 2014)	Ly (Cyprus)	none	44	26.5	17.5
	Ath (Cyprus)	none	36	25	11
(Millogo et al., 2014)	France	none	38	20	18
(Piattoni et al., 2011)	Italy	none	26	18	8

(Turanli and Saritas, 2011)	np	fly ash	47	16	31
(Uguryol and Kulakoglu, 2013)	Turkey	none	22	32	10
(Wouatong et al., 2017)	MW4 (Cameroon)	none	61	52	9
	MW3 (Cameroon)	none	60	54	6
	MW2 (Cameroon)	none	48	47	1
	MW1 (Cameroon)	none	70	54	16
	HC3 (Cameroon)	none	63	46	17
	HC2 (Cameroon)	none	47	36	11
	HC1 (Cameroon)	none	70	48	22
	ME2 (Cameroon)	none	51	45	6
	BE1 (Cameroon)	none	55	44	11
B (Cameroon)	none	70	52	18	
(Wu et al., 2013)	China	none	36	19	17

Table 2-25. Atterberg limits of earth used for extruded earth bricks

Reference	Origin	Stabilizer	w _l (%)	w _p (%)	I _p
(Fgaier et al., 2016)	A (France)	none	100	28	72
	B (France)	none	60	29	31
	C (France)	none	24	21	3
(Maskell et al., 2014)	UK	cement or lime	24	16	8

Table 2-26. Atterberg limits of earth used for cob

Reference	Origin	Stabilizer	w _l (%)	w _p (%)	I _p
(Coventry, 2004)	Crediton (UK)	none	36.6 ± 0.6	22.2 ± 1.5	17.6 ± 6.2
	Tedburn (UK)	none	48.1 ± 4.5	27.2 ± 0.9	20.9 ± 3.9
	Halstow (UK)	none	69.6 ± 5.5	34.1 ± 1.6	35.5 ± 4.1
	Bridgnorth (UK)	none	21.1 ± 1.7	3.5 ± 7.8	17.6 ± 6.2
(Harries et al., 1995)	Carboniferous (UK)	none	43	27	15
	Permian (UK)	none	28	20	8

Table 2-27. Atterberg limits of earth used for rammed earth

Reference	Origin	Stabilizer	w _l (%)	w _p (%)	I _p
(Bui et al., 2009)	FRE (France)	none	27	19	8
(Ciancio et al., 2013)	1 (Australia)	none	16	13	3
	2 (Australia)	none	26	12	14
	3 (Australia)	none	18	8	10
	4 (Australia)	none	25	11	13
	5 (Australia)	none	35	16	18
	6 (Australia)	cement	15	10	5
	7 (Australia)	cement	17	9	8
	8 (Australia)	cement, lime	22	10	12
	9 (Australia)	cement, lime	39	15	23
	10 (Australia)	cement	15	11	4
(Gerard et al., 2015)	Belgium	none	32.5	15	17.5
(Gomes et al., 2014)	PD (Portugal)	none	41.2	25.1	16.1
	VC (Portugal)	none	46.1	26.7	19.4
	CI (Portugal)	none	35.5	22	13.5
	Ar (Portugal)	none	26	20	6
(Venkatarama Reddy and Prasanna Kumar, 2011)	S1 (India)	cement	40	19	21
	S2 (India)	cement	32	12	20
	S3 (India)	cement	27	9	18

Appendix C Optimum Proctor characteristics of earth construction materials

Table 2-28. Optimum moisture content and dry density of earth used for CEB

Reference	Origin	Stabilizer	w _{opt} (%)	ρ _{dopt} (kg/m ³)
(Donkor and Obonyo, 2015)	USA	none	9	1785
(Dove et al., 2016)	U (UK)	alginate	16	1820
	V (UK)	alginate	18	1980
	W (UK)	alginate	14	1920
(Hakimi et al., 1996)	1 (France)	cement	9.8	1930
	2 (France)	cement	11.6	1860
(Hakimi et al., 1999)	TEO (Algeria)	cement	12.2	1880
	TMA (Algeria)	cement	12.6	1760
	TRM (Algeria)	cement	12.0	1750
(Laborel-Préneron et al., 2017)	France	none	14	1988
(Leitao et al., 2017)	Portugal	alkaline activation	12.1	1710

Table 2-29. Optimum moisture content and dry density of earth used for rammed earth

Reference	Origin	Stabilizer	W _{opt} (%)	ρ _{dopt} (kg/m ³)
(Araki et al., 2016)	H2 (Japan)	MgCl ₂	10.3	1996
(Cristelo et al., 2012)	Portugal	sodium silicate, sodium hydroxyde	12.2	1958
(Gerard et al., 2015)	Belgium	none	15	1876
(Gomes et al., 2014)	Av (Portugal)	none	8	2018
	PD (Portugal)	none	17.8	1733
	VC (Portugal)	none	21.5	1651
	CZ (Portugal)	none	11.3	1600
	Cl (Portugal)	none	15.6	1814
	Ar (Portugal)	none	8	2018
(Silva et al., 2013)	S1 (Portugal)	none	12	1920
	S2 (Portugal)	none	12	1840
	S3 (Portugal)	none	12	1710
	S4 (Portugal)	none	10	2010
(Tripura and Singh, 2015)	India	cement	19	1710
(Venkatarama Reddy and Prasanna Kumar, 2011)	S1 (India)	cement	15.6	1835
	S2 (India)	cement	10.8	1910
	S3 (India)	cement	9.4	2000
	S4 (India)	cement	9.4	1980
	S5 (India)	cement	9.4	1950