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Article Strategy for the Mix Design of Building Earthen Materials Made of Quarry By-Products

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Abstract: The use of quarry by-products can enable the commercialization of a clay building material (reconstituted earth) thanks to minimal valorized and perennial stocks of materials. This study shows that quarry by-products can be used to mix design a clay-based building material for the manufacture of CEB. These soils are composed of quarry tailing and clayey muds. Proctor and dry compressive strength tests have shown that the proportion of mud that achieves the highest possible compressive strength is a balance between increasing density through the aggregate arrangement, increasing clay activity, and decreasing density through the increase in water content. These tests resulted in the formulation of materials with compressive strengths of 5.8 MPa and 8.4 MPa at densities of 2135 kg/m³ and 2178 kg/m³. The influence of mud incorporation on the material granulometry and on its characteristics was also studied. Moreover, a model allowing us to link the compressive strength, the clay activity, and the dry density is proposed for the materials composed of quarry by-products. This model enables us to facilitate the mix design and the standardization of the earth material.

Keywords: compress earth blocks; mix design; earth construction; quarry by-products



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1. Introduction

Raw earth construction has been used for thousands of years. There is no consensus about when humankind started to build with earth materials [1–3], but it could have begun with the start of early agricultural societies [1]. Today, this material is still used in many countries [4], but in most nations, this construction method has been replaced by concrete, and raw earth is now almost considered to be marginal [5].

The variability of earthen resources makes its democratization and its standardization complex [6–10]. The use of methods focusing on earth formulation and processing can thus be a solution to ease the earth formulation for construction purposes and to adapt this material. For instance, it could be possible to adapt some concrete formulation methods because earth construction can be compared to concrete where cement is substituted by clay [11]. Furthermore, it is possible to optimize the forming process. For example, compressed earth block (CEB), made by the compaction of soil at low moisture content, is a relatively recent development in the field of earthen building materials. This structural material can be considered a modern evolution of adobe, i.e., without the addition of vegetal fibers [1,12,13].

Nowadays, the building industry must tackle new issues; the scarcity of non-renewable resources and the reduction of materials' embodied energy are two of them [14,15]. Modern building techniques, especially reinforced cement concrete solutions, hardly respond to these issues, as cement production is responsible for an important part of global CO₂ emissions [16–18]. In this context, raw earth construction is regaining consideration due to its environmentally friendly character [19]. Moreover, its widespread nature is significant;

in some regions, most of the excavated earth could be used to create earthen building materials to supply the local industry [20]. Finally its hygrothermal properties allow for the regulation of the indoor atmosphere of a building [21].

For example, France presents an important density of quarries, with 3304 sites listed by the national office of quarries and geological questions (BRGM) in 2020 [22]. These quarries were developed to supply resources for the building industry, such as sand or gravel, to design concrete or road basements [23]. As a result, quarry by-products can be seen as a local material homogeneously spread over French territory. Available industrial machines and material volumes have also made it possible to produce a homogeneous stock of materials with constant properties (which can be an advantage over excavated earth).

During quarrying operations, the accumulation of quarry wastes could create ecological problems at the scale of a given quarry, such as fine particles' emissions in the ground and in water [24–26]. In 2006, the quantity of washing sludge produced in France was estimated at 88 million tonnes. Ref. [27] refers to a 2005 study stating that in India, the annual production of quarry co-products was 200 million tonnes. Developing the use of quarry wastes could lead to both environmental and economic benefits, saving space for exploitation and reducing waste production [24]. There are many ways to valorize quarry wastes (agriculture, waste neutralization, filler for paper and plastic, replacement of cement in concrete, ...), but they are still underused resources [24,28–32].

This paper investigates the potential usability of quarry muds as clayey and loamy fractions in a mix design of clay-based earthen material to produce manufactured CEB. The potential use of unusable secondary aggregates (quarry tailings) and quarry-crushed sands as the coarser fraction of this earthen mix is also investigated. The optimal ratio between quarry mud and coarser quarry waste or resources is studied for different materials to optimize the mix design of the earthen material and the optimal compaction level for each mix. Finally, a method by which to formulate an optimal earth mix from several quarry wastes is suggested by linking an inherent material property (clay activity) and a processing property (density) to aim for a final mechanical property (compressive strength). Therefore, the present study complements previous studies on the ability of different quarry by-products as earth material [33,34], providing a better understanding of their formulation.

2. Materials and Methods

2.1. Materials

There are two main types of quarries, namely, sand quarries and rock quarries, and they both produce different types of waste. During treatment, the material is crushed to produce aggregates that present specific shapes. Aggregates that do not fit these dimensions are considered to be a waste product called quarry tailings (QT) and are often left unused on the quarry site. Aggregates for concrete need to be washed to reduce their fraction of silt and clay; this washing of crushed rocks in quarries generates siliceous mud (SM), and the washing of sand in sand quarries makes clayey mud (CM). These two types of mud are generally left onsite and dry naturally.

Two different types of muds, CM and SM, are studied here to improve characteristics of crushed sand and quarry tailings. The SM is treated on site to maintain only its fine particles, presenting a maximum grain size of 20 μ m. The particle size distribution (PSD) of particles finer than 80 μ m was determined via laser diffraction for the 4 materials. The laser diffractometer Mastersizer 200 from Malvern was used according to the ISO 13320 standards [35]. For the sand and the quarry tailings, the PSD of the coarser fraction was determined by sieving according to the NF EN ISO 17892-4 standards [36]. Figure 1 displays a coarser granulometric distribution for the CM than for the SM. The plasticity limit and plasticity index, shown on the Table 1, is obtained according to the method proposed by Feng available in [37–39].



Figure 1. Particle size distribution of quarry muds, sand, and quarry tailings.

Materials	Clayey Mud (CM)	Siliceous Mud (SM)	Sand	Quarry Tailing (QT)
Plasticity limit	39.5	26.0	19.0	23.4
Plasticity index	23.8	13.8	6.3	11.8
Methylene blue value (MBV)	1.9	1.8	0.1	0.1

Table 1. Atterberg limits and MBV of quarry muds, sand, and quarry tailings.

The SM mud has a finer grain size than the CM mud. Its proportion of grains smaller than 2 μ m is 23.88%, whereas it is 9.39% in the case of CM mud (these proportions are defined via laser granulometry and are therefore underestimated). The VBS of the two materials are similar but the CM mud shows a higher plasticity index, suggesting a greater quantity of clay in the CM sludge. The SM mud would then be composed of more siliceous elements. This last point seems plausible because the SM mud comes from the rock-crushing process, whereas the CM mud comes from the sand-washing process.

Sand and quarry tailings (QT) present a low clay proportion. Their methylene blue value (MBV), fine particle proportions, and Atterberg limits are low. The main difference between these two materials is their PSD. Sand presents a PSD ranging from 0 to 4 mm, while for QT, it ranges from 0 to 16 mm. The analysis of fine mud formulations (CM and SM) with only sand or only QT could be relevant because the produced materials would be suitable for different purposes: sand could be used to create small blocks, and QT could be used to create elements with greater dimensions, such as structural blocks.

2.2. Methods

The aim of this study is to manufacture CEB presenting the highest achievable dry compressive strength. In this work, the key-parameters of CEB mix design and process are mix moisture content, mud/coarser resource ratio, compaction pressure to reach the final dry density, and compressive strength.

The understanding of earth-based materials' behavior, such as that of natural soils, under dynamic compaction is typically assessed with a Proctor test in roadworks [40,41]. This method consists of compacting earth dynamically at different moisture contents in a mold with standardized dimensions. The variation of compaction energy and/or the compacted volume leads to the use of different standards [42–45]. The Proctor test is used to find the optimum moisture content leading to the maximum dry density for a

given compaction energy. This optimal water content is a balance leading to the densest packing; that is, sufficient water to lubricate contact, but not so much as to limit its volume. The material's behavior during the test depends on its nature. Clayey material typically presents a low optimum density for a high optimum moisture content [46,47]. The increase in compaction energy reduces the optimum moisture content and increases the optimum dry density.

Proctor tests compact layers by applying a series of impacts, which differs from static compaction where a constant load is applied continuously [48–52]. The heterogeneity of statically compacted soil during CEB fabrication and compacted dynamically during a Proctor test are different. During a Proctor test, layers of soil are compacted, and the material could be locally saturated. For statically compacted samples, process-induced heterogeneity results in a density gradient parallel to the load direction. Moreover, during a static compaction test, the interstitial pressure could increase, caused by the inability of the material to evacuate water [53]. However, a proctor test can still be relevant because of its convenience with respect to implementation under standardized conditions.

In this study, a modified Proctor test is carried out. The test realized was conducted in accordance with the standards NF P 94-093 [44]. Five measurements were performed with 1% increments between moisture contents for water content ranging from 5% to 15%. The produced samples were used to determine the moisture contents. Different formulations were studied:

- Sand;
- QT;
- CM and sand (5 formulations);
- SM and sand (5 formulations);
- CM and QT (5 formulations).

For the formulations with different mud additions, the mud contents are distributed between 10% and 50%, with an interval of 10% between each formulation.

The samples were also used to realize a dry compressive strength test. The drying protocol consists of drying in the open air for a minimum of 24 h to avoid a large gradient in water content which could lead to cracking. After this period, drying is performed in a ventilated oven until mass stabilization. It is considered that the dry state was reached when the mass variation of a sample was less than 0.1% between 2 measurements taken as 48 h. The compression test was performed at a loading rate of 6mm/min to follow the standards NF EN 13286-41 [54]. The inner dimensions of the Proctor mold are 101.5 mm in diameter and 116.5 mm in height. The samples present a 1:1 slenderness.

3. Results and Discussion

An optimum density and moisture content was obtained for each formulation studied. The observed compaction behavior is similar compared to other results described in the literature [46,55–57]. The evolution of dry density according to water content follows a parabolic curve that presents optimum values.

The sand presents an optimum Proctor dry density of 22.20 kN/m³. A 10% CM incorporation in the sand increases the optimum dry density to 22.30 kN/m³ and reduces the optimum moisture content to 0.6%, as is displayed in Figure 2. However, at more than 10%, the optimum dry density decreases and the optimum moisture content increases. The types of mud and aggregate used influence the behavior of the optimum's values. The 10% SM incorporation in sand increases the optimum density from 22.20 to 22.80 kN/m³, but a higher proportion of SM reduces density; however, it remains higher than the optimum density of the initial sand formulation until an SM proportion of 30%, as is shown in Figure 2. The loss of density with the increase in mud proportion is higher for the formulations with CM than with SM. This could stem from its higher clay activity, which generates an increase in water demand to reach a given consistency (higher specific surface) [58–60].



Figure 2. Proctor results for formulations with SM and sand.

The CM addition in quarry tailings does not permit the material to reach a higher density, as displayed in Figure 3. Without CM, the optimal dry density is 22.70 kN/m^3 , and it decreases to 22.50 kN/m^3 with 10% of CM. The optimum moisture content increases, respectively, to 6.1% and 6.2%.



Figure 3. Proctor results for formulations with CM and sand.

A 10% mud incorporation has a slight influence on the optimal Proctor characteristics. For a mud variation of 10%, the maximum variation of optimum dry density is 0.84 kN/m^3 for all the formulations studied. The maximum variation of moisture content is 1.3% for an average of 0.7%.

It is assumed that an increase in density for a low content of mud comes from a better aggregate arrangement (the filler effect). This effect consists of incorporating fine elements in a coarser granulometry to fill the voids. This effect is also used in the mix design of concrete-containing quarry mud [61–64]. The size of mud particles is small enough to fill the voids in the material. This effect is visible in Figure 4, which highlights the impact of mud proportion on the void ratio of the formulated materials. This ratio decreases from 0.237 to 0.234 for a 20% CM incorporation, and from 0.237 to 0.211 for the formulation

of sand with 20% of SM. The void ratio of the formulations with SM is lower than the formulation with only sand until the mud proportions reach 30% for CM and 40% for SM. However, for a mud proportion higher than 10%, the obtained dry density starts to decrease both of the studied formulations. This behavior could stem from the increase in the clayey activity in the material, which implies a higher water demand to lubricate soil particles in order to reach a given workability [65,66]. For mud contents higher than 10%, the addition of SM implies a lower void ratio increase than the addition of CM. It is also assumed to be correlated to the higher clay activity of CM, which implies a stronger increase in the water demand as the mud proportion is increasing. This void ratio reduction can also be attributed to the resulting PSD of the mix; the wider the PSD, the denser the particles assembly can be.



Figure 4. Influence of the mud proportion on the void ratio at the Proctor optimal water content of the formulation with quarry muds and sand.

Indeed, it is usual in granular materials such as concrete to optimize the particle packing to improve the characteristics of the material [67,68]. The uniformity coefficient (or Hazen's coefficient) (Cu) and the curvature coefficient (Cc) are sometimes used to assess the particle packing, among other things, for the study of hydraulic conductivity through granular materials [69–72].

The Cu factor reflects the particle size distribution. A high Cu indicates a wide PSD, which in this case comes from a high fine particles content. A particle size distribution can be considered uniform when the Cu is less than 3, and it can be considered wide or varied when the Cu is greater than 3. The Cc reflects the variety of grain size. Figure 5 displays significant Cu values for mud contents ranging from 0% to 60%. Thus, sand particle size is already spread, and the addition of quarry mud will optimize the particle packing.

The Cu accuracy can be discussed. The diameter at 10% cumulative undersized particles mass is determined using the laser diffraction method. This is also the case for the 60% undersized particle mass diameter for formulations with mud proportions of 60% or more. This particle size analysis method underestimates the fines content [73,74], which influences the Cu and Cc values. In this study, as we use the Cu and Cs value for the sake of comparison, determining the fines particles' exact size is not crucial.



Figure 5. Influence of the mud proportion on the Cu of the formulation with quarry mud and sand.

In the same way as the Hazen coefficient, the D90/D10 factor can be used to assess the uniformity of the particle size distribution [75]. This factor represents the ratio of the 90% cumulative passing diameter to the 10% cumulative passing diameter. This factor is not suitable for the study of these materials because the D10 value is too low compared to D90 value for the uncertainties; due to the measurement of D10, it can be considered negligible. Moreover, the addition of mud causes a discontinuity in the particle size distribution, as can be seen in Figures 6 and 7.



Figure 6. Particle size distribution of mix design with sand and SM.



Figure 7. Particle size distribution of mix design with sand and CM.

Figure 8 shows optimal density evolution of formulations with quarry mud and sand according to the Cu. The maximum dry density of formulations with CMs is reached for a Cu value of about 210 for a mud proportion of about 30%. In the case of formulations with SM, the value associated with the Cu optimum seems to be 480 for a mud proportion of about 30%. The Cu optimum does not correspond to the optimal density because it decreases when the coefficient increases for the two muds studied. Furthermore, their incorporation increases the Cu, which shows an improvement in the granular packing but not in the density. The optimization of the granular skeleton does not seem to be the only parameter influencing the dry density. Figure 9 shows that the optimum Cu of formulations with CM addition correlate with the optimum compressive strength. However, this correlation between Cu and compressive strength cannot be shown for formulations with SM. The optimization of the Cu, and thus of the granular packing, does not seem to be the only parameter influencing the compressive strength.



Figure 8. Evolution of the dry density according to Cu.



Figure 9. Evolution of the compressive strength according to Cu.

To conclude, to improve the density of a given aggregate, the added mud needs to be as fine as possible in order to benefit from a better filler effect [76,77]. It also needs to present a low plasticity index (low clayey activity) to limit the water demand with the increase in the mud proportion [78,79]. However, the increase in density is not the only parameter that should be optimized, as an increase in the clay activity with the mud addition would probably lead to an increase in the compressive strength of the formulated material [80–82]. The maximal compressive strength of a given formulation is reached for the sample with the highest density. This observation is in agreement with the literature, which already correlates the dry density with the compressive strength [80,83–86].

The mud addition in QT mix design does not increase the density, as is shown in Figure 10. The mud granulometry is probably too fine compared to the QT dimensions to improve the granular packing. The increase in clay activity is not sufficient to compensate for the decrease in density, which results in a stabilization in compressive strength in a range of 20.00 kN/m^3 . A slight increase in compressive strength of 0.3 MPa can still be observed in Figure 10 for a 10% addition of CM.

The compressive strength depends on the type of mud used in the formulation, considering that different muds could present various mineralogical and granulometric properties. The incorporation of CM in sand leads to an optimum mud proportion of 30% and allows access to an optimum compressive strength of 5.77 MPa, as shown in Figure 10. The use of SM in formulations with sand permits an increase in the material's compressive strength until a proportion of mud of 50%, which leads to a final compressive strength of 8.44 MPa. The maximal proportion of mud that was studied in this experimental campaign was 50%. Hypothetically, the compressive strength could still increase with a higher mud content. As expected, this result is not in accordance with the optimum density results, and the mud content necessary to reach the optimum compressive strength is higher than is necessary to reach the optimum dry density. Thus, the density is not the only parameter influencing the compressive strength; the clayey activity also needs to be considered. To formulate earthen material using mud and a coarser aggregate, a compromise needs to be found between the increase in dry density and the increase in clayey activity in order to reach the higher compressive strength value.

The incorporation of a small proportion of quarry mud in an aggregate material increases its density via the filler effect and also increases its compressive strength via filler effect and due to the mud clayey activity. When the mud proportion exceeds a specific limit (depending on the clay activity of the mud and the PSD), dry density starts to decrease because of both the water demand necessary to lubricate particles so as to reach a given consistency and the decrease in the PSD wideness when the final compressive strength continues to increase by means of the clayey activity modification. Beyond another limit,

the maximum compressive strength of the designed material is reached, and an increase in the mud proportion leads to a decrease in the density and a decrease in the compressive strength [87–90].



Figure 10. Evolution of optimal characteristics of formulations with quarry muds and sand.

In concrete technology, models can be used to predict mechanical characteristics, such as the one developed by Feret or Bolomey [91–95]. An analogy between these methods could be used for earthen materials, wherein the dependence on the water/cement ratio could be replaced by the dry density (porosity description) of the designed material, and the cement activity could be replaced by the clayey activity. Therefore, for the development of this model, the MBV of each formulation has to be estimated accurately. The MBV of an aggregate with a quarry mud addition could be estimated via a simple proportionality law:

$$MBV = MBV_{auarry\ mud} \times mud\ content + MBV_{aggregate} \times aggregate\ content$$

Figure 11 highlights the ability of the previous equation to predict the MBV of a formulation. In accordance with previous results, the quantity of added quarry mud necessary to reach a given compressive strength could be estimated by using the model presented in Figure 12, with both the mud MBV and the aggregate MBV.



Figure 11. MBV measured experimentally and with a law of mixture.

The dry density could also be compared to a process-related parameter since it is partly controlled by the compaction pressure, compaction rate, compaction conditions (static or dynamic), and targeted water content for a given compaction method [65,96,97]. The MBV could be used to quantify the clayey activity of a material [98–101]. These two parameters are linked to the compressive strength in Figure 12 and allow us to ascertain a general behavior, as proposed in [102]. A master curve linking the ratio of compressive strength to the MBV and the dry density appears and demonstrates the link between the parameters. Two other clayey muds were added to sand and tested to verify this behavior (CM2 and CM3). The obtained results confirmed this trend, and it appears that a predictive model could thus be established to link the dry compressive strength of formulated earthen materials with their clayey activity and dry density. Moreover, statically compacted samples with 1:1 slenderness and formulated with 30% of CM and 70% of sand were tested. The obtained results also seemed to be in accordance with this behavior. The compaction method, the density, the MBV, and the compressive strength measurements bring several uncertainties that could create a small discrepancy between the actual strength of a building material and its estimated strength using this predictive model. However, the load-bearing potential of a raw earth material in construction could be estimated with this predictive method with quite good accuracy. It is important to keep in mind that clay activity is measured here with the MBV that refers to a dosage of the surface of the clay particles. This "surface" evaluation can be link to the size of the particles that interact with water, both on the pore size and at the end on the capillary suction, which is responsible for the dry compressive strength of the earth [103–106]. This is the reason why the MBV value was chosen as an indicator of the clayey activity that influences compressive strength at a dry state.



Figure 12. Link between material, confection, and mechanical characteristics.

4. Conclusions

The results of Proctor tests indicate the possibility to reconstitute an earthen material with quarry by-products via the addition of quarry mud in sand or quarry tailings. The influence of mud on the material properties is driven by the mud characteristics. The mud used in this study induces a filler effect when added to sand and QT. Mud incorporation increased the optimum moisture content, and sometimes reduced the dry density, despite the filler effect, due to the increase in clay activity and the decrease in the wideness of the PSD. This phenomenon is significant for the mud with the lowest plasticity index. Incorporating 10% siliceous mud into the sand matrix increases its optimum density by 35 kg/m^3 and its MBV by 100% (0.1 to 0.2). Moreover, the addition of mud has increased

clayey activity and compressive strength. The sand and quarry mud formulations achieved compressive strengths of 5.8 MPa and 8.4 MPa on scale 1 samples. To formulate an earthen material with quarry mud addition, a balance needs to be made between the increase in the dry density and the increase in the clayey activity to reach the best compressive strength.

Democratizing earth construction is difficult due to its variability among other things. A solution could be found by using a single and universal model between the clayey activity (MBV), a processing characteristic (dry density), and a mechanical characteristic (compressive strength). It can facilitate the formulation of earthen materials by estimating the necessary dry density of a material with a known MBV to reach a targeted compressive strength. The simplicity of determining the MBV of a material makes this method relevant and easily achievable. Another use of this model can be the estimation of the contribution made by mud (and thus in clay activity) to reach a sufficiently strong earth material. Moreover, a formulation method of earthen materials can help its democratization and standardization.

In this perspective, the law linking compressive strength, MBV, and density needs to be evaluated with other types of clay and with natural earthen materials. It has been shown that the optimal amount of quarry mud necessary to reach an optimal compressive strength value can be high (greater than 50%). For these formulations, it will be necessary to evaluate new parameters at the block scale, such as drying shrinkage or cracking.

Moreover, there are still many studies to be performed in order to fully assess the model effectiveness:

- Effect of compaction energies and vertical loads to assess the model's effectiveness over higher density ranges;
- Effect of the type of clay because it can influence the model; the MBV is influenced, in particular, by the swelling capacity of some clays (montmorillonite, smectite, etc.);
- Assessing the model's suitability with respect to other earthen construction methods;
- Assessing the limits of this model and its accuracy, especially for low MBV values which require high measurement precision;
- The effect of stabilization with hydraulic or bio-based binders generally increases compressive strength. It may be possible to establish different evolution curves depending on the type of binder and its proportion. With the same objective in mind, the addition of fibers could be studied.

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