



Article Physicochemical, Mineralogical and Microscopic Evaluation of Sustainable Bricks Manufactured with Construction Wastes

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Abstract: At an international level, enormous volumes of construction and demolition wastes are generated: 170 million tons/year in the USA, 500 million tons/year in the European Union (EU) and 12 million tons/year in Mexico. Alternative uses for these heterogeneous materials, such as the manufacture of sustainable bricks, are potential solutions to this growing environmental issue. Based on previous studies, and in compliance with Mexican standards, four different types of secondary materials were utilized in the composition of a sustainable brick matrix. Temperature and solar radiation used for drying purposes were determined, as well as weight loss, resistance and initial maximum absorption. In order to characterize the resulting matrix, observations were made with a scanning electron microscope, and the chemical composition of the samples was determined by detecting basic compounds using mapping through SEM-EDS microanalysis, connected to the SEM unit. Finally, thermogravimetric analyses were performed to correlate mechanical and chemical behavior, and resistance to high temperatures of the mixtures. The results obtained showed that all-in-one (AiO) is the most appropriate material for brick manufacturing, *Opuntia ficus-indica* mucilage improves physical properties, such as increased compressive strength and reduced water absorption, while wood residues, clay minerals and illite enhance mechanical properties.

Keywords: bricks; construction waste; mineralogical; physical properties; thermogravimetric analyses

1. Introduction

Bricks, especially the various types of conventional ceramic bricks [1], are the elements most commonly used in the construction of exterior and interior walls [2–4], and the demand for them in the building industry is expected to grow considerably leading to large manufacturing increases with the corresponding expansion of virgin raw materials extraction [5]. Their production requires the baking of virgin clay in an oven at high temperature (of the order of 1000 °C), affecting the environment in two ways: the use of non-renewable materials and the release of a significant amount of greenhouse gases—averaging 0.41 kg of carbon dioxide (CO₂) per brick [6,7].

Therefore, the search for alternatives that can reduce the environmental impact of the ceramic brick industry, such as the use of recycled second generation [8] materials from construction and demolition (CD) and processes that do not require baking, is a priority. The so-called second generation materials, particularly CD in view of the enormous quantities that are generated, represent a sustainable source of materials that is ready to be used. Staggering CD figures are reported in the United States

(170 million tons/year [2,9]), the European Union (500 million tons/year [3]) and Mexico (12 million tons/year [10]). Taking into account the population [11], the global figures corresponding to the three countries mentioned above [2] are equivalent to 0.53, 0.98 and 0.09 ton/year per capita, respectively, (these figures must be taken with caution in countries lacking legislation, or where uncontrolled landfill is common practice).

The incorporation of CD recycled aggregates (up to 25% or 50% maximum contents), together with Ordinary Portland Cement (OPC) or Fly Ash (FA), does not significantly affect the compressive strength of concrete bricks; and even bricks manufactured exclusively with replacement materials (100% CD aggregates) show resistance values of up to 49 at 28 days [12]. In a similar study, CD wastes, wood residues, and water with a cactus pulp extract (*Opuntia ficus-indica*) were used to manufacture an unbaked clay brick having compressive strength and water absorption characteristics appropriate for use as a non-structural adobe [13]. Finally, the use of clays and stabilizing lime in the manufacture of unbaked bricks has also been investigated, showing that these materials can be energy and cost efficient (no baking requirement) [14].

As to the use of second generation materials from other industries for the production of bricks, previous studies have focused on the use of FA [5] in an 84:6:10 (residue:FA:clay) ratio and 12.5 to 15% water; [15], with FA making up, in some cases, up to 40% [16] or even more [17]. In the literature, abundant itemized information regarding various properties (linear shrinkage, apparent density, apparent porosity, water absorption, compressive strength and flexural strength) of bricks manufactured with FA, is available [18]. On the other hand, the use of additives has also been studied with encouraging results [19]. The manufacturing processes usually include a phase of pressure compaction—of the order of 20–25 MPa [14]—and subsequent baking in oven at temperatures between 980 and 1050 °C [14,20]. Compressive strength [14,16] up to 12.4 MPa [16] or even 40 MPa [19]; water absorption [14,19,21] of the order of 13.8% [16]; apparent [22] density [14,23] up to 28% below reference bricks [19], porosity, and fissures and cracks expansion [16] have been determined. In all cases, the properties studied have been considered acceptable [14,16,19].

Blast-Furnace Slag (BFS), as well as kaolin, and granite-basalt residues [24], have also been used as a replacement clay to manufacture baked bricks [25]. Physical, mechanical, leaching [25,26] and chemical [24] tests have been conducted, concluding that heavy metal concentrations (determined by leaching) are acceptable, establishing a direct relationship between the increase in BFS content and the compressive strength; and an inverse relationship as regards absorption [25]. Similarly, it was discovered that the ratios of the different components making up the matrix of the bricks have a relevant impact on the various properties studied [21]. Finally, it was evidenced that a baking temperature ranging from 1000 to 1250 °C was appropriate to obtain bricks of acceptable quality [24,25].

Similarly, soil and sand (SS) of petroleum effluents mixed with stone materials have been used to make baked bricks. In a study [27], the results of the physical, chemical and mechanical tests led to the conclusion that the use of SS in brick manufacturing reduces water and fuel requirements, generating a product that complies with all the regulations and "encapsulates" toxic metals through vitrification (leaching studies validate compliance with the requirements of the US Environmental Protection Agency).

Some special cases have also been studied, such as the use of a mixture of organic materials, for example sawdust (5%), oil polluted soils (15%), compost (10%) and marble (15%) in the manufacture of light bricks baked at 1050 °C, that achieved compressive strength, porosity and absorption appropriate for use in construction [28,29].

Likewise, the use of paper waste as an additive in the manufacture of porous and lightweight clay bricks has led to the obtainment of bricks showing adequate thermal conductivity and compressive strength [28,29].

Ceramic bricks and tiles manufactured using quarrying and mining materials, for example granite rock cutting residues, have also been studied. Their physical and mineralogical characteristics can be considered comparable to those manufactured with conventional materials, and can therefore

partially replace them [28]. In another study, tailings from gold mills mixed with red soils were used to make bricks. The different replacement percentages suitable to obtain compressive strength, water absorption and shrinkage equivalent to the reference bricks were determined [30].

Dredging sediments have also been used as a base material for baked bricks (replacing the usual clay) and, in this case also, the ASTM criteria were met [22,31].

Finally, residues that could be considered inappropriate for use in construction, such as cigarette butts, have also been investigated as materials to be potentially incorporated in baked clay bricks. The density, strength, thermal conductivity and leaching values of the bricks obtained with this material have demonstrated that this material can also be considered suitable for use in the manufacture of light bricks [32].

Therefore, it is clear that the search for new alternatives of secondary materials to replace the usual non-renewable materials for making bricks or blocks is desirable, feasible, and even necessary, with CD wastes being apparently the most suitable option because of their intrinsic properties and widespread availability. Likewise, the incorporation of organic renewable materials, such as wood or other vegetable wastes, could also be a further suitable source of complementary materials. Moreover, if in addition to using the combination of the above materials, baking in oven could be omitted, then the brick manufacturing industry would drastically reduce its environmental footprint. Therefore, an experimental study for the manufacture of a sustainable brick combining the above mentioned elements has been proposed in this research in order to technically and environmentally validate its application in the construction industry.

2. Materials and Methods

2.1. Materials

Four different types of secondary materials were used in the composition of the sustainable brick matrix, resulting in a product that fulfills the regulatory requirements and possesses characteristics comparable to those of a traditional brick.

The materials used were:

- Excavation wastes obtained from a company that manufactures adobe bricks (19°05′30.7″ N 98°19′39.9″ W); non-standardized field tests determined that it was a hard plastic clay-sandy material appropriate for being used in the matrix (Figure 1a).
- 2. Two types of construction and demolition wastes obtained from a CD processing plant (19°19′12.3″ N 99°03′16.2″ W). On the one hand, CD with crushed cementitious materials from only concrete (OC)—classified as type A according to NADF-007-RNAT-2013 [33], with particle size (PS) of 9.5 mm and 6.3 mm to fines (Figure 1b). On the other hand, construction wastes known as all-in-one (AiO), classified as type B materials, and originating from an uncontrolled mixture of bricks, blocks, ceramics, mortars, paving stones, masonry and prefabricated materials, with a particle size ranging from 6.3 mm to fines (Figure 1c).
- 3. Sawdust wastes obtained from a composting plant ($19^{\circ}18'39.2''$ N $99^{\circ}10'37.5''$ W), including residues from cutting and pruning trees, branches and shrubs from the surrounding zone. The only requirement was PS ≤ 5 mm, achieved through mechanical sieving, in order to facilitate a correct mixture with the other materials (Figure 1d).
- 4. A vegetal liquid mix of gelatinous viscous aspect was processed using water and mucilage from nopal plants (*Opuntia ficus-indica*). The mucilage was produced by removing the epidermis from each tuber, and then the section of its central part (mesophyll) was cut in cubes with side length 2 cm that were introduced in water in a 1:3 (kg of nopal:liters of water) ratio for three days. The substance was then drained using a colander, and filtered applying manual pressure with a cloth to obtain the final substance (Figure 1e).



Figure 1. Materials (**a**) Excavation wastes; (**b**) only concrete (OC); (**c**) all-in-one (AiO); (**d**) Wood waste; (**e**) Mucilage and Water.

2.2. Methods and Experimentation Process

2.2.1. Mixtures Design

Two dosages and the use of water or water plus mucilage, and the differential use of OC or AiO in the 6.3 mm size fraction, were investigated. The rationale for the election of these variables originated in a previous study that determined that the use of mucilage influenced the resistance of the bricks [13]; although OC and AiO could be considered as "equivalent" materials, AiO is produced in a higher volume and, contrary to OC (recycled concrete with CD aggregates), it has no application in solutions, and thus this technique would turn AiO into a useful material. The nomenclature used for the mixtures was: X-XX-X, in which the first X refers to control trials or referent (R), or to the study variables (V); XX can be OC when a PS 6.3 mm OC is used, or AiO when a PS 6.3 mm AiO is used; and the last X refers to the use of water (W) or mucilage (M). The suitable percentages of each material used, obtained as a result of the dosage experience of the mixtures studied, are presented in Table 1. Approximately 12 kg of each study mixture were manufactured to make the test bricks. The substitution of different materials (OC and AiO) was carried out by weight, since their volumetric weights are close. The specific quantities of each material used correspond to values resulting from a previous study of mixture optimization [13].

Mixture	Excavation Wastes (%)	Sawdust Wastes (%)	PS 6.3 mm OC (%)	PS 6.3 mm AiO (%)	PS 9.5 mm OC (%)
R-OC-W	62	4	17	-	17
V-OC-M			17	-	
R-AiO-W			-	17	
V-AiO-M			-	17	

R-OC-W: Reference sample-sand from Only Concrete-Water; V-OC-M: Variable of study-sand from Only Concrete-Mucilage; R-AiO-W: Reference sample-sand from All in One-Water; V-AiO-M: Variable of study-sand from All in One-Mucilage

2.2.2. Sustainable Brick Manufacturing Process

The solid materials were manually mixed with a shovel until they were homogeneously integrated. Then, two-thirds of the liquid component (W or M) were added to the mixture which was allowed to rest during 10 min to facilitate the hydration process. Afterwards, the materials were mixed until a uniform consistency was obtained. Finally, the remaining liquid component was added under continuous mixing until suitable handling consistency was achieved.

Four bricks were made from each mixture for compressive strength, absorption and microstructural characterization tests. Once the mixture was ready, it was placed in the brick making machine. The manufactured sustainable bricks were 26 cm long, 12 cm wide and 5.5 cm tall, in accordance with NMX-C-441-ONNCCE-2013 [34]. The process consisted of an initial manual compaction of three layers having a thickness of approximately 2 cm each, accomplished with a 15×7 cm wood plate. Then, a second manual compaction was performed with the cap of the molding machine. Once the molding process had been completed, the bricks were extracted from the mold by means of the extraction lever.

The sustainable bricks were then exposed outdoors for three days for drying. Afterwards, they were introduced into a solar desiccator to accelerate the drying process. The daily monitoring of internal and external temperatures, and the solar radiation captured by the desiccator allowed for establishing the conditions to which the bricks were exposed (Figure 2). Data were recorded during five days after the initial external drying and readings were performed every 30 min during the maximum solar intensity period (around noon). The purpose of these determinations was to reference the testing environment and collect data for future correlations with other investigations. A thermocouple-thermometer data logger model EA15 (Extech, Nashua, NH, USA) and a sensor with pyrometer LabQuest-2 (Vernier Software & Technology, Beaverton, OR, USA) were used to determine temperatures and solar radiations, respectively. The drying process of a sustainable brick was assumed to be complete when a constant weight was obtained in two readings taken on two consecutive days.



Figure 2. Environmental conditions of the brick drying process.

Figure 3a,b shows images of the configuration and functioning of the two devices (brick making machine and desiccator) specifically designed for this study.



Figure 3. (a) Configuration of brick making machine; (b) Desiccator.

2.2.3. Brick Specifications and Test Procedures

Simple compressive strength tests and initial maximum water absorption tests were performed in accordance with NMX-036-ONNCCE-2013 and NMX-037-ONNCCE-2013, respectively. For the compressive strength tests, a 120-t manual compression machine, ELACONSA brand, was used, while the water absorption tests were performed with auxiliary laboratory equipment such as a mercury thermometer with a range from -22 to 100 °C and a digital scale 5 kg \times 1 g, ADIR brand, model 1676, in compliance with NMX-037-ONNCCE-2013. The compressive strength tests were performed at 14 days while the water absorption tests were performed at 28 days.

To characterize the matrix of the resulting material, observations were made using scanning electron microscopy (SEM) with JSM-6510 Series Scanning Electron Microscope machine, Jeol Ltd., Tokyo, Japan machine, with $30 \times$, $65 \times$ and $200 \times$ magnification images of resin-embedded bricks and posterior surface polishing. Later, the chemical composition of the samples was determined through the detection of basic compounds using the microanalysis technique with an Energy Dispersive Spectrometer (EDS) (Inca 200, Oxford Instruments, Abingdon-on-Thames, UK) connected to the SEM unit.

Finally, a thermal gravimetric analysis (TGA) was performed to correlate the mechanical, chemical and resistance behaviors to the high temperatures of the mixtures. The study was conducted in the TGA composed of an oven (Nabertherm Industrieofenbau, Lilienthal, Germany) equipped with a data acquisition system (model LSM-200, Amtsgericht Arnsberg, Arnsberg, Germany) that automatically records the weight and temperature to which the samples are submitted within the test crucible (maximum volume capacity of 10 mL). The temperature ramp was 3 °C per minute in the range from 50 to 950 °C.

The samples used for the TGA study were obtained from manually crushing untested bricks to obtain particles smaller than 4 mm. A granulometry study was performed to normalize particle size distribution. This, together with the volumetric weight obtained from usual procedures [35] permitted to obtain scale samples representative of the original.

3. Results and Discussion

3.1. Brick Drying by Solar Radiation

Figure 4 shows the weight loss evolution expressed in percentage. There is an obvious similarity in the drying process of all the study samples, characterized by a rapid and constant loss of humidity until Day 8, followed by a relative stabilization during the following four days.



Figure 4. Weight loss caused by brick desiccation.

3.2. Volumetric Weights

Figure 5 shows the sample profiles and volumetric weights; all profiles follow a similar trend, leading to the conclusion that all of the samples were subjected to a similar process.



Figure 5. Profiles and volumetric weights of the crushed samples.

3.3. Compressive Strength and Initial Maximum Absorption

Figure 6 shows the results of the compressive strength tests and the minimum acceptable limits, in both craft bricks (3.0 MPa) and extruded bricks (4.0 MPa) in accordance with NMX-C-036-ONNCCE-2013 [36] (in the particular case of the European Code, for clay bricks intended to be used in buildings in highly seismic zones, the minimum limit is raised to 5.0 N/mm^2) [37]. The grey bars indicate the compressive strength resulting from the use of OC or AiO with W only to hydrate

the mixture; while the blue bars refer to mixtures with OC or AiO but with the addition of M. Thus, globally, it is possible to determine the compressive strength of the studied variables as follows (all of them are greater than the minimum limits): compared to their respective reference samples, Variable of study-sand from Only Concrete-Mucilage (V-OC-M) led to a 9% compressive strength increase at 0.53 MPa, while Variable of study-sand from All in One-Mucilage (V-AiO-M) led to a 7% compressive strength increase at 0.65 MPa. These data evidence the favorable effect of the use of component M in the mixtures. Comparing both mixtures, V-AiO-M is 22% more resistant than V-OC-M, and thus the use of AiO as an OC replacement material can be considered—at least as far as resistance is concerned.



Figure 6. Compressive strength and initial maximum absorption values.

The orange tilted lines show the absorption variation of the study samples deriving from the differentiating use of W or M: Compared to their corresponding references, the initial maximum water absorption value obtained with V-OC-M mixture is 15% lower, while it is 13% lower with V-AiO-M. Moreover, both samples also meet the maximum absorption requirements established in NMX-C-037-ONNCCE-2013 [38]. As regards initial maximum water absorption and compressive strength, the use of M generates identical deviations (2%), as compared to their corresponding references, enhancing coherence and solidity. OC shows a 52% lower absorption capacity than AiO and since the absorption of the components of a matrix is a parameter of interest (dosage, durability, etc.); this fact will have to be taken into account in deciding whether a sustainable brick is appropriate for a given application. Finally, the existence of other mechanical parameters (freezing–thawing, fire), physical parameters (density, water steam permeability, efflorescence, thermal conductivity, water movement, salt contents) or geometrical parameters (aspects, cracks, lumps, flaking, etc.) that may also have to be complied with in other countries [37], must be mentioned.

3.4. Thermal Gravimetric Analysis

The TGA test was selected as a calibration indicator of the mechanical behavior of the sustainable bricks because it permits the determination of the limit (or fire resistance) and link to the matrix basic components.

The TGA results presented in Figure 7 indicate a weight loss, for all samples studied, ranging from 14% to 18% with respect to the initial weight. The maximum loss band is located in the adjacent

range between 300 °C and 600 °C; limits were established by the moving average curves with base 30 (\overline{X}_{30}) (Equation (1)) (30 was the value selected to determine the curves for favoring a better sensitivity coupling to the curves obtained from the TGA):

$$\overline{X}_{30} = \frac{\sum_{i=1}^{n} x_i}{n} \tag{1}$$

where: (\overline{X}_{30}) = moving average; n = 30, where each individual value (x_i) represents the percentage difference of weight loss between consecutive TGA measurements.



Figure 7. Weight loss vs. temperature increase.

The weight loss (from the limits of the bands previously established) to the final weight ratio was obtained by comparing the partial and total areas under the curves: percentage weight loss vs. temperature increase. The calculation process was based on the trapezoid method according to Equation (2) for each pair of increase values of its weight loss percentage (W) and its respective temperature (T), in order to determine the total area under the desired curve.

$$\int_{0}^{i} f(x)dx = (t_{2} - t_{1}) \left[\frac{W_{1} + W_{2}}{2} \right]$$
(2)

where: t_1 (°C) is the initial temperature of the studied pair of values; t_2 (°C) is the final temperature of the studied pair of values; W_1 (%) is the initial weight percentage of the studied pair of values; W_2 (%) is the final weight percentage of the studied pair of values; and f(x) (%-°C) is the area under the W-T curve.

Comparing the areas obtained in the maximum loss band to the total area led to the determination that they occur in 33.8% vs. 33.9% of the total area (a difference of only 0.1%), which corresponds to a weight loss variation from 72% to 64% (8%)—similar to the critical area of the maximum loss band with significant sample weight loss; so its study and linkage with the behavior of the mixtures is binding.

The analysis of the individual mixtures permitted the determination of a 13% weight loss percentage with V-OC-M, which is 2% less than the control mixture, showing thus that the use of M brings about a structural improvement to the matrix (Figure 8a). A similar trend was observed with

V-AiO-M (11% weight loss compared to the initial weight, representing a 1% reduction) (Figure 8b). In both cases, the correlation between mechanical resistance increase or absorption reduction and weight loss reduction indicates microstructural improvements (better union of constituent particles) and increased densification of the sustainable brick matrix.



Figure 8. (a) Thermal Gravimetric Analysis (TGA) of OC; (b) TGA of AiO.

As regards the use of OC and AiO materials, the comparison between the two curves shows a 1% difference between them, indicating that the use of AiO material in the matrix is clearly appropriate.

In order to determine the compounds or materials that contribute to the mechanical improvement of the matrix of the different samples, Figure 9 presents the compounds and materials identified in previous studies. Absorbed water, organic charcoal, wood residues, clay minerals, pyrite, kaolinite, illite and alpha quartz have a significant impact on weight loss (shaded area).



Figure 9. Weight loss ranges for different materials due to the increase in the temperature of ceramic matrices. Data from [20,21,23,26,39,40].

The general behavior of each one of the study samples may possibly be due to the total combination of the different compounds constituting it. However, it is also evident that some of these compounds may have a significant direct impact. Thus, in order to determine the incidence of each one of the compounds or materials, a comparison of the areas below the curves in the maximum loss range was performed for each compound present in this zone.

The comparison between V-OC-M and its reference highlighted three compounds that showed a higher incidence, which are, in descending order of importance in the matrix: wood residues, clay minerals and illite. Similarly, the comparison between V-AiO-M and its reference established that wood residues and clay minerals were also the two compounds having the greatest impact on the general behavior of the samples, the third one being, in this case, organic carbon [40].

Illite (to a greater extent) and kaolinite (to a lesser extent) are present in both mixtures (V-OC-M and V-AiO-M), both compounds being more abundant in the first mixture, and thus causing the greatest weight loss by incineration. The above comments are linked to the results of compressive strength and absorption tests, in which resistance increases and absorption reduction were observed in the samples that lost less weight in the thermo-gravimetric analysis compared to their corresponding reference samples.

To validate the previous relationships, the matrix of each sample was observed by SEM. In Figure 10a,b two images are presented in which zones of low density or high porosity (circles), presence of fissures (arrows) and tortuous interfacial transition zone (ITZ) with poor element integration can be identified in the case of V-OC-M, while the opposite is observed in the case of V-AiO-M.



Figure 10. SEM of study samples (**a**) Variable of study-sand from Only Concrete-Mucilage (V-OC-M); (**b**) Variable of study-sand from All in One-Mucilage (V-AiO-M).

TGA tests had previously established that the basic chemical elements are carbon, oxygen, aluminum and silica. By mapping these elements with the complementary EDS technique of the SEM study, their significant presence is validated and this could explain the mechanical and physical behavior of the studied mixtures, since the greater presence of said elements in the reference samples could lead to a worse performance. Each white point identifies the location of the chemical elements in the sample, making it thus possible to compare their concentrations or determine their presence. Figure 11a,b show the chemical elements of each mixture, identifying their presence, the images corresponding to the same mixtures, but to different observation zones. Figure 11a (V-OC-M) shows greater concentrations or presence of these elements (most significant in the case of Al, C and Si) than Figure 11b (V-AiO-M); this being the element concentrations leading to property reductions.



Figure 11. Micro EDS through mapping of the main elements established in SEM assays for the samples (a) V-OC-M; (b) V-AiO-M.

4. Conclusions

The use of construction wastes for the manufacture of sustainable bricks can be an environmentally friendly and technologically feasible alternative since, on the one hand, it makes use of materials which are deemed residues and no thermal baking is involved in their manufacture and, on the other hand, it meets the requirements of the local regulations for application as construction elements for nonstructural purposes.

The use of nopal mucilage in the mixture improves the mechanical and physical properties of the bricks, increasing their compressive strength and lowering their initial maximum water absorption compared to bricks that are manufactured only with water. Moreover, it enhances the resistance to high temperatures. Likewise, the matrix of the bricks incorporating mucilage shows a better adhesion between its particles, a smaller amount of pores and stronger zones of interfacial transition.

In contrast to concrete wastes, the use of construction wastes (AiO) is not regulated in Mexico (except its use as landfill or highway base and sub-base). However, this study validates the fact that construction wastes could be used as an alternative in the manufacture of sustainable bricks since, besides complying with the minimum parameters established by the local regulations, its performance is comparable to that of bricks manufactured with only concrete (CD).

Therefore, taking into account exclusively the results exposed in this paper, the alternative materials used in this research could replace virgin raw materials for making sustainable bricks for non-structural purposes; however, it is important to emphasize that these materials are wastes (diversity of constitutive substances and complexity of its treatment), and thus further studies are required before these materials can be considered technically and environmentally appropriate for use in the building industry.

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