

Characterizing Madagascar's Ancestral Walls And Indonesian Laterite For Data-Driven Unfired Brick Design

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Abstract: Lateritic soils used in Madagascar's ancestral Tamboho walls offer a low-carbon route to unfired masonry, yet the chemistry–microstructure–performance links needed for design and conservation remain unclear. This study quantified those links across inner cores and weathered crusts from four Imerina plateau sites by combining bulk oxide analysis (X-ray fluorescence), phase and crystallinity identification (X-ray diffraction), microstructural imaging and microchemistry (SEM/EDS), and standardized mechanical and physical testing. Inner cores exhibit higher alumina with moderate silica and iron, phase assemblages dominated by kaolinite–gibbsite–quartz with minor Fe-oxides, and denser fabrics with continuous Al–Si-rich films, whereas crusts show uniformly lower oxide contents, relatively stronger gibbsite signatures, and more connected porosity. Cores achieve higher dry unconfined compressive strength (1.61–2.10 MPa) than crusts (1.28–1.46 MPa) and retain 49–64% of strength after 24 h immersion; densities cluster near 1.60 g cm⁻³ for cores versus 1.50 g cm⁻³ for crusts, with porosity and absorption tracking microstructural openness. Regression models link dry strength positively to Fe₂O₃ and Al₂O₃/SiO₂ and relate wet-strength retention to crystallinity and lower porosity, explaining most variance. Collectively, the results define practical formulation windows for unfired laterite bricks and a compact verification suite (XRF/XRD checks plus porosity/absorption and dry/wet strength), and they inform compatible conservation strategies for Tamboho walls while guiding sustainable masonry design in tropical climates.

Keywords: Lateritic soils; Tambohon'ny Ntaolo; Unfired laterite bricks; XRF, XRD, SEM/EDS; Microstructure–property relationships

1. Introduction

Earthen construction is receiving renewed attention as cities and regions grapple with the carbon footprint of conventional building materials and the need for resilient, locally appropriate technologies. Cement manufacture alone is estimated to contribute roughly 8% of global CO₂ emissions, underscoring the urgency of advancing low-carbon alternatives for building envelopes and infill systems [1]. In tropical and subtropical settings, lateritic soils—residual products of intense weathering—offer a compelling pathway because they are abundant, require minimal processing, and can gain strength through natural hardening, often without high-temperature firing [2], [3], [4], [5]. Madagascar's Central Highlands preserve a long-standing tradition of laterite-based defensive and boundary structures known as *Tamboho Gasy* or *Tambohon'ny Ntaolo*, providing an exemplary natural laboratory for examining composition–microstructure–property relationships in earthen materials under real climatic exposure [6], [7].

Historically, “*tamboho*” denotes an earthen wall enclosing and protecting a space, and “*Ntaolo*” refers to the ancestors whose craft established the practice. These walls emerged and proliferated in *Imerina* during the late eighteenth and early nineteenth centuries, including early works attributed to King *Andrianampoinimerina*, and were constructed from locally

available laterite mixed with organic additions such as rice straw and cow dung [6], [7]. Ethnographic and architectural accounts emphasize their socio-economic roles—demarcation, security, and symbolism—while technical observations point to their durability: many walls have persisted for more than a century without cementitious binders, withstanding seasonal wetting and drying cycles typical of the highland climate [6], [7], [8]. The persistence of these structures raises a fundamental materials question: which aspects of their chemistry, mineralogy, and microstructure jointly account for their strength and water resistance in service?

Over the past decades, laterite has been characterized as an alumino-silicate system dominated by sesquioxides (Fe_2O_3 and Al_2O_3) and low to moderate silica, with mineral phases typically including kaolinite, gibbsite, quartz, and hematite/goethite [2], [3], [4], [5]. Recent accounts specific to *Tamboho* walls document dry compressive strengths around 1.2–2.7 MPa and porosities on the order of 15.85–18.65%, with the materials maintaining cohesion after several days of water immersion—properties that are remarkable given the absence of hydraulic cements [8]. Yet these characterizations, while valuable, remain largely descriptive and site-specific. They have not been translated into quantitative, predictive links connecting bulk oxide ratios, phase assemblages, and pore structures to mechanical performance, especially wet strength retention.

This absence of a predictive framework constrains conservation practice and sustainable design. Without robust relationships that connect elemental/mineralogical makeup and microstructure to performance, attempts to fabricate compatible unfired replacement bricks or to design new low-carbon masonry inspired by *Tambohon'ny Ntaolo* remain trial-and-error. A general solution is to adopt an integrative, multi-scale approach that couples compositional analysis with microstructural imaging and mechanical testing across multiple sites and wall layers. By assembling coherent datasets from X-ray fluorescence (XRF), X-ray diffraction (XRD), scanning electron microscopy/energy-dispersive spectroscopy (SEM/EDS), and standardized strength/porosity measurements, it becomes feasible to infer which chemical–mineralogical variables and textural features are the principal drivers of strength and durability [2], [5], [8].

Prior literature offers important building blocks for such an approach. Classic frameworks of laterite genesis and hardening emphasize hydrolysis, leaching of silica, and subsequent precipitation/crystallization of alumina- and iron-rich phases that progressively cement grains during wet–dry cycling [2], [4], [5]. In parallel, studies on earthen bricks demonstrate that organic stabilizers can beneficially modify the pore structure and cracking behavior. Rice straw—rich in lignocellulosic fibers and containing reactive silica—has been reported to improve tensile behavior, reduce shrinkage, and enhance thermal performance when appropriately pretreated [9], [10], [11], [12]. Cow dung, with its fibrous content and microbial activity, has been shown to contribute to water retention, densification, and durability in earthen composites [13], [14], [15]. The net result in unfired bricks is often an increase in compressive strength and moisture resistance relative to unstabilized controls [16], [17].

At the same time, the literature underscores performance trade-offs and the need for quantitative guidance. Reported strengths of unfired earthen blocks vary widely with mix design and compaction, and property gains from organic additives can be contingent on dosage, fiber aspect ratio, and pretreatment [16], [17]. Benchmarks against fired bricks reveal that, while firing typically yields higher nominal compressive strength, unfired systems can achieve competitive values while offering substantially lower embodied energy and greenhouse emissions—particularly when agricultural by-products are incorporated [18], [19], [20], [21], [22]. For heritage conservation, strength parity alone is insufficient: compatibility in composition and microstructure is essential to minimize chemical and hygric incompatibilities with original fabric [7], [8]. These considerations reinforce the necessity of data-driven “formulation windows” that target oxide ratios, phase assemblages, and porosity consistent with durable *Tamboho* walls.

Closely related studies have catalogued the mineral phases common to inner and outer wall layers—quartz, kaolinite, gibbsite, hematite—with differences in relative abundance attributable to weathering gradients. Reports suggest that outer surfaces exhibit silica leaching and enrichment in kaolinite/gibbsite, while inner layers retain higher silica, with concomitant differences in densification and pore connectivity [2], [5], [8]. Yet few contributions attempt to model how variations in Al_2O_3 – SiO_2 – Fe_2O_3 – TiO_2 ratios, crystallinity indices, and pore metrics translate into predicted dry and wet strengths—nor do they systematically compare multiple geographic sites and stratigraphic positions within the same heritage system [18], [21], [22],

[23]. In short, the literature provides a qualitative mechanism and a set of empirical observations, but not an integrated, quantitative design framework tailored to Madagascar's ancestral walls.

The present study addresses this gap by assembling a multi-site, multi-layer dataset from *Tambohon'ny Ntaolo* walls and reference laterites, linking chemistry, mineralogy, and microstructure to strength and wet-strength retention through coordinated XRF, XRD, SEM/EDS, and mechanical testing. Specifically, we (i) compare inner and outer layers from RAD, MAN, AMB, and MAH sites; (ii) quantify bulk oxide ratios and phase assemblages; (iii) characterize pore architecture and grain-bonding textures; (iv) relate these descriptors to dry and wet compressive strengths and porosity; (v) benchmark against a reference soil; and (vi) synthesize outcomes into data-driven formulation windows for compatible unfired laterite bricks. Our working hypotheses are that (1) Al_2O_3 – SiO_2 – Fe_2O_3 – TiO_2 ratios, together with phase assemblage, govern bonding and strength; (2) lower porosity and higher crystallinity are associated with higher dry and retained wet strengths; (3) weathering produces systematic inner–outer differences via silica leaching and gibbsite enrichment; (4) microstructural heterogeneities, including Fe-rich nodules and pore textures, modulate water resistance; and (5) integrating multi-technique data enables predictive models that generalize across sites [2], [5], [8]. By advancing from descriptive characterization to quantitative structure–property relations, the study seeks to inform both conservation of Malagasy heritage walls and the development of low-carbon, context-appropriate unfired laterite masonry.

2. Materials and Methods

2.1 Study Area and Sampling

The study targeted ancestral lateritic walls in Madagascar's central highlands. Four representative sites on the Imerina plateau were examined: Miadapahonina (RAD; Itasy Region), Manandriana (MAN; Analamanga), Ambohitrabiby (AMB; northern flank of Antananarivo), and Ambohimandray (MAH; Analamanga). Elevations span roughly 1,250–1,450 m, with a cool, humid subtropical climate characterized by wet summers and dry winters. Bedrock comprises basalt and gneiss mantled by deeply weathered ferrallitic profiles; soils are lateritic and enriched in Al–Fe oxides with moderate silica and plasticity. Approximate coordinates are given in Table 1 to orient the sites. Each

location preserves 19th-century earthworks, enabling sampling of both inner and weathered outer materials.

Sampling followed a stratified design by wall depth. The outer layer (coded “ext”) corresponds to the weathered crust (~2–3 cm), while the inner layer (coded “in”) represents the underlying intact fabric. After removing the crust, ~2 kg of bulk material per layer were collected into polyethylene bags, sealed, and labelled (RAD_in, RAD_ext, MAN_in, MAN_ext, AMB_in, MAH_in). A reference laterite (ID1) from Ampel, Bolyolali, Indonesia was also collected to benchmark composition and physical behavior. Table 1 lists codes, coordinates, and brief contextual notes, and Figure 1 shows representative hand specimens prior to preparation.



Figure 1: On-site wall blocks

Table 1: Geographical Coordinates of the samples collection

Sites	Designation	Geographical Coordinates	
		Latitude	Longitude
Site 1	Miadapahonina (RAD)	18°48'36.78"S	47°35'25.84"E
Site 2	Manandriana (MAN)	18°48'49.94"S	47°35'05.25"E
Site 3	Ambohitranenaina Ambohitrabiby (AMB)	18°47'54.41"S	47°35'20.05"E
Site 4	Ambohinaorina Ambohimandray (MAH)	18°46'16.70"S	47°21'40.40"E
Site 5	Ampel Boyolali (ID1)	-7°44'47.02"S	110°58'85.67"E

2.2 Materials and Sample Preparation

All samples were air-dried, then oven-dried at 105 °C to constant mass. Dried blocks were gently crushed (rubber-coated hammer, porcelain mortar) and sieved to <2 mm; the <2 mm fraction was split by technique. For X-ray fluorescence (XRF), aliquots were agate-milled to <75 µm and pressed into 25 mm pellets (~5 g) at ~20 t; a light boric-acid dusting produced smooth, reproducible surfaces. For X-ray diffraction (XRD), powders were micronized to <10 µm and back-loaded into circular holders to obtain a flush, planar surface without binder; film-supported mounts were used only when necessary for very loose powders. For scanning electron microscopy with energy-dispersive spectroscopy (SEM/EDS), ~1 cm³ fragments were sawn, polished with 800-grit SiC, rinsed, dried, mounted on aluminum stubs with carbon tape, and sputter-coated with ~10 nm Au-Pd; additional powder-on-tape mounts were prepared to observe fine agglomerates.

Mechanical specimens were prepared from laterite mixed with deionised water to near-plastic consistency. Cubes (50 mm) were cast for unconfined compressive strength (UCS) and, where required, prisms (50 × 100 mm) for modulus. Specimens were air-dried for seven days then cured at 40 °C to constant mass. Wet-strength tests used cubes immersed for 24 h, surface-dried immediately before testing; retention was expressed as (wet/dry) × 100 %. Dry density was computed from mass and dimensions; apparent porosity and water absorption were derived by saturation–drying from mass differences before and after 24 h immersion. Throughout preparation, cross-contamination was minimized by cleaning tools and using dedicated sieves. A minimum of three replicates was produced per condition.

Quality controls applied during preparation included mass-yield tracking and replicate consistency checks. Acceptance criteria used subsequently for each technique are summarized in Table 2.

Table 2: Quality control measures and acceptance criteria (CRM = certified reference material)

Parameter	Acceptance criterion	Measures
XRF major oxides	RSD ≤ 5 %; LOD ≤ 0.05 wt %	CRM and blanks; duplicate pellets
XRD precision	Peak repeatability ≤ 0.02° 2θ; Δ-crystallinity ≤ 1 %	Si standard; replicate mounts; film-peak exclusion
SEM/EDS	Elemental-sum uncertainty ≤ 3 at. %	Coating checks; repeat points/lines; instrument standards
Compressive strength (UCS)	SD ≤ 0.15 MPa	Calibrated load cell; ≥3 cubes/condition
Density/porosity	Variation ≤ 2 %	Calibrated balance/caliper; repeat saturation–drying

2.3 Analytical Techniques

- **XRF (bulk oxides):** An energy-dispersive configuration with a rhodium X-ray tube (typ. 40–50 kV) and silicon-drift detector was used under helium purge to improve low-energy sensitivity. Each pellet was counted in triplicate for ~300 s. A standardless fundamental-parameter approach was applied; elements with $Z \geq 13$ were reported as oxides normalized to 100 %.
- **XRD (phases and crystallinity):** Powder patterns were collected in Bragg–Brentano reflection geometry using Cu $K\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$). Scans covered $10\text{--}80^\circ 2\theta$ with 0.02° steps and $\sim 0.3\text{--}0.4 \text{ s}$ dwell; a spinner ($\sim 20 \text{ rpm}$) was enabled to improve particle statistics. Background subtraction and light smoothing preceded phase identification with an indexed reference database. Semi-quantitative abundances were estimated by reference-intensity-ratio methods. Crystallinity indices were calculated as the ratio of crystalline peak area to total diffractogram area.
- **SEM/EDS (microstructure and microchemistry):** Coated fragments were imaged in high vacuum at $\sim 15 \text{ kV}$ and $\sim 10 \text{ mm}$ working distance to document pore networks, particle packing, and Fe-oxide nodules. EDS point analyses and line scans ($\sim 60 \text{ s}$ dwell) were performed on selected fields; elemental maps for Al, Si, Fe, and Ti were acquired at moderate resolution. Powder mounts were examined in variable-pressure mode ($\sim 20 \text{ kV}$) to study fine agglomerates.
- **Mechanical and physical tests:** UCS was measured on 50 mm cubes at $\sim 0.25 \text{ MPa}\cdot\text{s}^{-1}$, following standard practice. Dry density was recorded on oven-dried cubes; porosity and absorption were computed from saturated versus dry masses after 24 h immersion. Results are reported as means $\pm$ SD with $n \geq 3$. Built-in QA/QC included certified references and blanks for XRF, a silicon standard for XRD alignment with replicate mounts, and verification of load-cell calibration before each series.
- **Data reduction and modeling:** Composition (XRF), phase/crystallinity (XRD), and microstructural metrics (SEM/EDS, porosity) were screened for collinearity and entered into correlations and simple regressions ($\alpha = 0.05$; VIF screening) to predict dry UCS and wet-strength retention. The workflow that links measurements to the formulation window for unfired laterite bricks is presented later in the Results.

3. Results

3.1 Chemical Composition (XRF)

XRF analyses confirm that the inner walls exhibit typical lateritic chemistry. Alumina and iron dominate over silica, with cores containing 42–45 wt % Al_2O_3 , 29–31 wt % SiO_2 , 6.9–8.7 wt % Fe_2O_3 and 1.35–1.55 wt % TiO_2 . MAN_in is slightly richer in alumina, MAH_in has the highest iron, and RAD_in the lowest. By contrast, the exposed crusts (RAD_ext, MAN_ext) display substantially lower oxide contents, with Al_2O_3 and SiO_2 reduced to $\sim 18\text{--}19$ and 12 wt %, respectively, and Fe_2O_3 and TiO_2 to about 6 and 1 wt % (Table 3). Figure 2 illustrates these patterns clearly, underscoring the leaching of aluminium and silica and the residual nature of lateritic soils. These overall geochemical gradients underpin the mineralogical and mechanical trends discussed below.

Table 3: Chemical composition by site and layer (mean $\pm$ standard deviation).

Sample	Al_2O_3 (wt %)	SiO_2 (wt %)	Fe_2O_3 (wt %)	TiO_2 (wt %)
RAD_in	43.15 ± 0.64	29.02 ± 0.47	6.95 ± 0.08	1.35 ± 0.02
MAN_in	44.83 ± 0.80	29.04 ± 0.91	7.97 ± 0.37	1.55 ± 0.08
MAH_in	42.20 ± 0.19	31.34 ± 0.18	8.65 ± 0.12	1.42 ± 0.02
AMB_in	10.65 ± 0.00	16.64 ± 0.00	10.22 ± 0.00	0.73 ± 0.00
RAD_ext	18.72 ± 0.15	11.89 ± 0.08	5.91 ± 0.03	0.94 ± 0.02
MAN_ext	18.19 ± 0.21	12.52 ± 0.05	6.21 ± 0.03	1.02 ± 0.01

3.2 Mineralogical Phases and Crystallinity (XRD)

XRD patterns for all analysed samples identify quartz, kaolinite, gibbsite, and hematite/goethite, with a small amorphous fraction. Semi-quantitative phase proportions and crystallinity indices are summarised in Table 2, and representative diffractograms are shown in Figure 4. Across the inner layers (RAD_in, MAN_in, MAH_in), quartz and kaolinite are the dominant phases, accompanied by ~20–25 % gibbsite, ~6–10 % iron oxides, and ~3–5 % amorphous content. The crusts (RAD_ext, MAN_ext) display lower quartz (~25–27 %) and higher gibbsite (~30 %) relative to cores, while kaolinite and iron-oxide contents remain broadly comparable. Crystallinity varies little among sites or layers (~71–73 %), indicating a similar degree of structural order throughout the materials.

These mineralogical patterns are coherent with the oxide chemistry reported in the chemical composition: inner layers that show high Al_2O_3 and moderate SiO_2 align with abundant kaolinite and gibbsite together with iron oxides, whereas outer layers—where oxides are generally lower—exhibit attenuated quartz peaks and relatively stronger gibbsite contributions. The narrow spread of crystallinity values suggests that phase proportions, rather than large changes in overall crystallinity, are the main mineralogical feature distinguishing cores from crusts. Figures 3,4,5, and 6 illustrate these contrasts by showing weaker quartz reflections and enhanced gibbsite features in the crust diffractograms when compared side-by-side with their corresponding cores.

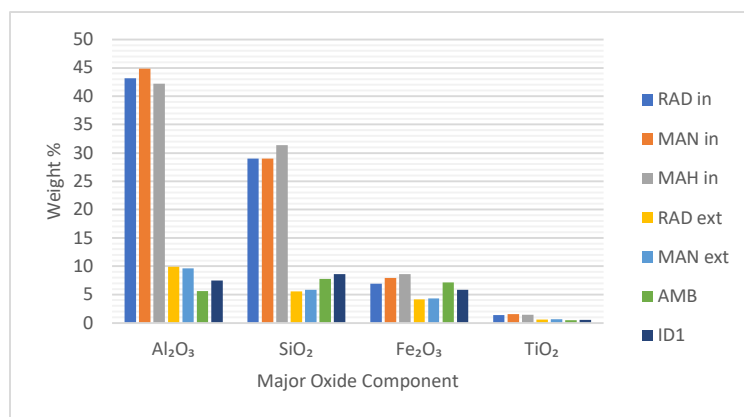


Figure 2: Mineralogical composition of the samples

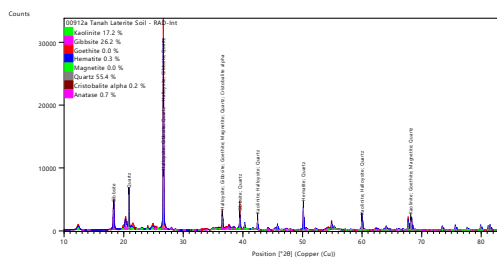


Figure 3: XRD of RAD_in sample

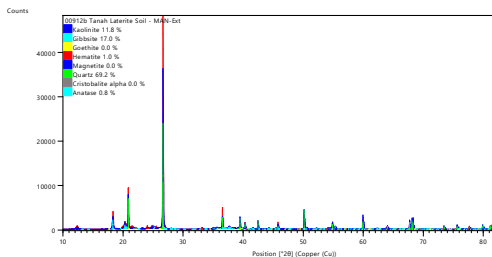


Figure 4: XRD of MAN_ext sample

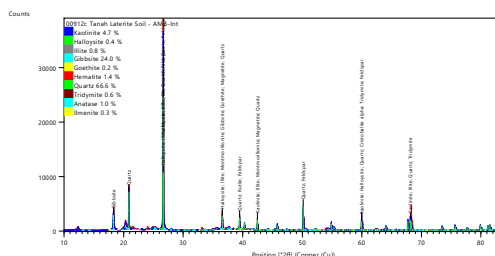


Figure 5: XRD of AMB_in sample

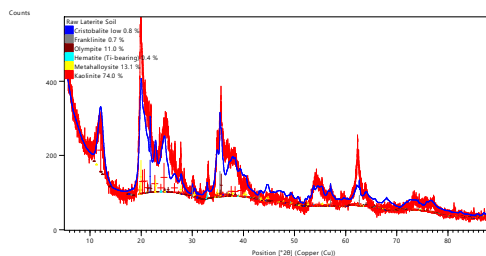


Figure 6: XRD of MAH_in sample

Table 4: Mineralogical composition and crystallinity (mean $\pm$ standard deviation).

Sample	Quartz (%)	Kaolinite (%)	Gibbsite (%)	Hematite/Goethite (%)	Amorphous (%)	Crystallinity (%)
RAD_in	35 $\pm$ 3	30 $\pm$ 2	20 $\pm$ 2	10 $\pm$ 2	5 $\pm$ 2	72.3 $\pm$ 0.5
MAN_in	32 $\pm$ 3	32 $\pm$ 2	25 $\pm$ 2	8 $\pm$ 1	3 $\pm$ 1	72.5 $\pm$ 0.5
MAH_in	40 $\pm$ 2	28 $\pm$ 2	20 $\pm$ 1	7 $\pm$ 1	5 $\pm$ 1	71.5 $\pm$ 0.5
AMB_in	38 $\pm$ 2	30 $\pm$ 2	22 $\pm$ 1	6 $\pm$ 1	4 $\pm$ 1	72.0 $\pm$ 0.5
RAD_ext	25 $\pm$ 3	30 $\pm$ 2	30 $\pm$ 2	10 $\pm$ 1	5 $\pm$ 1	71.0 $\pm$ 0.5
MAN_ext	27 $\pm$ 3	28 $\pm$ 2	30 $\pm$ 2	10 $\pm$ 1	5 $\pm$ 1	71.2 $\pm$ 0.5

3.3 Microstructure and Microchemistry (SEM/EDS)

SEM observations distinguish compact inner fabrics from more open outer crusts. Inner core (RAD_in) display tightly packed angular grains with clay-size aggregates that bridge interparticle contacts. Grain boundaries frequently show smooth, film-like coatings consistent with an alumino-hydroxide/clay binder, and isolated Fe-rich nodules occur within the matrix. In contrast, the exposed crusts (RAD_ext, MAN_ext) show a higher incidence of intergranular voids and micro-fissures, locally disrupted coatings, and grains with relieved edges, indicative of near-surface weathering and fabric loosening. These textural differences are visually apparent at comparable magnifications and are consistent with the reduced mechanical performance reported for crust specimens.

EDS spot and map analyses corroborate the oxide chemistry in chemical composition section. Matrix compositions are dominated by O, Al and Si, with Fe and Ti as minor constituents. Elemental maps reveal relatively uniform Al and Si within the fine matrix, while Fe is more heterogeneous, concentrating in discrete nodules or rims. Between layers, the inner cores retain continuous Al–Si–rich coatings, whereas the crusts exhibit patchier coverage and more exposed grain surfaces; Fe-bearing spots are present in both but remain spatially localized. Taken together, the SEM/EDS results support a picture of cores with better interparticle bonding and crusts with higher connectivity of pores and micro-cracks, providing a microstructural basis for the observed differences in strength and wet retention.

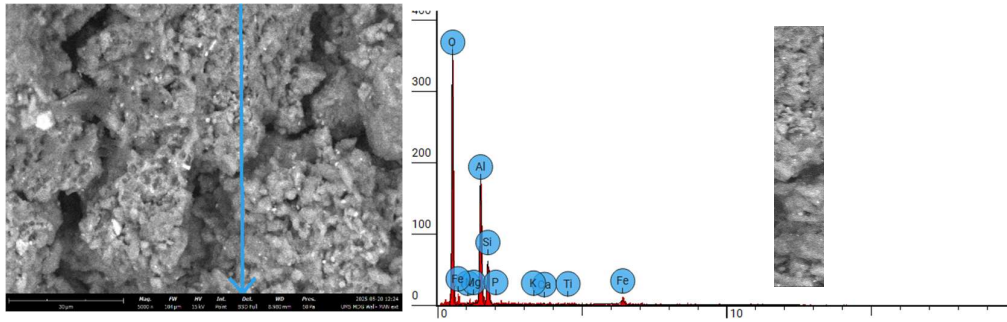


Figure 8: RAD_ext linescan and elements contents

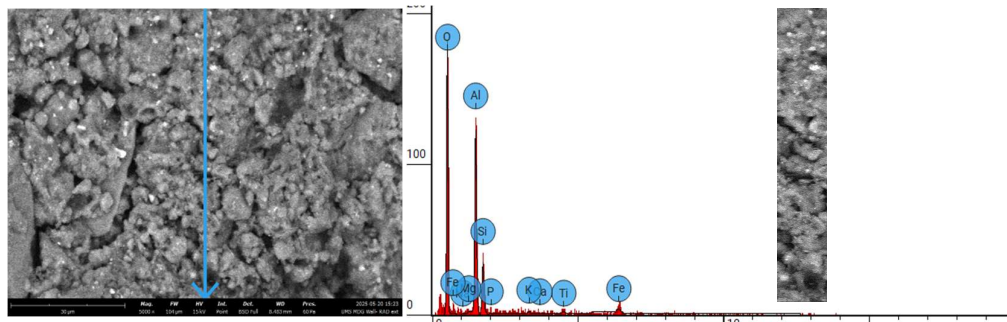


Figure 9: RAD_in linescan and elements contents

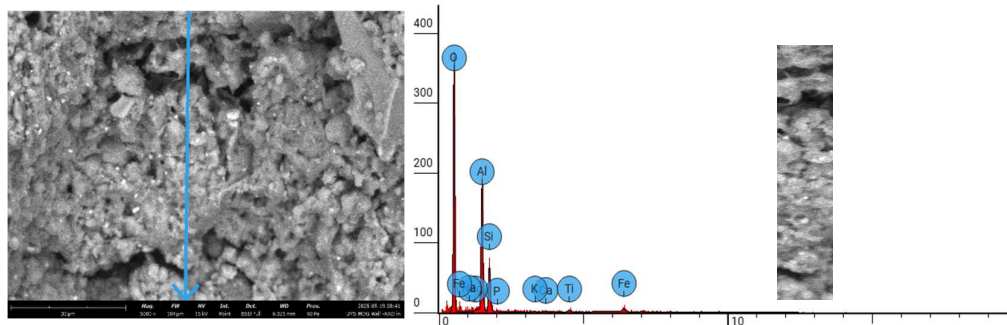


Figure 7: MAN_ext linescan and elements contents

3.4 Mechanical and Physical Properties

Compression testing shows that inner cores are consistently stronger than outer crusts (Table 5). Among cores, RAD_in records the highest dry UCS at 2.10 MPa, followed by AMB_in at 2.02 MPa, MAN_in at 1.72 MPa, and MAH_in at 1.61 MPa. For crusts, RAD_ext reaches 1.46 MPa and MAN_ext 1.28 MPa. After 24 h immersion, wet UCS decreases for all specimens: cores range from 1.16 MPa (RAD_in) and 1.10 MPa (MAN_in) to 1.00 MPa (AMB_in) and 0.94 MPa (MAH_in), while crusts are 0.80 MPa (RAD_ext) and 0.74 MPa (MAN_ext). Wet-strength retention for cores spans 49.2 % (AMB_in) to 64.1 % (MAN_in); crusts retain 54.8 % (RAD_ext) and 57.6 % (MAN_ext).

Physical indices mirror these trends. Core densities are 1.60 g cm^{-3} for all four sites, with porosities of 15.6 % (RAD_in), 16.5 % (MAN_in), 15.5 % (MAH_in), and 16.8 % (AMB_in) and water absorption of 15 % for each. Crust densities are 1.50 g cm^{-3} , with porosities of 13.7 % (RAD_ext) and 15.7 % (MAN_ext) and water absorption of 12 % for both. The pairing of higher compressive strength with core material and lower values for crusts is consistent across all metrics reported.

Table 5: Mechanical and physical properties.

Sample	Dry UCS (MPa)	Wet UCS (MPa)	Wet retention (%)	Density (g cm ⁻³)	Porosity (%)	Water absorption (%)
RAD_in	2.10 ± 0.17	1.16 ± 0.11	55.4 ± 4.7	1.60 ± 0.05	15.6 ± 0.5	15 ± 2
MAN_in	1.72 ± 0.05	1.10 ± 0.08	64.1 ± 3.6	1.60 ± 0.05	16.5 ± 0.4	15 ± 2
MAH_in	1.61 ± 0.11	0.94 ± 0.14	58.4 ± 6.0	1.60 ± 0.05	15.5 ± 0.3	15 ± 2
AMB_in	2.02 ± 0.07	1.00 ± 0.12	49.2 ± 4.7	1.60 ± 0.05	16.8 ± 0.1	15 ± 2
RAD_ext	1.46 ± 0.15	0.80 ± 0.08	54.8 ± 5.9	1.50 ± 0.05	13.7 ± 0.3	12 ± 2
MAN_ext	1.28 ± 0.04	0.74 ± 0.11	57.6 ± 8.0	1.50 ± 0.05	15.7 ± 0.4	12 ± 2

3.5 Composition–Microstructure–Performance Links

Regression analysis links composition and microstructure to strength. Dry strength increases with iron content and the Al₂O₃/SiO₂ ratio and decreases slightly with crystallinity. Wet retention improves with crystallinity and declines with porosity. Despite the small sample size, the models explain most variance ($R^2 \approx 0.98$ for dry strength and 0.96 for wet retention; Table 6). These trends suggest that higher iron and alumina favor stronger bonding and that ordered microstructures and low porosity enhance water resistance. Scatter plots (not shown) support these relationships. Such insights are consistent with studies that emphasize the joint roles of composition and microstructure in unfired brick performance [22], [23] and provide guidance for formulating durable, unfired laterite bricks.

Regression results indicate clear associations between composition, microstructure, and performance. For dry UCS, the model shows positive coefficients for Fe₂O₃ and Al₂O₃/SiO₂, and a negative coefficient for Crystallinity; Porosity is weakly positive. For wet-strength retention, the model yields a positive coefficient for Crystallinity and a negative coefficient for Porosity, with small effects for Fe₂O₃ and Al₂O₃/SiO₂. Despite the limited sample size, model fits are high (dry UCS $R^2 = 0.98$, RMSE = 0.09; wet retention $R^2 = 0.96$, RMSE = 1.2). These patterns align with the quantitative coefficients in Table 6. Scatter plots (not shown) support the directions of these associations.

Table 6: Regression coefficients for composition–performance models (β and p -values shown for each predictor).

Predictor	β (dry UCS)	p -value (dry)	β (wet retention)	p -value (wet retention)
Intercept	40.65	0.31	−171.13	0.66
Fe ₂ O ₃	0.45	0.18	0.23	0.92
Al ₂ O ₃ /SiO ₂	1.15	0.27	−10.54	0.37
Crystallinity	−0.63	0.31	3.82	0.55
Porosity	0.08	0.54	−2.12	0.32

Model fit: Dry UCS — $R^2 = 0.98$, RMSE = 0.09; Wet retention — $R^2 = 0.96$, RMSE = 1.2.

3.6 Site and Layer Comparisons

Across cores, RAD_in and AMB_in exhibit the highest dry strengths (2.10 MPa and 2.02 MPa), followed by MAN_in (1.72 MPa) and MAH_in (1.61 MPa). In terms of chemistry, MAN_in contains the highest Al₂O₃ (44.83 wt %), with RAD_in at 43.15 wt %, MAH_in at 42.20 wt %, and AMB_in at 10.65 wt %. Crusts show broadly similar responses between sites: RAD_ext and MAN_ext have lower strengths (1.46 MPa and 1.28 MPa dry; 0.80 MPa and 0.74 MPa wet), lower oxide abundances after

conversion to oxides, and gibbsite around 30 % with reduced quartz ($\approx 25\text{--}27\%$) relative to cores. Porosity for crusts is 13.7 % (RAD_ext) and 15.7 % (MAN_ext), compared with core values of 15.5–16.8 %.

Taken together, the core–crust contrast exceeds inter-core differences: cores combine higher strength with the inner-layer chemistry and mineralogy (higher alumina with quartz–kaolinite–gibbsite assemblages), whereas crusts exhibit lower oxide contents and increased gibbsite relative to quartz. These site- and layer-resolved observations are consistent with the regression results in the previous section, reinforcing that composition (e.g., Fe_2O_3 , $\text{Al}_2\text{O}_3/\text{SiO}_2$), microstructural attributes (porosity), and crystallinity act jointly to govern compressive strength and wet-strength retention.

4. Discussion

4.1 Mechanistic interpretation (from composition and phases to properties)

The multi-scale evidence assembled here supports a coherent mechanism linking bulk chemistry to phase assemblage, microstructure, and, ultimately, macroscopic performance. Inner layers contain higher Al_2O_3 (42–45 wt%) with moderate SiO_2 (29–31 wt%) and Fe_2O_3 ($\approx 7\text{--}9\%$), while outer crusts show uniformly lower oxide contents. In mineralogical terms, both layers are dominated by kaolinite–gibbsite–quartz with minor hematite/goethite, but crusts display attenuated quartz peaks with relatively stronger gibbsite signatures. These patterns are consistent with progressive desilicification under lateritic weathering and the re-precipitation of Fe as goethite/hematite that contribute to cementation and cuirasse development [2], [5], [24], [25], [26], [27]. The chemistry to phases link is therefore expressed as Al-rich, moderately siliceous cores that retain a balanced kaolinite–gibbsite–quartz matrix, and silica-depleted crusts in which gibbsite becomes relatively more prominent.

Phase assemblage, in turn, maps onto distinctive microstructures. SEM shows that cores possess denser fabrics with tighter particle packing and more continuous Al–Si-rich films that bridge grain contacts, whereas crusts exhibit disrupted coatings, higher void connectivity, and localized micro-fissures. Such textures align with interfacial “gel–oxide interlocking” in which aluminosilicate hydrates nucleate on Fe-oxide-bearing grains and reinforce interparticle bonds; where coatings are locally removed or discontinuous, pore throats remain larger and more percolating [28], [29], [30]. The phases to microstructure step are therefore mediated by oxide availability and surface activity of Fe oxides, which provide anchoring sites for gels and favor fabric densification.

The microstructure to mechanics pathway is evident in the paired strength data. Inner cores consistently attain higher dry UCS and comparable or better wet retention than crusts, commensurate with tighter packing and more pervasive grain-bridging films. Two strengthening routes are implicated: (i) gel–oxide interlocking that builds bonded bridges and refines pore throats, and (ii) densification driven by packing, which reduces connected porosity and increases the number of effective load paths [28], [31], [32], [33]. The crusts, with more open fabrics and fissures, show lower dry and wet strengths, consistent with capillary pathways that accelerate softening under immersion.

An integrative view emerges in which near-surface weathering decreases quartz/Si content, promotes Al-rich phase development (gibbsite/kaolinite), and leaves Fe-oxide nodules localized. Cores therefore harden by assembling denser, better-bonded fabrics, whereas crusts weaken as porosity increases and coatings are lost. Within this framework, the narrow crystallinity range observed across samples ($\approx 71\text{--}73\%$) is informative: it implies that changes in lattice perfection are not the primary control on performance; rather, phase proportions and pore architecture dominate strength and wet-strength retention, consistent with the role of pore-size distributions and throat connectivity in hydric degradation [30]. The regression models reinforce this hierarchy by identifying Fe_2O_3 and $\text{Al}_2\text{O}_3/\text{SiO}_2$ as positive contributors to dry UCS, crystallinity as weakly negative for dry strength but positive for wet retention, and porosity as a negative predictor of wet retention, an ordering that mirrors the chemistry to phases to microstructure to mechanics’ cascade.

4.2 Comparison with literature

The empirical chains observed here accord with classic laterite concepts in which hydrolysis, silica leaching, and Fe/Al re-precipitation drive hardening and cuirasse formation (as framed in the Introduction; see Alexander & Cady, 1963; Gidigas,

1991). Ferricrete phases are widely recognized to control particle contacts and capillary transport, with microscopy often showing Fe-oxide grains as nucleation sites for gels that densify the matrix and improve mechanical and hydric performance [28]. Against this backdrop, the present multi-site, multi-layer dataset adds quantitative, site-resolved links connecting oxide ratios, phase balance, microstructural descriptors, and both dry and wet UCS.

Relative to earthen-material studies focused on unfired or low-temperature lateritic bricks, our results align with reports that a higher “ferricrete index” (hematite/goethite abundance $\times$ massive texture) suppresses capillary connectivity and enhances compressive strength and dimensional stability [22], [23], [34], [35], [36], [37], [38]. In particular, the observed pairing of higher strength with denser inner fabrics and of lower strength with more open crust fabrics matches the hydric context in which naturally cuirassed laterites often show lower water absorption than compositionally similar but weakly cuirassed mixes at equal compaction [39], [40], [41]. While the exact values depend on specimen preparation and curing, the mechanistic direction is consistent: refined pore networks and robust gel–oxide interfaces reduce water uptake and improve retention of strength after immersion.

4.3 Practical implications (design guidance and heritage compatibility)

The composition–microstructure–performance relations distilled from the dataset can be translated into practical guidance for unfired laterite bricks and for conservation interventions. First, oxide ratios matter: the inner-layer windows for Al_2O_3 , SiO_2 , Fe_2O_3 , and TiO_2 provide realistic targets for compatible bricks. In design terms, the $\text{Al}_2\text{O}_3/\text{SiO}_2$ ratio emerges as a primary compositional lever for dry strength, while Fe_2O_3 contributes to interfacial activity and potential gel anchoring [22], [23]. Second, the desired phase balance aims at kaolinite–gibbsite–quartz matrices with minor Fe oxides; excessive quartz depletion is undesirable because it coincides with porous, weak crust fabrics. Third, porosity must be steered toward the lower envelope evidenced by the stronger cores. Process variables, compaction energy, water content near optimum, and controlled curing, should be tuned to refine pore throats and promote continuous Al–Si–rich coatings at grain contacts, replicating the microstructural attributes observed by SEM [42], [43], [44], [45], [46], [47].

For heritage practice, replacement units should match inner-layer chemistry and phase proportions rather than crust compositions, since the latter correlate with lower dry and wet strengths. A minimal acceptance suite is therefore advisable prior to deployment: a simple oxide check (bar/ternary plots from XRF), a phase screen (XRD) to confirm kaolinite–gibbsite–quartz with minor Fe oxides, and basic physical verification (porosity/absorption and UCS, dry and after 24 h immersion). This compact protocol operationalizes the study’s structure–property logic and reduces the risk of hydric incompatibility when integrating new unfired units into existing fabric [48], [49].

4.4 Limitations and future research

Several qualifications are warranted. Outer-layer chemistry is presently available for two sites; extending paired inner–outer coverage to all sites would sharpen the statistical contrast and test the generality of the observed trends. Phase abundances are semi-quantitative; a full Rietveld approach with internal standards would decrease uncertainty in the kaolinite/gibbsite/hematite balance and could refine crystallinity estimates. The regression models, while well-fitted, are based on modest sample numbers; coefficients and p-values should be regarded as preliminary until validated on larger, independent datasets. These constraints temper interpretation without altering the core mechanism, which is robustly supported across techniques.

Future work should therefore expand the paired datasets and add pore-system metrics that capture size distributions and throat connectivity (e.g., image-based measures or mercury intrusion porosimetry) to complement SEM. Durability protocols that interrogate the hydric pathways, sorptivity, cyclic wetting–drying, and freeze–thaw/salt crystallization where climatically relevant, are essential to map performance envelopes against porosity and phase proportions [30]. Finally, controlled trials that vary compaction energy, water content at molding, and curing humidity, and, where appropriate, explore low-dosage organic additions, would clarify processing windows that deliver the observed gains in wet-strength retention while preserving compatibility with the inner-layer chemistry established here.

In sum, the dataset substantiates a mechanistic chain from oxide ratios through phase balance to microstructure and mechanical response. By framing this chain in operational terms, oxide targets, phase checks, porosity envelopes, and a compact verification suite, the study supplies a practical template for both conservation of ancestral lateritic walls and the design of compatible, low-carbon unfired bricks that leverage locally available laterites while mitigating hydric risks [28], [34], [35], [39].

5. Conclusion

This study establishes an experimentally grounded chain from composition to phase assemblage, microstructure, and macroscopic performance for lateritic materials drawn from Madagascar's *Tamboho* walls. Inner cores are chemically richer in alumina with moderate silica and iron, organize into kaolinite–gibbsite–quartz matrices with minor Fe-oxides, and display denser fabrics with more continuous Al–Si-rich coatings. In contrast, weathered crusts show uniformly lower oxide contents, relatively stronger gibbsite signatures, and a more connected pore network. These differences manifest mechanically: cores reach higher dry strengths (1.61–2.10 MPa) than crusts (1.28–1.46 MPa) and retain 49–64% of strength after immersion, while density, porosity, and absorption align with the observed fabric compactness.

Across sites, the narrow crystallinity range suggests that phase proportions and pore architecture, rather than lattice perfection, control performance. Regression results substantiate this hierarchy by identifying Fe_2O_3 and $\text{Al}_2\text{O}_3/\text{SiO}_2$ as positive contributors to dry strength, and crystallinity combined with lower porosity as favorable for wet-strength retention. Together, these relations delineate actionable formulation windows—oxide targets, kaolinite–gibbsite–quartz phase balance with minor Fe-oxides, and lower apparent porosity—along with a minimal acceptance suite (XRF/XRD screens plus porosity/absorption and dry/wet UCS) for both conservation and new unfired bricks.

The contribution lies in translating qualitative laterite models into quantitative, site-resolved structure–property links that can be operationalized in practice. Future work should broaden paired inner–outer datasets, refine phase quantification with full Rietveld methods, and incorporate pore-size distribution metrics and durability protocols (wet–dry, sorptivity, freeze–thaw/salt) to confirm and generalize the proposed design window. In doing so, low-carbon, context-appropriate earthen masonry can be formulated with greater predictability while maintaining compatibility with historic fabric.

6. CRediT authorship contribution statement

Solofo Roger Rakotoarisoa: Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Methodology, Formal analysis, Data curation, Conceptualization. **Herry Purnama:** Writing – review & editing, Supervision, Validation. **Rijalalaina Rakotosaona, Ahmad M Fuadi:** Supervision, Validation.

7. Disclosure Statement

No potential conflict of interest was reported by the authors

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