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Critical review and methodological framework

Accelerated ageing protocols for mortars incorporating agricultural and agro-industrial organic residues: degradation mechanisms, testing standards, and a harmonized durability assessment framework

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Abstract

Agricultural and agro-industrial residues are increasingly introduced into building mortars as lightweight fillers, fibrous reinforcements, or binder-replacement materials. Their effects on density, strength, water transport, and thermal performance have been widely explored, whereas durability assessment remains difficult to compare because residue composition, binder chemistry, curing, accelerated exposure and damage indicators vary substantially among studies. This article critically examines ageing mechanisms relevant to gypsum-, lime-, and cement-based mortars containing untreated or processed organic residues and distinguishes these materials from mortars containing biomass-derived ashes. A focused evidence review of recent experimental investigations and technical recommendations is combined with a scope-based analysis of existing EN, ISO, ASTM, and RILEM methods. Water-mediated deterioration, fibre–matrix interfacial changes, soluble-organic leaching, temperature and moisture cycling, salt crystallisation, freeze–thaw damage and biological susceptibility are considered according to exposure relevance rather than as interchangeable durability tests. On this basis, a harmonised, application-specific testing framework is proposed. It combines material traceability, matched unaged controls, non-destructive monitoring, staged single-stressor screening, conditional multi-stressor exposure, post-ageing mechanical and hygrothermal characterisation, and hierarchical statistical analysis. Explicit guidance is provided for selecting exposure modules and interpreting retained performance without confusing accelerated laboratory cycles with a validated service-life prediction. This work does not report newly generated experimental results or claim validation of the suggested cycles. Its contribution is a reproducible methodological framework for future interlaboratory verification and for comparing organic-residue mortars under clearly defined conditions.

Keywords
accelerated ageing; organic residues; agro-industrial waste; gypsum plaster; lime mortar; cement mortar; hygrothermal cycling; durability; circular construction; test harmonization

1. Introduction

Reducing the consumption of virgin aggregates and developing building materials that make use of locally available residual biomass are established objectives in circular construction. Orange and other citrus peels, pistachio shells, olive-processing residues, spent coffee grounds and crop fibres have been considered as mortar constituents, while rice-husk and olive-pomace ashes have been investigated as reactive or filler-type mineral components. These interventions can affect density, thermal conductivity, workability, water demand and mechanical response, although their benefits are conditional on particle processing, dosage and binder chemistry [1–8]. The central limitation for practical application is not merely the absence of adequate initial properties. A mortar must preserve the functions required by its intended exposure class throughout a meaningful part of its service life.

A recent investigation of gypsum plasters containing pistachio shells and orange peels reported changes in physical, mechanical and water-absorption properties, illustrating the potential of agricultural by-products in internal finishing systems [1]. A 2026 investigation has also reported on waste-modified gypsum binders [36]. Work on cement mortars containing spent coffee grounds showed that dosage and curing time strongly influenced strength, so an initially favourable formulation could not be identified from residue identity alone [2]. More recently, powdered olive stone was examined as an aggregate substitute in lime-based restoration mortars [3], and olive pomace ash was assessed as a cement replacement [4]. These studies do not examine interchangeable material classes: raw organic particles, lignocellulosic fibres and combustion ashes differ in chemical reactivity, water affinity and degradation pathways.

The ageing literature presents a second source of heterogeneity. Coating-mortar reviews identify capillary absorption, repeated wetting and drying, shrinkage, frost action and aggressive-ion exposure as common durability indicators, but test severity and specimen conditioning are frequently not harmonised [9,10]. A hemp-based cementitious mortar study employed a total-weathering approach derived from external thermal insulation composite systems (ETICS), while research on lime mortar highlighted differences in damage induced by conventional and newly developed salt-weathering procedures [11,12]. Consequently, performance reported after a given number of cycles cannot be compared without examining cycle duration, moisture state, temperature gradient and the end-point definition.

The aim of this article is to establish a technically defensible framework for evaluating the ageing of mortars containing agricultural and agro-industrial residues. It addresses four questions: which residue–binder combinations require distinct degradation hypotheses; which existing test standards can be used directly and which demand adaptation; how can accelerated exposures be organised without imposing physically implausible stress on the material; and what measurements allow between-formulation comparisons while acknowledging uncertainty? The outcome is a proposed laboratory framework, not a new certified standard or a validated predictive model.

2. Review design and evidentiary boundaries

2.1. Focused evidence assessment

This article was prepared as a critical, method-oriented narrative review rather than a systematic review or a quantitative meta-analysis. Source identification was undertaken in October 2026 through publicly accessible journal and publisher records, university repositories, standards

catalogues, the RILEM publication archive and targeted bibliographic searching. Search expressions combined terms such as “agricultural waste mortar ageing”, “bio-based plaster durability”, “hemp mortar wet dry cycling”, “gypsum orange peel”, “olive pomace ash mortar”, “salt crystallisation RILEM”, and the identifiers of relevant EN, ISO and ASTM methods. Recent peer-reviewed work, predominantly from 2021–2026, was prioritised, while older normative documents were retained where they continue to define test procedures.

Sources were considered pertinent if they described (i) raw or processed agro-residues in cementitious or non-cementitious mortar, (ii) relevant ageing mechanisms or accelerated exposure, (iii) characterisation of water transfer, mechanical deterioration or hygrothermal response, or (iv) published procedures applicable by direct scope or reasoned adaptation. Concrete-only studies and research on biomass-derived ashes were used to explain particular mechanisms, but were not treated as direct evidence for raw organic-fillers in plaster. No complete database export, duplicate-removal log, full-text eligibility register or PRISMA screening count was generated. Accordingly, no claim of exhaustive coverage or systematic evidence synthesis is made.

2.2. Evidence-to-protocol translation

Three levels of evidence are distinguished throughout the paper. “Normative method” denotes a procedure covered by a published standard within its stated application scope. “Transferred method” denotes a standard or published technical recommendation developed for another porous material or product and therefore requiring a declared adaptation and validation study. “Proposed condition” denotes a transparent laboratory setting introduced here for comparative screening and not yet experimentally calibrated. This distinction is essential for freeze–thaw protocols originally developed for concrete or natural stone, and for biological challenge methods originally developed for coatings or plastics [13–17].

A transfer is considered methodologically justified only when the damage mechanism, relevant moisture state, specimen geometry and end-point indicators remain interpretable. The number of laboratory cycles is reported as accumulated exposure, not as a number of equivalent field years. Service-life extrapolation requires field calibration or an independently established damage kinetics model.

3. Residue typology, binder chemistry and functional application

3.1. Unburnt plant particles and powder additions

Fruit peels, nutshells, olive stones and spent coffee grounds typically contain varying proportions of cellulose, hemicellulose, lignin, extractives and water-soluble organic substances. Their incorporation as discrete particles can lower composite density and modify pore connectivity, while their affinity for water may change the effective mixing-water availability and moisture distribution during ageing. Fruit-peel pectin and soluble sugars may also alter setting and binder interactions; however, the net effect depends on extraction, drying, particle size and dosage and cannot be presumed identical across residues [1,2,5]. Raw organics may be more sensitive than inert mineral grains to leaching, swelling, biological colonisation and surface degradation.

Residue preparation must therefore be regarded as part of the material definition. Air drying, low-temperature oven drying, washing, grinding and sieving are not neutral pre-treatments: they can change available extractives, initial moisture and particle morphology. The same label, such as “olive waste”, may refer to wet pomace, dried and milled pomace, ground olive stone, or incinerated ash. These forms require distinct test identifiers and mass-balance boundaries [3,4,6].

3.2. Lignocellulosic reinforcement

Long or short vegetable fibres may bridge microcracks and modify post-peak behaviour. In alkaline matrices, however, natural fibres may undergo chemical alteration, mineral deposition within their structure, loss of flexibility and weakening of the fibre–matrix interface. Water cycling can additionally induce dimensional changes and local debonding. Recent investigations and reviews of hemp and other plant fibres demonstrate that binder composition and pretreatment affect the response to wetting–drying cycles [11,18–20]. Consequently, flexural strength alone is insufficient to describe fibre-reinforced mortars. Fracture response, residual toughness, crack pattern and interfacial morphology should also be considered when these are central to the proposed function.

3.3. Biomass-derived ash: a distinct comparison class

Combustion ashes obtained from rice husks, olive pomace and other residues largely lack the original organic structure and may act as porous fillers or supplementary cementitious constituents, depending on thermal history, fineness and chemical composition. Their relevant durability processes therefore include reaction-product evolution, pore refinement, ion ingress and binder chemistry rather than the same biological or swelling mechanisms experienced by unburnt plant particles. Reports on rice-husk ash and olive pomace ash indicate that effects on mechanical and transport properties are dosage-dependent [4,7,8]. Ash-bearing mortars can be included as a separate benchmark, but they should not be pooled with raw organic-containing materials in a single unstratified durability ranking.

3.4. Binder and use-context dependence

Gypsum plaster is primarily intended for dry internal conditions. Moisture exposure can affect the gypsum matrix independently of the organic addition; prolonged water immersion or freeze–thaw testing may therefore test an implausible or prohibited application rather than intrinsic material quality. Air-lime mortars depend on carbonation and may continue changing during testing; natural hydraulic lime and cement-based mortars involve different proportions of hydraulic reaction and pore solution alkalinity. Restoration mortars also require compatibility with historic substrates in terms of stiffness, water-vapour transport and salt behaviour [21,22]. Exterior exposure modules should therefore be applied only to formulations designed and specified for exterior use.

Table 1. Material classes, relevant ageing hypotheses and exposure priorities.

Material class	Illustrative source or form	Main ageing hypothesis	High-priority tests	Critical caveat
Gypsum + unburnt powder	Citrus peel, pistachio shell, spent coffee grounds	Moisture uptake, extractive migration, matrix–particle debonding, biological susceptibility	RH cycling, capillary uptake, vapour transfer, strength retention, surface inspection	Avoid immersion or frost exposure unless a justified use case exists
Air-lime + particles	Powdered olive stone, plant granules	Moisture-related dimensional mismatch; carbonation-dependent property evolution	Wetting–drying (if exterior), adhesion, capillary uptake, carbonation, salt screening where relevant	Monitor carbonation and substrate compatibility
Hydraulic lime/cement + fibres	Hemp or straw fibres	Alkali exposure, swelling–shrinkage, fibre/interface deterioration	Wetting–drying, crack mapping, residual flexure/toughness, microscopy	Report binder pH and pretreatment
Cement + unburnt fine residues	Spent coffee grounds, milled organic waste	Altered hydration and water partition; interfacial changes	Water transport, strength development, wetting–drying, exposure-specific chemistry	Equal workability and equal water/binder are different controls
Mineral ash comparator	Rice-husk ash, olive-pomace ash	Pozzolanic/filler effects, carbonation, salt or frost susceptibility	Standard cement-mortar durability suite selected by use	Not a proxy for biodegradable organic inclusions

4. Mechanisms governing ageing of agro-residue mortars

4.1. Moisture transport, swelling and microcracking

Liquid-water uptake and water-vapour exchange depend on the pore network of the binder, interfacial transition zones and the sorptive properties of the inclusion. Adding a light and absorbent particle may raise total porosity but does not necessarily increase capillary transport: pore connectivity, internal curing and binder reactions can act in opposite directions. It is therefore necessary to separate total water uptake, capillary absorption coefficient, sorption isotherm and vapour resistance factor rather than using a single “water resistance” endpoint [7,9,10]. Cyclic variation in moisture content can impose strain mismatch between hygroscopic particles and a comparatively brittle mineral matrix, potentially leading to interface separation and higher transport rates after ageing.

A physically meaningful accelerated test should specify whether a specimen is exposed to liquid water, high relative humidity or alternating vapour conditions. These exposures cannot be assumed equivalent, particularly for gypsum-based plasters. After each conditioning episode, specimens should be equilibrated to a defined reference state before comparing mass and mechanical measurements. Otherwise, differences in transient water content can be incorrectly interpreted as permanent material loss or ageing-induced property change.

4.2. Chemical interactions and soluble fraction loss

Water-soluble sugars, salts, organic acids or extractives can enter the pore solution and modify setting or chemical stability. In highly alkaline systems, plant-polymer hydrolysis and compositional changes may affect reinforcement durability [19,20]. In calcium sulphate systems, the primary durability concern may instead involve moisture sensitivity of both matrix and inclusion. A proposed diagnostic approach is to measure extract pH and conductivity, soluble organic carbon when feasible, and major ionic species before and after water-mediated exposure. A shift in these indicators alone does not prove harmful degradation; chemical evidence should be combined with microstructural and functional changes.

4.3. Frost damage and critical moisture state

Freezing and thawing can damage water-filled porous networks when internal stress exceeds local strength or strain accommodation capacity. The intensity of the process depends on moisture saturation, cooling schedule, pore structure and sample dimensions. The standard ASTM C666/C666M-26 specifies resistance testing for concrete, while EN 12371:2010 is intended for natural stone [13,14]. Neither establishes an automatically transferable pass/fail threshold for a gypsum or lime mortar containing organic inclusions. Frost trials must be restricted to relevant applications and be preceded by evidence that the selected preconditioning reproduces a credible in-service moisture state.

4.4. Salt crystallisation and exposure realism

Salts may crystallise within a porous finishing layer during transport and evaporation, inducing surface scaling, local fracture or adhesion loss. EN 12370:2020 addresses natural stone. The RILEM TC 271-ASC recommendation instead differentiates a salt-accumulation stage from subsequent damage propagation, aiming to preserve the characteristic transport-and-evaporation mechanism; its reported laboratory verification concerns stone and fired-clay brick, not all mortars [15,16]. A comparison of different ageing methods for lime mortars showed that test choice can change apparent aggressiveness and interpretation of treatment performance [12]. Transfer to agro-residue mortars should therefore begin with small-scale feasibility testing and transparent deviations from the original recommendation.

4.5. Thermal stress, solar exposure and long-term functional drift

Thermal cycling can contribute to cracking through differences in expansion and modulus between binder and organic inclusion. Photochemical alteration of exposed organic matter may also be relevant for external renders, but UV exposure should not be imposed on internal plasters merely because accelerated weathering equipment is available. For insulating mortars, the test endpoint must include thermal conductivity at a stated moisture content: a change in conductivity between damp aged and dry unaged samples is not a valid measure of permanent material deterioration. Research on hemp-based and other waste-modified insulating mortars already illustrates the value of assessing microstructure and hygrothermal properties before and after weathering [10,11,23].

4.6. Biological colonisation

Biological growth depends on accessible nutrients, surface pH, available water, temperature and exposure duration. The presence of organic biomass does not, by itself, establish fungal growth under typical service conditions. Nevertheless, bio-based residues can change surface nutrient availability and moisture retention. ASTM D3273-21 concerns mould growth on interior coatings, and ISO 846:2019 concerns microbial effects on plastics; both may inform control logic, but neither should be declared a standard microbial durability test for mortar [17,24]. For mortars, biological susceptibility is better evaluated through a separately described research protocol with appropriate controls, microbiological competence and field-relevant relative humidity.

5. Standardised methods: applicability and gaps

The assessment of durability requires combining product-specific strength and adhesion standards with hygrothermal characterisation and, where justified, ageing methods adapted from adjacent fields. EN 13279-2:2014 addresses gypsum binders and gypsum plasters; EN 1015-11:2019 covers flexural and compressive strength of moulded mortar specimens; EN 1015-12:2016 deals with adhesive strength of renders and plasters on substrates; EN 1015-18:2002 concerns capillary water absorption of rendering mortars with mineral binders; and EN 1015-19:1998 with Amendment A1:2004 addresses water-vapour permeability of rendering and plastering mortars [25–29]. For broader building products, EN ISO 12571:2021 and EN ISO 12572:2016+A1:2024 respectively inform hygroscopic sorption and water-vapour transmission tests [30,31].

Product classification cannot be detached from scope. EN 998-1:2016 covers defined rendering and plastering mortars, whereas gypsum products are governed by the EN 13279 series [25,32]. Equally, the requirements for a thin internal finish differ from those of an external façade system or a conservation mortar. The 2024 edition of EAD 040083-01-0404 provides a relevant context for external thermal insulation composite systems with renderings, but its system-level acceptance criteria must not be assigned automatically to free-standing laboratory mortar prisms [33].

Table 2. Normative and technical references, with limits of transfer.

Test or property	Reference	Original scope	Suggested use in agro-residue mortars	Status
Gypsum plaster physical/mechanical tests	EN 13279-2:2014	Gypsum binders/plasters	Binder-specific baseline and residual properties	Direct, where product class agrees
Flexural and compressive strength	EN 1015-11:2019	Moulded mortar specimens	Matched strength at baseline and destructive endpoints	Direct, when specimen requirements are met
Adhesion to substrate	EN 1015-12:2016	Rendering/plastering mortar on substrate	Bond strength and failure mode after exposure	Direct within scope

Test or property	Reference	Original scope	Suggested use in agro-residue mortars	Status
Capillary water absorption	EN 1015-18:2002	Mineral-binder rendering mortars	Baseline/post-ageing water uptake	Direct or declared adaptation; 2026 revision remains a draft
Vapour permeability	EN 1015-19:1998+A1:2004; EN ISO 12572	Render/plaster; porous building products	Vapour transfer before and after ageing	Select test by product and report conditions
Sorption isotherm	EN ISO 12571:2021	Porous building products	Moisture-dependent functional response	Direct within scope
Salt crystallisation	EN 12370:2020; RILEM TC 271-ASC (2023)	Natural stone; stone and fired-clay brick	Exploratory transfer to exterior mortars	Adaptation requiring verification
Freeze–thaw	EN 12371:2010; ASTM C666/C666M-26	Natural stone; concrete	Climate- and exposure-specific mortar screening	Adaptation requiring verification
Fungal resistance	ASTM D3273-21; ISO 846:2019	Interior coatings; plastics	Inspiration for dedicated experimental controls	Not a direct mortar standard
External render system weathering	EAD 040083-01-0404	ETICS with renderings	Reference for relevant system-level exposure	System-level; not universally transferable

An additional standards-status consideration is important at the time of writing. A prEN 1015-18:2026 revision is recorded in current catalogues, but a draft is not equivalent to an adopted normative edition. This article therefore refers to the published EN 1015-18:2002 method unless the competent standards body has formally replaced it [27].

6. Proposed harmonised assessment framework (HAAM)

6.1. Design principle and intended use

The proposed Harmonised Agro-residue Ageing of Mortars (HAAM) framework is a modular research procedure designed to support reproducible comparisons, not an EN, ISO, ASTM or RILEM standard. It proceeds from product identification and exposure classification to non-destructive observation, controlled ageing and post-exposure characterisation. Its central principle is that stressors should be selected according to anticipated building exposure and material-specific deterioration mechanisms, rather than by the desire to expose all formulations to an identical exhaustive series of aggressive conditions.

Each tested formulation is assigned four identifiers: binder family (G, L, HL or C for gypsum, air lime, hydraulic lime or cement), residue state (U for unburnt, F for fibrous or A for ash), intended service exposure (I for protected internal, E for external, H for heritage repair), and processing history. The coding prevents pooling of physically different materials under the same generic residue name.

6.2. Raw-material traceability and conditioning

For each residue batch, the laboratory shall record crop or industrial source, collection date, known treatment agents, initial moisture content, drying temperature and duration, crushing or milling procedure, sieve fraction or fibre-length distribution, bulk and particle density, and any washing or chemical modification. Where analytical capacity allows, ash content, loss on ignition, soluble fraction, extract pH and conductivity, particle morphology and relevant chemical functional groups should be determined. For fibrous residues, fibre length and aspect ratio must be retained because they influence both mixing and fracture performance.

Mix compositions must be expressed by a clearly stated replacement basis, distinguishing percentage of binder mass, aggregate mass and aggregate volume. Volumetric replacement is generally more interpretable when replacing mineral sand with low-density organic particles; mass-based additions may be more appropriate

for fine powders used as admixtures. Both dry-mass and bulk-volume data should be reported to support re-analysis. Mixing energy, sequence of addition, fibre dispersion, target consistency and actual water-to-binder ratio are critical. If mixes are adjusted to a common workability, the resulting water ratios should be stated; if water ratio is held constant, differences in workability should not be concealed.

All samples need a matched residue-free control produced with the same binder family, comparable target application and documented mixing and curing procedure. A separate age-matched unexposed set is required to distinguish damage from continued cement hydration, lime carbonation and other maturation processes.

6.3. Staged experimental design and allocation

An economical two-stage design is recommended. Stage A screens the combinations of residue type, pretreatment and dosage using baseline measurements and a single dominant exposure. Stage B tests the most informative formulations under replicated exposure modules and selected coupled stressors. Suggested screening levels, such as 0%, 5% and 10% aggregate replacement by volume for particulate fillers, or 0%, 1% and 3% binder-mass addition for fine powders, are experimental starting points rather than validated optimal dosages. They must be adjusted to the material's workability and application constraints. These bases must never be combined in the same variable without conversion.

At least three independent mixing batches per formulation are recommended for confirmatory inference, with multiple specimens taken from each batch. Destructive measurements should normally target a minimum of six test specimens per formulation–exposure–time condition, subject to a pilot-based power calculation and the requirements of the selected standard. Independent batches, not individual test prisms cast from one batch, represent the fundamental production replicates. Specimen geometry follows the applicable mechanical test standard; separate dimensions may be required for thermal tests, capillary transport, adhesion and microscopy. A random allocation list should distribute specimens across exposure positions to avoid chamber location confounding.

6.4. Baseline measurements and monitoring points

Before accelerated exposure, record dry-equilibrated mass, bulk density, visible defects, dimensions and colour or surface reflectance where relevant. Measure initial mechanical strength, dynamic modulus or ultrasonic pulse velocity when validated for the specimen, capillary absorption, hygroscopic sorption, vapour permeability, thermal conductivity, and bond strength when a substrate is used. Not all tests are mandatory for every formulation; a core common set and an application-specific set should be distinguished. For thermal conductivity, report equilibrium temperature and relative humidity as well as the specimen's moisture conditioning.

Recommended observation checkpoints are baseline (T0), an intermediate checkpoint after approximately half the intended exposure (T1), and final exposure (T2). Non-destructive measurements can follow the same specimens, whereas strength and microstructural measurements may require reserved destructive subgroups at each checkpoint. Exposed and control samples must be equilibrated under identical conditions before comparisons of permanent damage. Visible grading is supported by fixed-geometry photographs, a reference scale and pre-defined definitions of cracking, spalling, staining and efflorescence.

6.5. Exposure modules

A controlled high-humidity module (H) is the recommended initial ageing screen for most unburnt organic-containing internal plasters. A preliminary, explicitly non-standard programme may alternate 12 h at 90% relative humidity and 30 °C with 12 h at 50% relative humidity and 30 °C, for 30 or 60 cycles. These values are proposed test settings, not field-calibrated severity levels. Chamber logging should document the actual

conditions and the time required for samples to approach moisture equilibrium. Gypsum plaster should normally be assessed with vapour rather than prolonged liquid immersion unless a credible moisture-abuse scenario is the stated aim.

A liquid wetting–drying module (W) can be assigned to exterior hydraulic or lime mortars, subject to water sensitivity. As a provisional comparative programme, 8 h controlled partial immersion or capillary contact may be followed by 16 h drainage and drying at a temperature not exceeding 40 °C, for a pilot-defined number of cycles. Complete immersion, intermittent spray and capillary contact are distinct boundary conditions, and the selected geometry, solution chemistry and exposed faces must be reported. Because drying at elevated temperature may alter plant particles and lime carbonation, a low-severity pilot should confirm that the intended transport mechanism predominates over unintended thermal artefacts.

A thermal-cycle module (T) may alternate 10 and 45 °C under controlled moderate relative humidity for 30 cycles where service exposure justifies it. This module should be applied independently of the humidity module before combined testing, enabling the effect of temperature alone to be examined. UV exposure is relevant only where organic residue is directly exposed to sunlight at the finished surface and should be separately quantified by spectral distribution and incident radiant dose, not merely the number of hours in a lamp chamber.

The frost module (F) is optional and relevant only for exterior mortars in climates where critical moisture and freezing can coexist. A screening programme might use controlled cooling to approximately −15 °C and warming to approximately +20 °C with 25 and 50 checkpoints, but cooling rate, specimen core temperature, moisture conditioning and thawing medium require pilot confirmation. These values are proposed adaptation parameters, not instructions reproduced from EN 12371 or ASTM C666/C666M [13,14]. Plasters intended exclusively for protected indoor service should not be rejected because they perform poorly in this non-representative module.

The salt module (S) is reserved for applications where salt transport is expected, including marine-exposed or salt-contaminated masonry. A feasibility phase may take inspiration from the RILEM TC 271-ASC accumulation-and-propagation concept, but adaptations for mortar surface roughness, binder chemistry, capillarity, efflorescence cleaning and specimen geometry must be documented [15,16]. Sodium chloride and sodium sulphate should not be mixed into an undifferentiated test without a justified environmental scenario. Soluble salts and their residual mass must be distinguished from true loss of matrix material.

A biological susceptibility module (B) can be added for internal or external organic-rich renders that are exposed to sustained high humidity. It should be carried out in an adequately equipped microbiology facility with pre-registered organisms or a defined naturally acquired exposure, uninoculated controls and clear contamination-control procedures. Outcomes may include surface coverage, microscopy and changes in relevant material properties. Elevated humidity alone, without organism identification and adequate controls, does not establish biodegradation.

Table 3. Illustrative HAAM modules and the minimum interpretation safeguards. Values are proposals, not validated service-life equivalents.

Module	Provisional exposure	Suggested checkpoints	Relevant materials	Minimum safeguard
H – relative-humidity cycling	12 h at 90% RH/30 °C; 12 h at 50% RH/30 °C	0, 30, 60 cycles	Internal gypsum; all unburnt residue systems	Record sample moisture equilibration and age-matched controls
W – liquid wetting/drying	8 h defined capillary contact; 16 h drying ≤40 °C	Pilot-defined; e.g., 0, 15, 30	Exterior lime, hydraulic lime, cement	Specify water chemistry, wet area, drying endpoint

Module	Provisional exposure	Suggested checkpoints	Relevant materials	Minimum safeguard
T – thermal cycling	10–45 °C under controlled RH	0, 15, 30 cycles	Temperature-exposed renders	Measure actual specimen rather than air temperature
F – frost cycling	About −15 to +20 °C, moisture-conditioned	0, 25, 50 cycles	Suitable exterior materials only	Pilot-validated saturation, rate and failure mode
S – salt accumulation/propagation	RILEM-informed, salt-specific protocol	Stage boundaries and mass/visual end points	Salt-exposed exterior or heritage mortars	Validate transfer from stone/brick; quantify retained salt
B – microbiological susceptibility	Defined humid controlled challenge	Baseline, intermediate, end	Organic-rich renders with credible moisture exposure	Dedicated microbiological controls and biosafety
C – selected coupled exposures	H→T, W→S or W→F, according to use	After each stage and at end	Only where combinations are realistic	Include both single-stressor controls; test sequence effects

6.6. Coupled exposures and order effects

Sequential exposure may be necessary because moisture conditioning can increase subsequent sensitivity to salt or frost action. Nevertheless, coupling should not replace the initial single-stressor comparison. A 2 × 2 design with unaged controls, stressor A alone, stressor B alone and A followed by B is the minimum arrangement required to explore non-additive effects; a reversed B-followed-by-A group may be added to identify sequence dependence. A significant interaction is not inferred simply because the combined group exhibits more damage than either isolated group. The expected additive response must be defined on an appropriate response scale and evaluated statistically.

For internal gypsum-based formulations, a humidity–temperature sequence is usually more pertinent than combining full immersion, salt saturation and freezing. For exterior lime or hydraulic mortars on contaminated substrates, an initially mild capillary salt loading followed by controlled humidity cycling may be more plausible. The purpose of the coupled-exposure module is to reproduce credible causal pathways, not to maximise destruction within the shortest possible laboratory time.

6.7. Suggested end-of-test rules

A module should be stopped according to an a priori rule where one of the following applies: specimen integrity no longer permits valid measurement; a safety or equipment limit is exceeded; a clearly documented application-specific functional limit is reached; or the complete planned number of cycles has been performed. Absolute strength or mass-loss pass/fail limits should not be invented for materials outside the applicable specification. Where a regulatory requirement or agreed project performance threshold exists, it should be stated separately from a research-based relative ranking.

7. Metrics, uncertainty and statistical inference

7.1. Residual performance indices

For a performance indicator X for which higher values represent improved function, the age- and exposure-matched retention ratio can be defined as

$$R_X(t) = 100 \times X_{aged}(t) / X_{unexposed}(t) . \quad (1)$$

Here, X_unexposed(t) is measured on the same formulation maintained without aggressive exposure to the same chronological age, rather than exclusively at the initial curing age. This correction matters for cement hydration and lime carbonation. For properties where lower values are preferable, such as water uptake in some applications, the raw aged/unaged ratio should be reported and discussed with its appropriate directionality rather than being called “retention”. A reduction in water absorption may be beneficial in some systems but could reflect harmful pore blocking in a heritage mortar that requires drying capacity. All ratios should be accompanied by absolute measurements and dispersion.

An exposure-specific relative effect of the residue can be expressed as a difference in logarithmic changes:

$$D_X = \ln[X_{add,aged}/X_{add,control}] - \ln[X_{ref,aged}/X_{ref,control}] . \quad (2)$$

This difference-in-differences formulation isolates the exposure-related proportional change in an amended mix relative to the analogous change in the residue-free mortar, provided measurements are positive, comparable and age-matched. It does not remove all confounding by differing porosity or moisture conditioning and must not be interpreted as a service-life prediction.

Apparent dry-mass change is expressed as

$$\Delta m_{dry}(t) = 100 \times [m_{dry}(t) - m_{dry}(0)] / m_{dry}(0) . \quad (3)$$

For salt-tested samples, this value may combine salt uptake, efflorescence and genuine loss of mortar; separate mass-balance observations or careful analytical correction are required before describing it as erosion. Uncontrolled variations in residual water must likewise be excluded.

A normalised crack-density metric may be expressed as total trace length per observed surface area (mm/mm²), while surface-area fraction affected by spalling or fungal coverage can be estimated from photographs using a locked segmentation procedure. Where fibre bridging is functionally relevant, residual flexural toughness or a crack-mouth-opening response can complement peak flexural strength. Thermal conductivity and water-vapour resistance are reported at matched hygrothermal states to avoid a false ageing effect.

7.2. Data hierarchy and reproducibility

The experimental design is inherently hierarchical: material batches contain specimens, and specimens may be measured repeatedly over time. For a continuous response Y, an appropriate model may include fixed effects of formulation, exposure, checkpoint and their interactions, a random batch intercept and, when justified, a specimen-level repeated measure structure. The model should be selected according to residual diagnostics and the number of independent batches. If the number of batches is too small to estimate variance components reliably, the analysis should emphasise batch-level summaries and confidence intervals rather than using all specimens as independent replicates.

The primary endpoint should be pre-specified for each material class, for example adhesion for bonded external renders, flexural retention for fibre-reinforced mortars, or moisture-conditioned thermal conductivity for internal thermal plasters. Multiple secondary endpoints may be reported, but exploratory multiplicity should be recognised. Confidence intervals and effect sizes are more informative than rankings based on isolated sample means. Missing observations caused by specimen disintegration are outcomes of the exposure, not innocuous missing data; their treatment must be disclosed.

7.3. Functional trade-offs and decision rules

A single weighted durability score would conceal whether a sample fails by moisture transfer, adhesion loss or microbial colonisation. In the first validation campaign, a vector of indicators is therefore preferred: mechanical retention, bond response, capillary uptake, vapour permeability, thermal property drift, visible damage and uncertainty. Formulations can be considered non-dominated only within a stated intended use and set of admissibility constraints. If a multi-criteria decision analysis is subsequently undertaken, threshold selection, indicator normalisation, weighting and sensitivity analysis must be declared; weights should not be presented as empirically estimated unless stakeholder or experimental evidence supports them.

8. Interpretation of currently available evidence

8.1. Initial properties do not establish durability

Evidence from gypsum plasters containing orange peels and pistachio shells demonstrates how agricultural residues may affect initial physical and

mechanical characteristics [1]. Studies of spent coffee grounds in cement mortar and plaster similarly highlight the dependence of results on dosage and processing [2,5]. None of these initial-property results is by itself a quantitative estimate of the change in performance after repeated hygrothermal or environmental ageing. An experimental article reporting reduced initial water absorption cannot therefore be cited as proof of reduced long-term moisture-induced damage without appropriate exposure tests.

8.2. Fibre ageing depends on the matrix and the stressor

The available hemp-based mortar literature demonstrates a more direct connection between accelerated exposure and material transformation. Studies of weathered hemp cementitious mortars considered not only strength but also hygrothermal and microstructural evolution [11], while 2026 research on hemp fibre composites evaluated the effect of wet–dry ageing and binder composition [18]. The reported outcome should not be extrapolated to citrus powders or nutshell aggregate systems. The fibre–matrix interface and the role of alkaline solution are distinctive mechanisms, whereas plant powders can primarily act as porous inclusions or modifiers of the fresh mix.

8.3. Severity is a property of the protocol as well as the material

The RILEM TC 271-ASC programme distinguishes salt accumulation from propagation and offers a mechanism-based alternative to repetitive high-concentration immersion-type weathering [15,16,37]. Gabrielli and colleagues compared salt and freeze–thaw procedures on lime mortars and found that the selected procedure influenced the observed damage and the interpretation of consolidant performance [12]. These results support the need for protocol descriptions that include salt concentration, specimen geometry, evaporation boundary, cycling regime and criteria for material loss. They do not imply that the RILEM procedure has already been validated on every type of agricultural-residue mortar.

8.4. Circularity and performance must be assessed on the same functional basis

Waste diversion alone is not sufficient to claim environmental superiority. The conversion of a residue into a mortar component may require drying, grinding, washing, sterilisation or thermal treatment; lower density may also be accompanied by higher binder consumption, shorter maintenance intervals or unacceptable exposure-related replacement. Under ISO 14040 and EN 15804 principles, a comparative environmental assessment requires an explicit functional unit, system boundaries and transparent allocation of upstream burdens [34,35]. For a render or plaster, a service-based functional unit—such as a defined square metre at a specified thickness and performance level over a declared period—can be more meaningful than comparing one kilogram of material. Because no harmonised life-cycle inventory or field-validated service lives are presented here, this paper does not claim quantified carbon savings or a verified environmentally optimal formulation.

9. Research agenda for protocol validation

The first validation phase should examine repeatability within a laboratory, using reference mortars spanning low and high water absorption, one internal gypsum formulation containing unburnt organic powder, and one exterior-compatible hydraulic or lime formulation containing a lignocellulosic inclusion. A preliminary study should establish whether selected humidity, liquid-water and temperature cycles create interpretable differences without generating mechanisms absent from field observations. Repeatability coefficients, measurement uncertainty and chamber-position variability should be reported.

The second phase should involve at least two independent laboratories, shared reference residues and defined preparation specifications.

Reproducibility should be analysed independently for mixing, ageing and end-point measurement because interlaboratory variation may arise from any of these stages. A successful protocol should discriminate between formulations in a consistent direction under comparable exposures while preserving plausible damage mechanisms. Stronger damage is not evidence of a better accelerated test unless the resulting deterioration is relevant to real service conditions.

Field-linked panels are the essential third phase. Panels exposed under recorded orientation, rainfall, wetting time, surface temperature, humidity and maintenance regime would enable comparison between laboratory damage indicators and actual weathering. An acceleration factor can be inferred only after the same observable mechanism and end-point are tracked across laboratory and field conditions. Short laboratory tests should not be assigned nominal equivalence to five, ten or twenty years of service without this validation.

10. Limitations

This manuscript is a focused critical review and a protocol proposal. It does not constitute a PRISMA-compliant systematic review, an experimental validation study or a certified test standard. The literature was not exhaustively screened through subscription bibliographic databases, and a pooled effect estimate was not calculated because materials, exposures and reported endpoints are insufficiently homogeneous in the reviewed examples. The provisional numerical conditions in Table 3 are intended to be evaluated through pilot work and may be inappropriate for particular gypsum formulations, heritage substrates or climatic exposures. Some referenced standards apply directly only to concrete, natural stone, plastics, coatings or full façade systems; their transfer to mortar is explicitly conditional. Finally, the framework does not supply universal acceptance limits or service-life predictions, which require a specified product class, exposure class and validated performance criteria.

11. Conclusions

The durability of mortar containing agricultural or agro-industrial residues depends jointly on the residual material's processing history, the binder chemistry, the intended application and the environmental exposure. Unburnt plant inclusions and fibres introduce potentially important water-mediated, interfacial and biological processes, whereas biomass ashes mainly modify mineral-binder reactions and pore structure. Pooling these materials into a single durability category conceals mechanisms that determine test selection.

Published mortar and plaster testing standards provide a basis for mechanical and water-transport characterisation, but several accelerated ageing methods routinely cited in the field were originally developed for different materials or whole façade systems. Direct adoption of their exposure parameters or acceptance thresholds without validation can therefore yield technically misleading comparisons. In particular, the suitability of full immersion, salt crystallisation or freeze–thaw depends on whether the tested mortar is actually intended for that environment.

The HAAM framework developed in this article structures the assessment around traceable residues, matched controls, age-corrected residual properties, progressive single-stressor trials, justified coupled exposures and batch-aware statistical analysis. Its proposed exposure settings must be tested for repeatability, mechanism fidelity and field correspondence before normative or predictive use. The immediate technical outcome is a reproducible experimental design that can support comparative durability studies while avoiding unsupported claims about long-term performance.

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