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Upcycling of Spent Licorice Root Fibers as Bio-Based Reinforcement in Sustainable Cementitious Plasters: Experimental Assessment of Mechanical and Biological Performance

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Abstract

This study investigates the development and performance assessment of an innovative cement-based bio-plaster incorporating fibers recovered from spent licorice roots (Glycyrrhiza glabra), an agro-industrial by-product generated during boiling and extraction processes in the confectionery and pharmaceutical industries. The research explores an upcycling strategy aimed at converting this residual biomass into a value-added reinforcement for sustainable construction materials, in line with circular economy and bio-based material principles. A reference cementitious formulation (Mix A) was compared with six fiber-reinforced mixtures containing 1.5, 3.0 and 6.0 wt.% licorice fibers. Two fiber conditions were investigated: thermomechanical treated fibers (Mixes B–D) and untreated raw fibers (Mixes E–G). Fresh-state workability and hardened-state performance were evaluated after 28 days of curing. Characterization included fungal resistance under controlled high relative humidity (99% RH) for eight weeks, pull-off adhesion tests on hollow clay brick substrates according to UNI EN 1015-12, and three-point flexural and compressive strength tests according to UNI EN 1015-11.

The results showed that the cementitious matrix was intrinsically resistant to fungal growth, whereas high fiber contents (6.0%) promoted significant mound development. Fiber incorporation generally reduced pull-off adhesion compared with the unreinforced reference (0.152 MPa). Conversely, low fiber contents provided relevant mechanical benefits: Mix B exhibited a 23% increase in flexural strength, reaching 2.26 MPa, while Mix E achieved a 26% increase in compressive strength, reaching 43.79 MPa. At 6.0% fiber content, progressive internal crushing and a pronounced cushioning effect were observed under compression. Overall, 1.5–3.0 wt.% represents the most promising fiber range, offering a favorable balance between mechanical performance, biomass valorizations and potential application in sustainable construction. Moisture exposure should nevertheless be carefully considered in future applications.

Keywords: Spent licorice root fibers, Bio-based materials, Circular economy, Sustainable construction, Cementitious composites.

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1. Introduction

The construction sector is increasingly adopting circular-economy strategies aimed at reducing resource consumption, waste generation and the environmental impact of building materials. In this context, the incorporation of renewable resources and agro-industrial residues into cement-based composites offers an opportunity to replace part of conventional raw materials while promoting waste valorization and circular material flows (Ginga et al., 2020; Oluleye et al., 2022).

Lignocellulosic fibers are promising bio-based reinforcements due to their renewable origin, low density and favorable specific properties (Asim et al., 2015; Ardanuy et al., 2015). However, their high hydrophilicity and water absorption may affect workability, fiber–matrix adhesion and durability, while the alkaline cementitious environment can influence fiber stability. Pre-treatment can therefore improve fiber morphology and compatibility with inorganic matrices (Onuaguluchi and Banthia, 2016; Rocha et al., 2022).

Spent licorice roots (Glycyrrhiza glabra) represent an underutilized lignocellulosic residue with potential for recovery as a secondary bio-based resource. Madeo et al. (2025) demonstrated the recovery of fibers from licorice-processing waste and their potential application in green building products, while Madeo et al. (2026) highlighted their broader potential within circular-economy pathways.

This study investigates spent licorice root fibers in a Portland cement-based plaster, comparing untreated and thermomechanical washed and cleaned fibers at 1.5, 3.0 and 6.0 wt.%. Fungal resistance under high relative humidity and adhesion to masonry substrates were evaluated to identify suitable fiber contents and pre-treatment conditions for the development of more sustainable cementitious materials.

2. Materials and methods

A commercial cement-based plaster containing Portland cement clinker (KELIX) was used as the reference matrix. Seven formulations were prepared: an unreinforced control (Mix A) and six fiber-reinforced mixtures containing 1.5, 3.0 and 6.0 wt.% spent licorice root fibers (Glycyrrhiza glabra). Thermomechanical washed and cleaned fibers were used in Mixes B–D, while untreated mechanically defibrated fibers were used in Mixes E–G. The theoretical water-to-solid ratio was initially fixed at 0.33. Due to the high-water absorption of the lignocellulosic fraction, the water content was experimentally adjusted for

the 3.0 and 6.0 wt.% formulations to maintain comparable fresh-state consistency. The mix proportions are reported in Table 1.

Table 1. Mix design of the investigated cementitious plaster formulations.

Mix	Reinforcement	Plaster [g] ± 0.1	Licorice Fiber [g] ± 0.1	Solid Phase [g] ± 0.1	Theoretical Water [g] ± 0.1	W/S
A	Control (0%F)	75.0	0.0	75.0	25.0	0.33
B	1.5% Clean F	73.5	1.5	75.0	25.0	0.33
C	3.0% Clean F	72.0	3.0	75.0	25.0	0.33
D	6.0% Clean F	69.0	6.0	75.0	25.0	0.33
E	1.5% Raw F	73.5	1.5	75.0	25.0	0.33
F	3.0% Raw F	72.0	3.0	75.0	25.0	0.33
G	6.0% Raw F	69.0	6.0	75.0	25.0	0.33

Note*: F = fiber; W/S = water-to-solid ratio.

2.3. Experimental Characterization and Testing Equipment

After 28 days of curing, the hardened composites were characterized through complementary biological, adhesion and mechanical tests.

Fungal resistance was evaluated on 100 × 50 × 20 mm specimens, one per formulation, exposed for eight weeks to a controlled high-humidity environment (99 ± 1% RH; 18.3 ± 2.8 °C). The specimens were placed above a shallow water layer without direct water contact, while deteriorated organic matter was used as a natural source of fungal spores (Figure 1). Surface colonization was periodically documented by visual inspection and photographic recording.



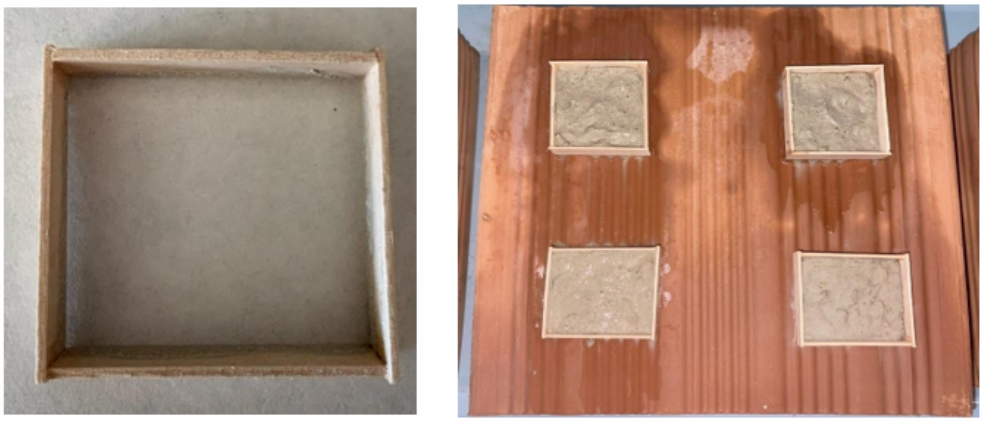
Figure 1. Experimental setup for monitoring fungal colonization of plaster specimens in a closed and sealed environment, with decomposing orange as an organic substrate and water to promote humid conditions.

Adhesion to masonry substrates was assessed according to the principles of UNI EN 1015-12 using three 50 × 50 × 10 mm specimens per formulation applied to pre-moistened hollow clay bricks (Figure 2). After 28 days, 50 × 50 mm metallic dollies were bonded to the plaster surface and subjected to progressively increasing perpendicular tensile loading. The pull-off strength was calculated as:

$$\sigma = \frac{F}{A}$$

where F is the mean failure load and A is the test area (2500 mm²).

Flexural and compressive strengths were determined according to UNI EN 1015-11 on 40 × 40 × 160 mm prisms after 28 days of curing (Figure 3). Three-point bending was followed by compression testing of the resulting half-prisms.



(a) (b)

Figure 2. (a) Manually fabricated 5 × 5 cm mound for plaster application; (b) application of the specimens onto a 10 × 25 × 25 cm perforated brick substrate after wetting.



Figure 3. Plaster specimens after 28 days of drying and conditioning, prior to the pull-off adhesion test.

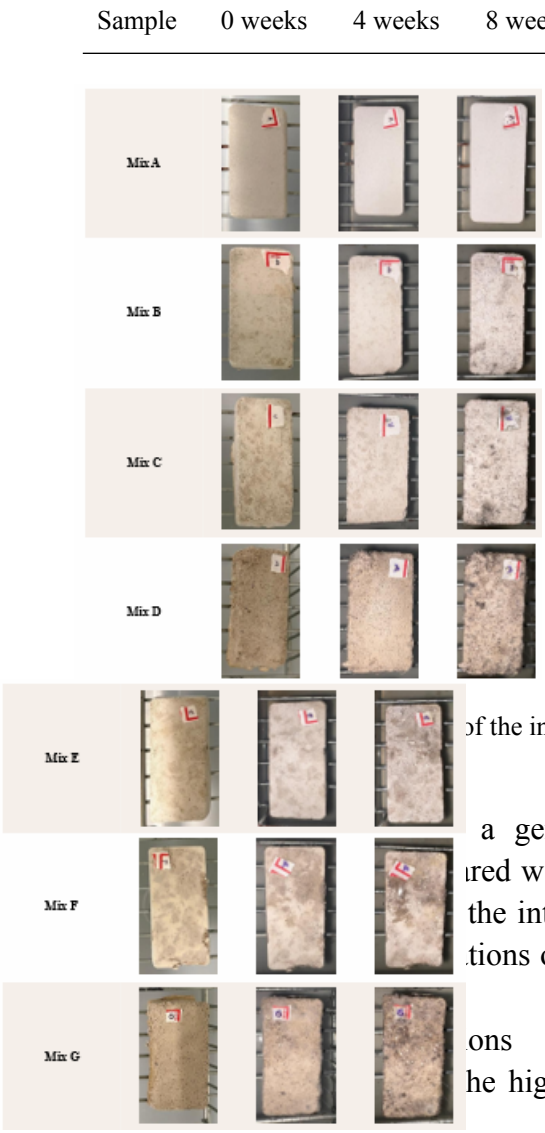
3. Results and discussion

3.1. Fungal Resistance

The fungal-resistance test revealed a clear relationship between fiber content and biological susceptibility. The unreinforced cementitious matrix (Mix A) showed no visible fungal colonization throughout the eight-week exposure, confirming the intrinsic resistance of the highly alkaline cementitious environment. Conversely, increasing the proportion of licorice fibers progressively enhanced surface colonization.

After four weeks, only limited fungal growth was observed in the 1.5 and 3.0 wt.% formulations, whereas pronounced colonization was already evident in Mixes D and G (6.0 wt.%). After eight weeks, Mixes D and G exhibited almost complete surface coverage. The lowest fiber content provided the highest biological resistance: Mixes B and E (1.5 wt.%) showed only slight surface colonization, while Mixes C and F (3.0 wt.%) exhibited intermediate growth (Figure 4).

These results indicate that fiber incorporation introduces an organic substrate capable of increasing fungal susceptibility under persistent high-humidity conditions. Accordingly, fiber contents of 1.5–3.0 wt.% appear preferable, while applications in continuously humid environments should be accompanied by appropriate moisture-control or protective measures.



of the investigated mixtures after 0, 4 and 8 weeks.

a general reduction in adhesion strength
ured with the unreinforced control (Figure 5).
the introduction of discontinuities within the
tions of the plaster–substrate interfacial zone

ons (Mixes B–D) exhibited relatively
he highest value within this group observed

for Mix C (3.0 wt.%). The untreated-fiber formulations (Mixes E–G) showed a progressive reduction in adhesion as fiber content increased. Despite the reduction relative to Mix A, the reinforced formulations maintained relatively consistent behaviour, indicating that moderate fiber incorporation does not produce a disproportionate loss of interfacial performance.

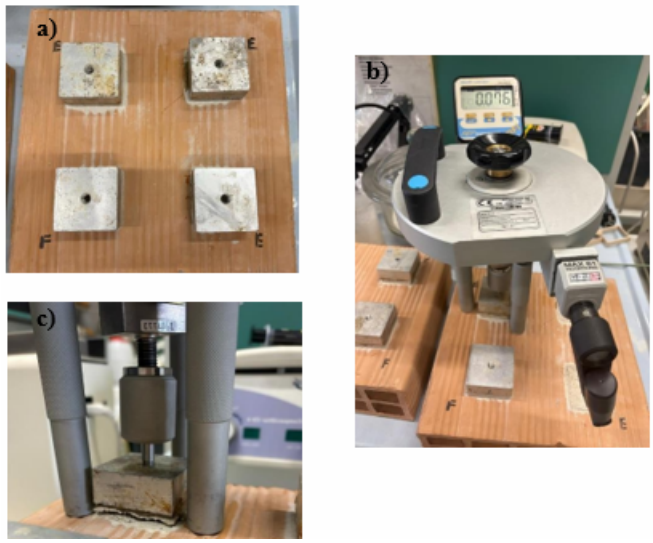


Figure 5. Pull-off adhesion test procedure: (a) bonding of the loading dollies to the specimen surface; (b) application of the tensile load using a pull-off tester; and (c) failure and detachment of the bonded dolly from the specimen.

Table 2. Pull-off adhesion strength of the investigated plaster formulations.

Mix	Fiber condition	Fiber content (wt.%)	Mean pull-off force (N)	Adhesion strength, σ (MPa)
Mix A	No fiber (reference)	0.0	379.50	0.152
Mix B	Treated	1.5	216.67	0.087
Mix C	Treated	3.0	225.00	0.090
Mix D	Treated	6.0	156.00	0.062
Mix E	Untreated	1.5	163.00	0.065
Mix F	Untreated	3.0	122.00	0.049
Mix G	Untreated	6.0	126.00	0.050

The pull-off test results are summarized in Table 2. The unreinforced reference plaster (Mix A) exhibited the highest adhesion strength, equal to 0.152 MPa. Fiber incorporation resulted in a general reduction in adhesion, although the magnitude of this effect depended strongly on both fiber content and pre-treatment.

For the thermomechanical treated fibers, adhesion strength was 0.087, 0.090 and 0.062 MPa at 1.5, 3.0 and 6.0 wt.%, respectively (Mixes B–D). The slight increase observed from 1.5 to 3.0 wt.% suggests that moderate fiber incorporation does not necessarily produce a monotonic deterioration of the interfacial response. At 6.0 wt.%, however, adhesion decreased markedly, indicating that excessive fiber incorporation may increase matrix heterogeneity and disrupt stress transfer across the plaster–substrate interface.

The untreated-fiber formulations showed lower adhesion values throughout the investigated range, with 0.065, 0.049 and 0.050 MPa for Mixes E, F and G, respectively. In this case, the decrease from 1.5 to 3.0 wt.% was more pronounced, while no substantial further reduction was observed at 6.0 wt.%.

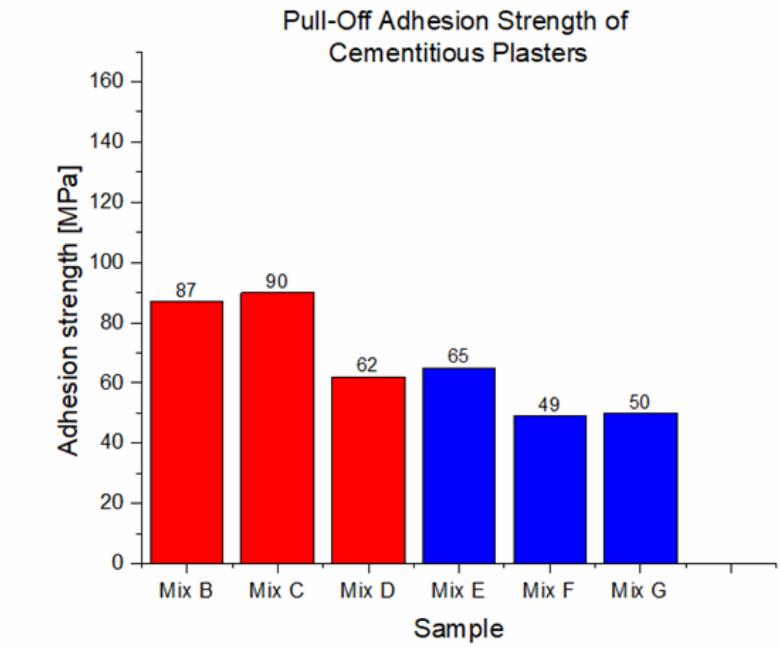


Figure 6. Pull-off adhesion strength of cementitious plasters reinforced with treated and untreated spent licorice root fibers at different fiber contents. Red bars indicate thermomechanical treated fibers (Mixes B–D), whereas blue bars indicate untreated fibers (Mixes E–G).

The direct comparison between treated and untreated fibers highlights the beneficial effect of the pre-treatment on interfacial performance. The treated formulations exhibited approximately 34%, 84% and 24% higher adhesion strengths than the corresponding untreated formulations at 1.5, 3.0 and 6.0 wt.%, respectively. This behaviour may be associated with the removal of soluble and loosely bound organic fractions and with morphological changes induced by the thermomechanical treatment, potentially improving fiber–matrix compatibility and interfacial stress transfer. Nevertheless, all fiber-reinforced formulations remained below the reference Mix A. The reduction can be attributed to the

increased heterogeneity introduced by the lignocellulosic phase and to the consequent modification of the plaster–substrate interfacial region. From an application perspective, the results indicate that fiber pre-treatment is relevant for limiting the loss of adhesion, while moderate fiber contents provide a more favorable compromise between biomass incorporation and interfacial performance.

6. Concluding remarks

This study demonstrated the potential of spent licorice root fibers (Glycyrrhiza glabra) as a secondary bio-based resource for cementitious plaster applications. The fungal resistance test showed that fiber incorporation progressively increased susceptibility to biological colonization under prolonged exposure to very high relative humidity, particularly at 6.0 wt.%, whereas the 1.5–3.0 wt.% formulations showed substantially better resistance.

Pull-off testing indicated a general reduction in adhesion compared with the unreinforced matrix; however, thermomechanical treated fibers consistently provided higher adhesion values than untreated fibers, with the most favorable difference observed at 3.0 wt.%. This result suggests that fiber pre-treatment may improve fiber–matrix interaction and represents a relevant parameter for future optimization.

Overall, moderate fiber contents (1.5–3.0 wt.%) appear to offer the most promising balance between material incorporation and resistance to aggressive humidity conditions. Although further optimization is required, particularly regarding fiber treatment, moisture resistance and long-term durability, the results support the feasibility of valorizing spent licorice residues as a component of more sustainable cementitious materials. This approach contributes to the principles of industrial symbiosis and circular economy by converting an agro-industrial waste stream into a potentially useful resource for bio-based construction applications.

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