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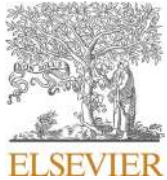
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New eco-bio-based insulating material from raw diss fiber and terracotta waste: hygrothermal performance and microstructural evolution

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ABSTRACT

In the context of sustainable construction, optimizing building envelopes to reduce environmental and energy impacts has become essential. This study investigates the combined valorization of two local wastes, terracotta waste powder (TWP), used as a partial substitute for hydrated lime, and raw diss fibers (*Ampelodesmos mauritanicus*), to develop a new eco-bio-based insulating composite for enhancing the hygrothermal performance of walls. Conducted within a circular economy framework, this research enhances the understanding of composites that integrate biobased resources with recycled mineral waste. The influence of TWP, fiber content and size was investigated through multiphysical characterization measuring density, porosity, capillary absorption, hygrothermal properties and mechanical strength, complemented by X-ray diffraction, micro-thermogravimetric analysis, scanning electron microscopy and optical microscopy. Increasing diss fiber content from 0 to 35 % of the binder mass resulted in a lighter, more porous composite while improving thermal insulation, with thermal conductivity decreasing from 0.357 to 0.166 W m⁻¹ K⁻¹. The moisture-buffering value also increased from 1.05 to 1.89 g m⁻².%RH⁻¹. Although mechanical strengths decreased, they remained suitable for the intended non-structural applications. Long fibers accentuated porosity and thermal improvements while enhancing flexural resistance, whereas short fibers favored moisture regulation and compressive strength. In addition, TWP contributed to maintaining and then moderately increasing the long-term compressive strength by improving the matrix microstructure, while also providing additional gains in thermal insulation and moisture regulation without significantly altering porosity, density or liquid water uptake. Overall, the material demonstrates promising potential for thermal insulation and moisture control in building envelopes while valorizing low-carbon local resources.

1. Introduction

The building and construction sector today play a central role in the ecological and energy transition, as it must both improve energy efficiency and reduce its environmental footprint. In 2023, it still accounted for approximately 34 % of global CO₂ emissions

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and nearly 32 % of worldwide energy consumption [1]. Climate change is further intensifying heating and cooling demands, making the building envelope a key parameter for both energy performance and indoor comfort [2] and highlighting the need for materials with enhanced thermal insulation and hygrothermal regulation capacities. However, conventional insulation materials often exhibit several drawbacks, including high production costs and significant environmental burdens during manufacturing [3]. More broadly, the construction sector consumes nearly 40 % of the 90 billion tons of raw materials extracted annually [4] and remains one of the largest generators of solid waste, contributing nearly 30 % of global construction and demolition waste [5]. In the southern Mediterranean region these waste streams are between 0.19 and 0.30 t·cap⁻¹·yr⁻¹, particularly in Algeria, Morocco, Tunisia, and Lebanon [6]. Recovering and recycling such waste therefore represent key strategies to reduce environmental emissions and relieve pressure on natural resources. In this context, the main challenge for the construction sector is to design buildings that are more comfortable, energy-efficient, and environmentally responsible. Using composites that combine recycled components and bio-based resources offers a particularly promising pathway. In this regard, the use of plant-based aggregates or fibers has attracted increasing interest [7–9], as it enables the development of low-carbon composites with low density, good hygrothermal and acoustic properties, mechanical reinforcement potential, and wide availability at moderate cost.

Among these renewable resources, diss fiber (*Ampelodesmos mauritanicus*), derived from a wild grass species that is highly abundant throughout the Mediterranean basin, particularly in Algeria and southern France, was traditionally used in vernacular buildings for its mechanical strength and hydric properties [10]. It exhibits competitive mechanical performance, comparable to that of sisal, with tensile strength ranging from 100 to 270 MPa depending on the extraction method [11,12]. Its rough surface morphology also promotes fiber-matrix adhesion in mineral binders [10,13]. These intrinsic properties make diss fiber a promising bio-based reinforcement for sustainable composites. Nevertheless, most previous studies have primarily focused on its mechanical contribution. In cement-based matrices, including pastes [10,13], self-compacting concretes [14], mortars [15], and alternative binders such as Tradical PF70 (composed of 75 % aerial lime, 10 % hydraulic lime, and 15 % pozzolan) [16], diss fiber incorporation generally increases flexural strength, toughness, ductility, and lightness, and reduces shrinkage, while is accompanied by a decrease in compressive strength and higher porosity. Diss fibers have also demonstrated effective reinforcement in polymer matrix [11,12,17] and in extruded earth materials [18].

The thermal insulation capacity of diss fibers has been widely investigated. Sellami et al. [19] reported that incorporating diss fibers (20–30 % by binder mass) into cement-based composites reduced the thermal conductivity by 76–87 %, compared with a reference matrix. Similarly, Colajanni et al. [20] and Megna et al. [21] observed that a lime-based formulation containing recycled glass powder and 20 % diss fibers exhibits a thermal conductivity of 0.21 W m⁻¹ K⁻¹, confirming the potential of this resource for the development of low-conductivity bio-based composites.

Regarding hygric behavior, diss fibers display pronounced hygroscopicity, characterized by a type II sorption isotherm and H3-type hysteresis, which are typical of lignocellulosic materials [22]. This moisture sensitivity can affect the behavior of composites under certain conditions [23], whereas it may also be exploited for hygrothermal regulation, as demonstrated for several bio-based materials such as hemp concrete [24,25]. This approach, however, requires appropriate hygrothermal design and consideration of the real service conditions of bio-based walls, particularly temperature, humidity, and cyclic moisture loads [26,27]. However, the use of diss fibers to optimize the hygrothermal properties of composites remains limited in the literature and, to our knowledge, only Zaid study [28] reports an improvement in moisture buffering capacity.

In most of the studies mentioned, thermal or chemical pre-treatments of diss fibers are used to remove compounds that delay binder setting and to improve interfacial adhesion and strength. However, this approach is not systematically required. For instance, Fertikh et al. [29] demonstrated that raw diss fibers interact favorably with clay-lime matrices due to their naturally rough texture, which ensures effective mechanical interlocking. Likewise, Colajanni et al. [20] reported satisfactory results for lime-pozzolanic glass composites containing untreated diss fibers. Moreover, such treatments may impair insulation performance [30,31] and alter the hygroscopic behavior of the fibers [22], thereby reducing their moisture-regulation capacity and limiting their relevance for hygrothermal improvement. They also tend to worsen the environmental balance and increase costs, which raises questions regarding the ecological relevance of these processes [32]. Overall, these observations highlight the key role of binder selection in the behavior of biocomposites and suggest that, beyond conventional cementitious matrices, further investigations on alternative binders and formulations are needed to identify solutions that limit, or even avoid, the need for diss fiber treatments.

Generally, lime-based binders, either aerial or weakly hydraulic, are preferred for plant-based composites because they yield lightweight, porous, and vapor-permeable materials that demonstrate good hygrothermal regulation [9,33]. Pozzolanic additions, such as metakaolin, are commonly introduced to reduce material alkalinity, limit Ca(OH)₂ content, and enhance fiber-matrix compatibility [34–36]. The use of pozzolanic materials in lime mortars has indeed been well established since Roman times [37], and many studies today show that valorizing construction wastes, such as sanitary ceramics, terracotta, glass, or sediments, as additions or partial binder substitutes can replace a portion of cement or lime, with economic and environmental benefits [38–41]. These materials typically contain an amorphous silica and alumina phases, which, when finely ground, exhibits pozzolanic reactivity with calcium hydroxide, to form C–S–H and C–A–S–H, thereby enhancing strength and microstructural refinement [39,41–43]. In particular, terracotta wastes, such as red brick powder, has been proven effective in lime mortars for heritage restoration of buildings [39], as well as in cement-based mortars [41,44]. Moreover, various works indicate that the optimal incorporation level of brick powder ranges between 10 % and 20 % as a binder replacement [41,45,46].

Some authors have shown that these brick powders can also be incorporated into bio-based composites. For instance, Abdelatef et al. [46] reported that adding 10 % recycled brick powder to a lime-based binder for hemp concrete improved thermal insulation and moisture regulation while maintaining mechanical properties comparable to those obtained with metakaolin- or hydraulic-lime-based mixtures. Conversely, Zúniga et al. [47] observed no strength improvement, though thermal conductivity decreased. These

discrepancies can be attributed to differences in formulation, replacement level, or the intrinsic properties of the waste powder, including mineralogical composition, origin, firing conditions, and particle fineness, which strongly influence its pozzolanic reactivity [41,48,49].

Although many studies have highlighted the interest in combining pozzolanic binders with plant fibers, the specific integration of terracotta waste powder (TWP) remains scarcely investigated. Existing studies also report limitations, in particular delicate trade-offs between hygrothermal and mechanical performance, likely due to raw-material variability or binder incompatibilities. The diversity of formulations reported in the literature also limits comparability, underscoring the need for further systematic optimization.

In this context, the present research aims to develop a new eco-bio-based insulating composite by valorizing two local resources: terracotta waste powder (TWP) from red bricks, used as a partial replacement for hydrated lime to produce a pozzolanic binder, and untreated diss fibers. This original combination, not yet reported in the literature, is designed to yield a low-impact carbon insulating material with enhanced hygrothermal performance for building envelopes, while limiting energy consumption and costs.

The study investigates the effects of fiber content and size, as well as the partial substitution of lime with TWP, through an extensive multiphysical experimental program. The latter included the measurement of density, porosity, capillary water absorption, and hygrothermal and mechanical properties, alongside XRD, TGA, SEM, and optical microscopy analyses to elucidate microstructural development and fiber-matrix interactions.

2. Materials and methods

2.1. Materials

This section describes the properties and preparation procedures of the raw materials used in this study, namely diss plant substrate (*Ampelodesmos mauritanicus*), for which two particle-size classes were produced (0–12 mm and 0–25 mm), as well as terracotta waste powder (TWP) and local hydrated aerial lime. It also presents the mixture proportions, mixing protocol, specimen fabrication and curing conditions.

2.1.1. Diss fibers (*Ampelodesmos mauritanicus*)

The diss fibers used in this study were extracted from the leaves of *Ampelodesmos mauritanicus* plant, harvested in June at Beni Ourtilane (Sétif, Algeria) under ambient conditions. After 15 days of air-drying, the leaves were cut into 25–30 cm segments and then oven-dried at 50 °C for 24 h until constant mass. Grinding was performed using a knife mill (Retsch SM 300) equipped with a 10 mm sieve and a six-disc rotor, operated at the minimum speed of 700 min⁻¹ to limit the production of fine particles. The resulting long fibers were sorted, sieved, and cut manually to obtain two size fractions (<25 mm and <12 mm) to assess the effect of particle size on the composite properties. The preparation protocol is illustrated in Fig. 1.

The physical properties of the fibers were characterized in accordance with the recommendations of RILEM Technical Committee 236-BBM [50]. Measurements covered particle size distribution (PSD), determined by mechanical sieving and image analysis (Fig. 3), bulk density, initial moisture content, and water absorption capacity (Fig. 4). The absolute density was obtained using a pycnometer with acetone as the reference liquid, in accordance with standards NF P 18-558 and NF T 51-063. Total porosity was calculated from the bulk and absolute densities. The results are presented in Table 1.

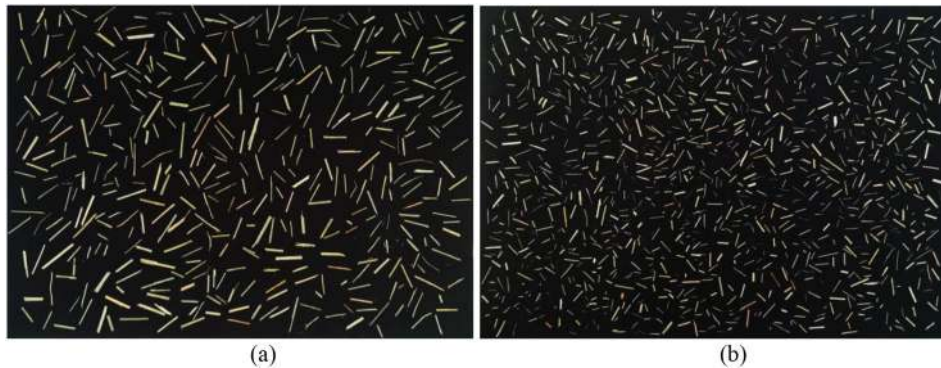


Fig. 1. Preparation steps of diss Fibers.

Table 1

Physical characteristics of diss fibers used in this study.

Diss fiber fraction	Dry bulk density ρ_d (kg·m ⁻³)	Absolute density ρ_a (kg·m ⁻³)	Total porosity ε_{tot} (%)	Initial moisture content Ω (%)	Water absorption rate W (%)
<25 mm	140.6 ± 1.9	990 ± 15	85.8	6.9 ± 0.3	81.5 % ± 2.8 (5 min)
<12 mm	168.2 ± 1.2		83.0		112.5 % ± 0.6 (24 h)

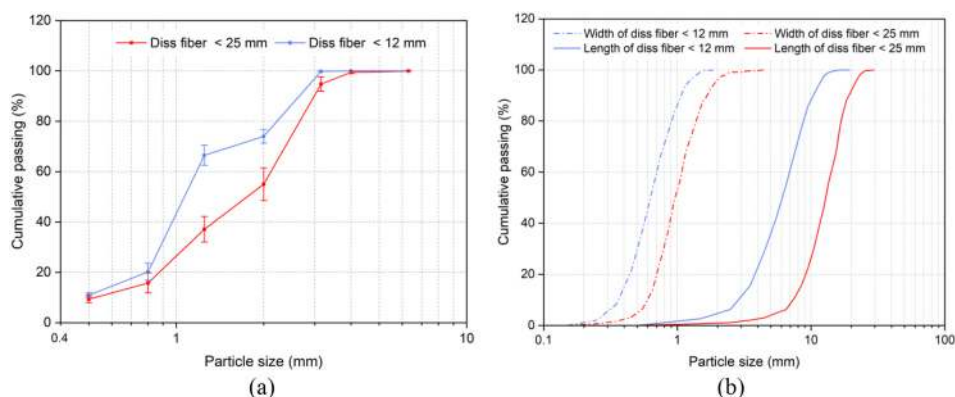
**Fig. 2.** Image scans of the two diss fibers fractions used for particle-size analysis: (a) < 25 mm and (b) < 12 mm.

Mechanical sieving curves (Fig. 3a) highlight distinct PSD profiles between the two diss fiber fractions. The coarser fraction mainly consists of particles in the 2–4 mm (44.4 %) and 0.8–2 mm (39.2 %) classes, whereas the finer fraction is concentrated primarily between 0.5 and 1.25 mm (55.6 %) and 1.25–3.15 mm (33.3 %). However, this method tends to underestimate actual fiber dimensions because of their elongated shape. To refine this characterization, an image-based analysis was performed using ImageJ software on subsamples of 429 and 852 particles, corresponding to the first and second fractions, respectively. The fibers were carefully arranged without overlap on black paper (Fig. 2) to allow precise segmentation. The results (Fig. 3b) indicate that 77.2 % of the fibers in the first fraction have lengths between 8 and 20 mm, while 74.8 % of those in the second fraction range from 3.5 to 10.5 mm in length. The widths ranged from 0.2 to 4 mm for the first fraction and from 0.2 to 2 mm for the second one.

Like many lignocellulosic materials, diss fibers exhibit a marked water affinity, due to their high porosity and polysaccharide-rich chemical composition. This hydrophilic nature, which can influence their interaction with mineral-based matrices, prompted a specific examination of their water-absorption capacity. When plotted as a function of the square root of time (Fig. 4a), the absorption process shows a two-stage behavior. An initial rapid and nearly linear phase (1–5 min) reached 81.5 % uptake, indicative of high hydrophilicity and microporous structure, and can be described by a logarithmic fit (Fig. 4b) with an initial rate of absorption (IRA) = 43.6 % and an apparent diffusion coefficient $K_1 = 55.4$ %. This is followed by a slower phase, corresponding to water migration into less-accessible inner pores and large capillaries already saturated, leading to final absorption of 112.5 % after 24 h. This behavior is consistent with previous observations for flax and hemp fibers [51,52], as well as with earlier findings for diss fibers [13].

2.1.2. Terracotta waste powder (TWP)

The terracotta waste powder (TWP) valorized in this work was obtained from defective or broken red construction bricks, recovered from the PROMAG SARRL brickyard (Hassi Ben Okba, Oran, Algeria). These rejects were first crushed using a laboratory jaw

**Fig. 3.** Particle-size analysis of diss fibers by mechanical sieving (a) and image processing (b).

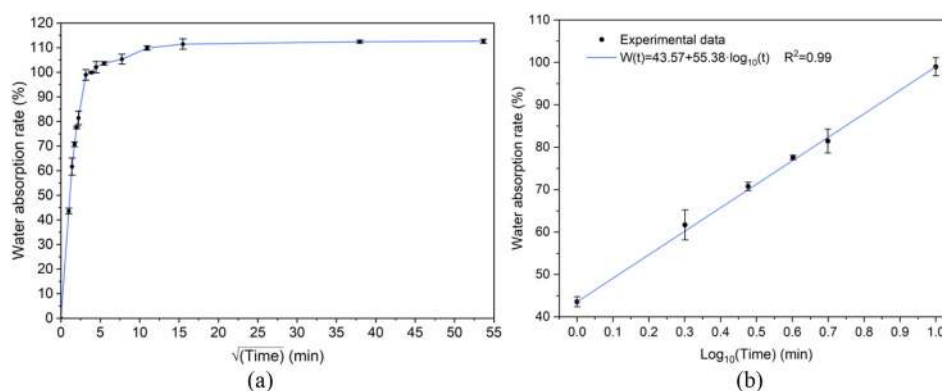


Fig. 4. Water absorption of diss fibers: (a) as a function of the square root of time over 48 h; (b) variation with $\log_{10}(\text{time})$ over the first 10 min.



Fig. 5. Preparation process of terracotta waste powder (TWP).

Table 2

Physical properties of terracotta waste powder (TWP).

Dry bulk density ρ_d ($\text{kg}\cdot\text{m}^{-3}$)	Absolute density ρ_a ($\text{kg}\cdot\text{m}^{-3}$)	Sieve passing at 45 μm (%)	Sieve passing at 80 μm (%)	Blaine specific surface area ($\text{cm}^2\cdot\text{g}^{-1}$)
705.6	2700 ± 21	78.52 – 82.10	99.92 – 99.98	6482

crusher, reducing the fragment size to 0.5–3.5 cm. They were then dried at 105 °C for 24 h and finely ground in a ball mill until the optimal fineness was reached. This particle-size refinement was verified by wet sieving at 45 μm , with a sieve residue between 17.90 % and 21.48 %. The powder preparation protocol is illustrated in Fig. 5, and the physical properties of the TWP are presented in Table 2.

2.1.3. Hydrated aerial lime

The selected binder for this study is a hydrated aerial lime (slaked lime), produced in Saïda (Algeria) by BMSD-SARL and marketed under the name “Fleur de chaux”. Its physicochemical properties, provided by the manufacturer, are presented in Tables 3 and 4. The apparent bulk density, measured in accordance with NF EN 459-2, was 352 $\text{kg}\cdot\text{m}^{-3}$.

2.1.4. Composition and mix proportions of the formulations

As part of this research devoted to the development of a new eco-bio-based insulating composite, six formulations were designed to assess the influence of diss fiber content, the partial substitution of lime with TWP, and fiber particle size on the composite properties.

Formulation F1, used as the reference, consisted of 85 % lime and 15 % TWP, without fibers. Formulations F2, F5, and F6 maintained this same matrix while incorporating 15 %, 25 %, and 35 % diss fibers shorter than 25 mm, respectively. The fiber dosage was expressed as the fiber-to-binder mass ratio (D/B). Formulation F3, consisting solely of 100 % lime and 15 % fibers, was designed to isolate the effect of TWP substitution. Formulation F4 was identical to F2 except that it used finer diss fibers, shorter than 12 mm, to evaluate the effect of particle size.

Table 3

Chemical composition of hydrated lime (“Fleur de chaux”, BMSD SARL, Algeria).

Ca(OH)_2 Total	Ca(OH)_2 Soluble	Fe_2O_3	Al_2O_3	MgO	Mn_3O_4	SiO_2	Fe	Al	Si	Mn	CO_2
85.57–95.50	≥ 90	0.116	0.109	0.125	0.1058	0.126	0.112	0.124	0.134	0.112	< 2

Table 4

Physical properties of hydrated lime ("Fleur de chaux", BMSD SARL, Algeria).

Fineness	Bulk density
98.4–99.6 % $\leq 90 \mu\text{m}$	270–370 (kg m^{-3})
87.6–93.2 % $\leq 50 \mu\text{m}$	
82.5–84.3 % $\leq 32 \mu\text{m}$	

Table 5

Composition and designation of the different formulations.

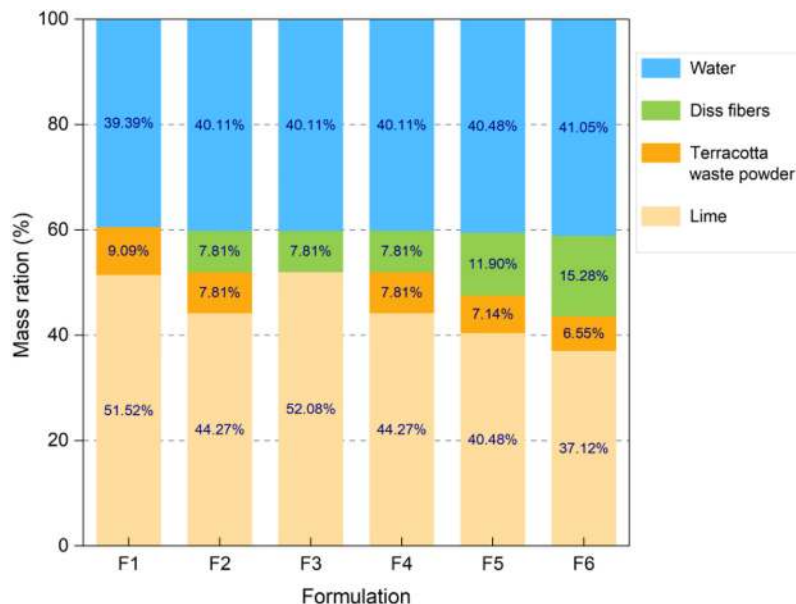
Formulation designation	Fiber size (mm)	Lime Binder	TWP Binder	Diss fiber Binder	W_{paste} Binder	$W_{\text{prewetting}}$ Binder	W_{tot} Binder
F1	-	0.85	0.15	0	0.65	0	0.65
F2	<25	0.85	0.15	0.15	0.65	0.12	0.77
F3	<25	1	0	0.15	0.65	0.12	0.77
F4	<12	0.85	0.15	0.15	0.65	0.12	0.77
F5	<25	0.85	0.15	0.25	0.65	0.20	0.85
F6	<25	0.85	0.15	0.35	0.65	0.29	0.94

The mixing water content was determined in two steps. In a first step, the consistency of the pastes based on lime alone or on lime and TWP was evaluated visually, by touch, and then using a flow table (15 blows). A water to binder ratio [$W/(\text{lime} + \text{TWP}) = 0.65$] was selected, producing an adhesive texture similar for both compositions, with a flow diameter between 100 and 110 mm. In a second step, a pre-wetting of the diss fibers was set at 81.46 % of their dry mass, based on the 5-min water absorption value. This step was deemed essential in order to prevent excessive absorption of the mixing water by the fibers and to preserve the workability of the mixture. This pre-wetting method follows recommendations from previous work on bio-aggregate materials, notably Chabannes et al. [53], and Jonathan Page et al. [52]. Since no specific standard exists for characterizing the consistency of such fiber-lime composites, workability was assessed visually and manually, to obtain a compact and cohesive grout, with a sticky consistency, suitable for producing stable, well-held specimens, as already reported by Zúniga et al. [47]. Satisfactory workability was obtained for fiber contents between 15 % and 35 %, whereas higher proportions led to a loss of cohesion in the mixture. The designations and constituent proportions of the different formulations are presented in Table 5 and are represented graphically in Fig. 6.

Where: Binder = (lime + TWP) and Water content = $W_{\text{tot}} = (W_{\text{paste}} + W_{\text{prewetting}})$.

2.1.5. Sample preparation and curing conditions

To ensure reproducible and homogeneous mix preparation, a rigorous specimen-fabrication protocol was applied. The plant-based fibers were first introduced into the mixer and dry-mixed for 30 s to promote uniform dispersion. The pre-wetting water was then

**Fig. 6.** Mass proportions of the constituents in the different developed formulations.

added gradually over 30 s, while mixing continued at low speed for approximately 5 min. With the mixer stopped, the dry binder (lime + TWP) was incorporated, and mixing was resumed for 2 min at low speed. Lumps were broken up manually before adding the mixing water (30 s), followed by a final mixing step of 2 min 30 s at low speed, until a mixture with a uniform distribution of components was obtained.

The resulting mixture was cast into pre-oiled steel molds, except for the cylindrical specimens, which were made in PVC, to facilitate demolding. Casting was performed in two successive layers, each vibrated on an automatic shaking table with 30 blows for each 30 s. The following specimen geometries were prepared:

- Cubic specimens ($50 \times 50 \times 50$ mm) for compressive strength;
- Prismatic specimens ($100 \times 100 \times 50$ mm) for thermal conductivity, water vapor permeability, moisture buffer value (MBV), and capillary water absorption tests;
- Prismatic beams ($40 \times 40 \times 160$ mm) for flexural strength;
- Cylindrical specimens ($\varnothing 75 \times 50$ mm) for water-accessible porosity measurements.

After casting, the specimens were stored under controlled laboratory conditions ($23 \pm 2^\circ\text{C}$, $55 \pm 5\%$ RH). Demolding was performed after 7 days, and the samples were then cured under the same environmental conditions until testing at 28, 56, or 90 days, depending on the property investigated. The preparation and fabrication steps of the different mixes are illustrated in Fig. 7.

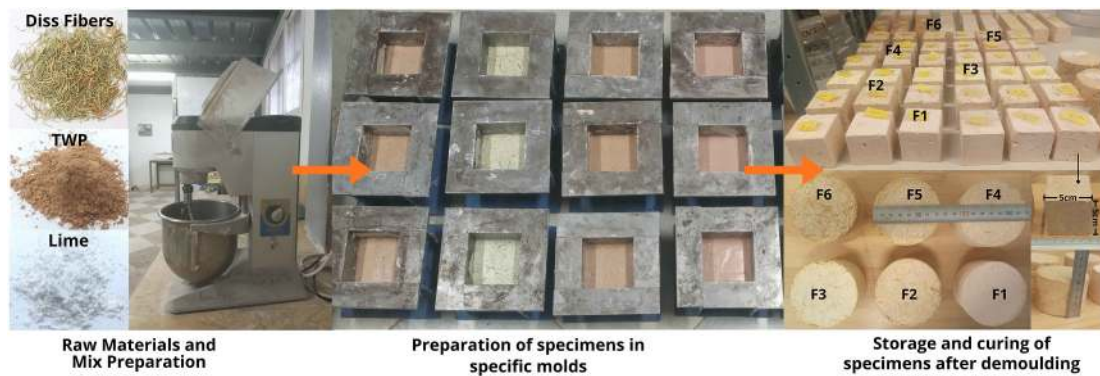


Fig. 7. Preparation and fabrication of mixes.

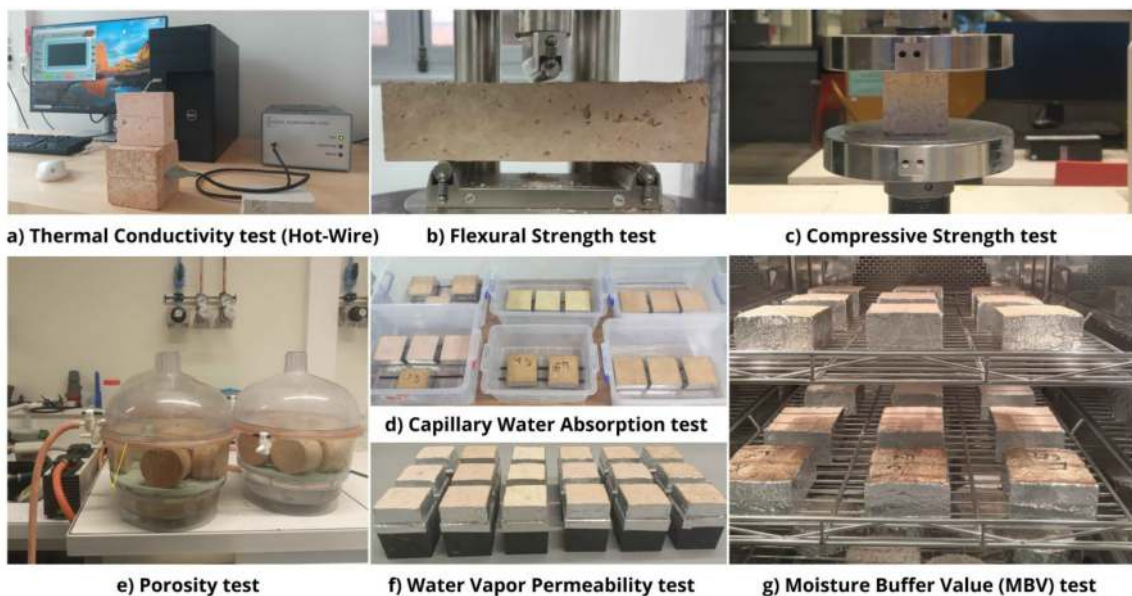


Fig. 8. Overview of the experimental characterization program and main tests performed on the developed composites.

2.2. Experimental characterization and test methods

This section presents the experimental procedures and test methods implemented in the multiphysical characterization of the eco-bio-based insulating composites developed in this study. The investigated properties include dry bulk density, open porosity, capillary water absorption, hygrothermal performance (i.e. thermal conductivity, water vapor permeability and Moisture Buffer Value (MBV)), and mechanical performance (i.e. flexural and compressive strength).

These measurements were conducted to evaluate the effects of diss fiber content and length, as well as the partial substitution of lime with TWP, on the overall behavior of the composites. The main tests performed on the different formulations are summarized in Fig. 8.

In addition to these macroscopic characterizations, mineralogical and microstructural investigations were performed to gain deeper insight into the matrix evolution and fiber-matrix interactions. These analyses involved X-ray diffraction (XRD), thermogravimetric and derivative thermogravimetric analyses (TGA/DTG), scanning electron microscopy (SEM), and optical microscopy.

The characterization ages were selected to provide both a common reference state for comparing the mixes and a more advanced state better suited to properties governed by moisture transfer. In particular, 56 days was taken as a sufficient maturity age for most properties, allowing the limitation of early-age effects, whereas MBV and water vapor permeability were measured at 90 days to ensure a more stabilized moisture state and a more consolidated pore network. However, compressive strength was monitored at 28, 56 and 90 days to describe the evolution of strength over time, and SEM/XRD/TGA analyses were carried out at 56 and 90 days to relate the evolution of the matrix and fiber-matrix interactions to the measured performance.

2.2.1. Dry density

The dry density of the eco-bio-based composites incorporating terracotta waste powder (TWP) and diss fibers was determined after 56 days of curing. Specimens were oven-dried at 50 °C and considered stable when the mass variation was less than 0.01 % between three weighings at 24 h intervals, following the protocol described by RILEM TC-275 HDB [54]. Mass was measured with an analytical balance (± 0.01 g), and the apparent volume was determined using a digital caliper (± 0.01 mm) from multiple readings on each face, accounting for geometric irregularities specific to each specimen.

2.2.2. Open porosity (water-accessible porosity)

Open porosity was evaluated by the water-accessible porosity test in accordance with standard NF P18-459 [55], allowing the quantification of the volume fraction of open pores that can be saturated during prolonged immersion. For each formulation, three cylindrical specimens (diameter = 7.5 cm, height = 5 cm), aged 56 days, were tested. After oven-drying at 60 °C to constant mass, the cooled specimens were placed under vacuum for 4 h to remove entrapped air, then immersed for 44 h. Masses were determined by hydrostatic weighing, first in water to obtain the immersed mass M_{water} , then in air to measure the water-saturated mass M_{air} , in addition to the dry mass M_{dry} . The open porosity was then calculated by relating the volume of water contained in the open pores to the total volume of the specimen according to the relationship:

$$\varepsilon = (M_{\text{air}} - M_{\text{dry}}) / (M_{\text{air}} - M_{\text{water}}) \times 100 \quad (1)$$

2.2.3. Thermal conductivity

Thermal conductivity (λ) characterizes the ability of the composite to resist heat transfer, making it an essential parameter for assessing its thermal insulation performance. In this research, this parameter was determined using the transient hot-wire method according to ASTM D5930-97 standard [56]. Measurements were carried out using a hot-wire conductivimeter (NeoTIM FP2C) equipped with a 12.9 Ω resistive probe connected to a computer via an FP2C acquisition unit. Specimens (100 × 100 × 50 mm) cured for 56 days were tested for each formulation. The probe, placed between two identical specimens, was held in optimal contact using a weight. Tests were performed with two orientations relative to the manufacturing direction, in parallel configuration ($\lambda_{||}$) and perpendicular configuration ($\lambda_{\perp}$), in a climate-controlled environment at 23 ± 2 °C and 50 ± 5 % RH. The influence of humidity on $\lambda_{||}$ was evaluated under humid conditions (85 % RH, 23 ± 2 °C) in a desiccator regulated with a saturated potassium chloride solution, and under dry conditions using silica gel to maintain zero humidity. Before thermal conductivity measurements at each relative humidity, the specimens were conditioned until hygrometric equilibrium was reached. Specimens previously stored under laboratory conditions at 23 ± 2 °C and 55 ± 5 % RH reached equilibrium after 9 days when transferred to 50 ± 5 % RH and after approximately 20 days when transferred to 85 % RH. For the dry condition, equilibrium was reached after 7 days at 0 % RH following oven drying at 60 °C for 48 h. The conditioning duration depended on the formulation, particularly the fiber content, with longer durations generally required at higher RH levels. For each formulation, three specimens were combined into three pairs to assess specimen-to-specimen reproducibility within the same formulation. Thermal conductivity was then measured five times for each pair to evaluate measurement repeatability. The mean value was considered representative, and the associated standard deviation was calculated.

2.2.4. Capillary water absorption

Capillary water absorption is a key indicator of hydric behavior of bio-based composites. The test was conducted following the procedure reported in the RILEM TC 275-HDB report [57], adapted from the existing standards EN 1925 and EN 772-11. Three specimens (100 × 100 × 50 mm) per formulation were tested, aged 56 days, after drying at 60 °C to constant mass. The lateral faces were wrapped in a waterproof aluminum film, and the specimens were then placed on plastic supports in a tray maintaining a constant water layer of 8 ± 2 mm beneath the lower face. Mass was recorded at predefined intervals (1, 3, 5, 10, 15, 30, 60, 120, 180, 240,

360 min, then 24 h and 48 h), with the tray left open to avoid any change in ambient humidity. The surface water absorption M_W ($\text{kg}\cdot\text{m}^{-2}$) was then calculated according to equation (2).

$$M_W = (M_t - M_0) / A \quad (2)$$

where: M_t is the mass of the specimen at time t (kg); M_0 is the initial mass of the specimen after drying (kg); and A is the immersed surface area of the specimen (m^2).

The evolution of M_W as a function of the square root of time is plotted, allowing the capillary absorption coefficient (C_A , in $\text{kg}\cdot\text{m}^{-2}\text{h}^{-1/2}$) to be obtained according to equation (3).

$$M_W = C_A \sqrt{t} \quad (3)$$

2.2.5. Moisture buffer value (MBV) test

Moisture Buffer Value (MBV) expresses the capacity of materials to regulate ambient humidity through reversible exchanges by absorbing or releasing vapor in response to variations in relative humidity [58]. In this study, MBV was determined according to the NORDTEST protocol [59]. For each formulation, three specimens ($100 \times 100 \times 50$ mm) aged 90 days were tested after mass equilibrium at $23 \pm 5^\circ\text{C}$ and $50 \pm 5\%$ RH. Five out of six faces of each specimen were covered with aluminum film, leaving only one face exposed. The specimens were then placed in a climatic chamber providing precise control of relative humidity following prescribed cycles comprising 8 h at 75 % RH (adsorption) followed by 16 h at 33 % RH (desorption), at a constant temperature of 23°C . These cycles were repeated until the mass change between three consecutive cycles was less than 5 %. Mass variations were recorded with at least one weighing per cycle and five during the adsorption phase of the last three cycles. The average mass variation was used to calculate MBV.

MBV, expressed in ($\text{g}\cdot\text{m}^{-2}\cdot\%\text{RH}^{-1}$), was calculated according to Equation (4).

$$MBV = (\Delta m) / (A \cdot (RH_{\text{high}} - RH_{\text{low}})) \quad (4)$$

where: Δm is the average mass change per cycle (adsorption/desorption) (g), A is the exposed surface area (m^2), RH_{high} the humidity in adsorption (%), and RH_{low} the humidity in desorption (%).

2.2.6. Water vapor permeability test

Vapor permeability assesses a material's ability to transfer water vapor through its pore network under an isothermal pressure gradient [60] and serves as a key indicator for its suitability in high-performance thermal insulation systems [61]. In this study, it was measured using the cup method in accordance with the EN ISO 12572 standard [62]. Two hygrometric configurations were used: 0/50 % RH for the dry cup and 50/85 % RH for the wet cup.

For each formulation, three specimens ($100 \times 100 \times 50$ mm) aged 90 days were tested after reaching mass equilibrium at 50 % RH and $23 \pm 2^\circ\text{C}$. The internal volume of the cups was filled either with a 30 mm layer of silica gel to maintain a relative humidity close to 0 % in the dry cup, or with a saturated KCl salt solution to ensure 85 % RH in the wet cup. An air layer of 16 ± 1 mm was provided beneath the specimen to ensure homogeneous vapor diffusion. After preparation, the cup-specimen assemblies were placed in a climatic chamber at 23°C and 50 % RH and weighed regularly over time. When the mass variation reached a quasi-linear behavior, the data were used to determine water vapor permeability by applying the relationships defined in EN ISO 12572. The final values correspond to the average of three measurements for each formulation.

2.2.7. Compressive strength

Compressive strength tests were conducted on three cubic specimens ($50 \times 50 \times 50$ mm) for each formulation, following EN 826 [63], to ensure reproducible results. Tests were performed under displacement control (5 mm min^{-1}) using an Instron 3369 universal testing machine equipped with parallel rigid platens to ensure uniform contact. Measurements were carried out after 28, 56, and 90 days of curing.

2.2.8. Flexural strength

Flexural strength was measured after 56 days of curing on three standard prismatic specimens ($40 \times 40 \times 160$ mm) for each mix. Tests were carried out in three-point bending over a 100 mm span, at a displacement rate of 0.3 mm min^{-1} , following NF EN 196-1 [64].

2.2.9. X-ray diffraction analysis

X-ray diffraction (XRD) analysis was carried out to characterize the mineralogical composition of the raw materials (TWP and lime) as well as the crystallinity of the diss fibers. Measurements were also performed on composites F1, F2, and F3, for which only the mineral matrices were analyzed after 56 and 90 days of curing, the fibers were previously removed by grinding and sieving, in order to identify and assess the crystalline phases formed. Diffractograms were obtained with a Malvern Panalytical Aeris diffractometer, using $\text{Cu K}\alpha$ radiation ($\lambda = 1.54182 \text{ \AA}$), at 30 kV and 10 mA. Scanning was conducted from 5° to 70° (2θ), with an acquisition time of 20 min per sample. Semi-quantitative estimation of crystalline phases was performed using PANalytical HighScore (Plus), with results compared against the ICDD (PDF-2) and COD24 databases. The crystallinity index of the diss fibers was calculated according to the Segal method [65], from the following equation:

$$CrI(\%) = (I_{200} - I_{am}) / (I_{200}) \times 100 \quad (5)$$

where: $I_{(200)}$ is the maximum intensity diffracted by the (002) plane at a 2θ angle between 22° and 23° , and I_{am} is the minimum intensity of the diffuse background (amorphous region), measured at a 2θ angle between 18° and 19° .

2.2.10. Thermogravimetric analysis

Thermogravimetric analysis (TGA) is a technique that records the mass loss of a sample as a function of temperature, thereby allowing the identification of decomposed components through comparison with reference data. In this study, TGA was first used to characterize TWP as a raw material, evaluating its thermal and chemical stability. Then, the analysis was conducted on the matrices of composites F1, F2, and F3 after 56 and 90 days of curing to assess the development of hydrated and carbonated products, complementing the X-ray diffraction (XRD) results. Tests were performed using a Netzsch STA 449 F3 Jupiter thermogravimetric analyzer under a nitrogen atmosphere with a flow rate of 20 mL min^{-1} . The temperature ramp was programmed from 25°C to 900°C at a rate of $10^\circ\text{C}\cdot\text{min}^{-1}$. Analyses were conducted on samples with masses between 10 and 100 mg, depending on the nature of the material, placed in cylindrical aluminum crucibles. The test results were analyzed using the TGA curve and the corresponding derivative thermogravimetry (DTG) curves.

2.2.11. SEM and optical microscopy

Digital optical microscopy (KEYENCE VHX-2000) was used for an initial characterization of the fibers and composites at the optical scale. The morphology of the raw diss fibers was examined to assess their initial state, and their anchorage within the mineral matrix was observed to evaluate the quality of their integration in the composite.

Scanning electron microscopy (SEM) was then carried out using a GeminiSEM 560 microscope, enabling high-resolution and more detailed analysis. Before testing, the samples were conditioned under vacuum and coated with a thin carbon layer. SEM observations were performed at an accelerating voltage between 1 and 5 kV. The structural morphology of the raw materials (diss fibers, TWP and lime) was first characterized to evaluate their initial condition. Observations then focused on composite samples (F1, F2 and F3), analyzed after 56 and 90 days of curing, to identify the phases formed during pozzolanic reactions and carbonation and to track the evolution of the microstructure. The analysis was conducted by focusing both on matrix areas where only binder phases appeared and on the surfaces of fibers embedded in the matrix, in order to better understand the hardening mechanisms and the cohesion of the composites. The results also provide complementary insight to the TGA and XRD analyses, contributing to a broader understanding of the mineralogical and structural transformations.

3. Results and discussion

In this section, the raw materials are first characterized by mineralogical and microstructural analyses in order to better describe their intrinsic properties. The results of the various tests conducted on the developed composites are then presented, discussed, and interpreted, with particular attention to material performance and to the influence of the main parameters, in particular diss fiber content and length, as well as the partial substitution of lime with TWP. Finally, complementary mineralogical and microstructural observations are provided to describe the time-dependent development of the matrix and its interaction with the fibers.

3.1. Microstructural and mineralogical characterization of the raw materials

3.1.1. TWP and lime

SEM observations of the TWP shown in Fig. 9a reveal particles of heterogeneous size and surface texture, ranging from angular grains with sharp edges to irregular fragments with smooth or rough surfaces. This morphology is typical of a calcined terracotta material, such as brick residues.

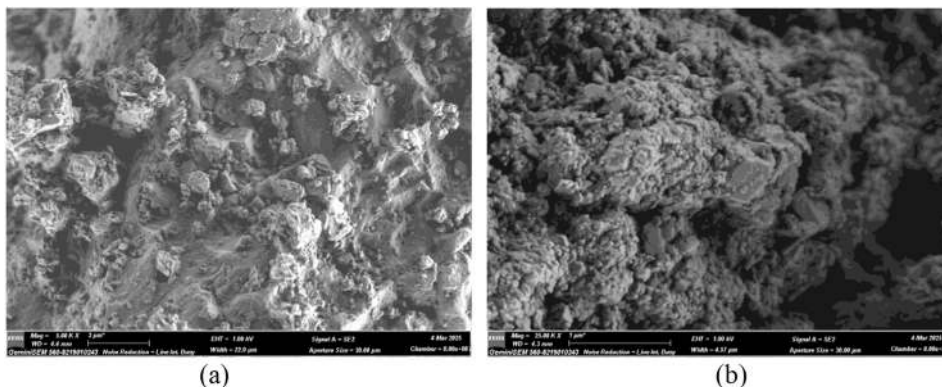


Fig. 9. SEM micrographs of (a) terracotta waste powder (TWP) and (b) hydrated aerial lime.

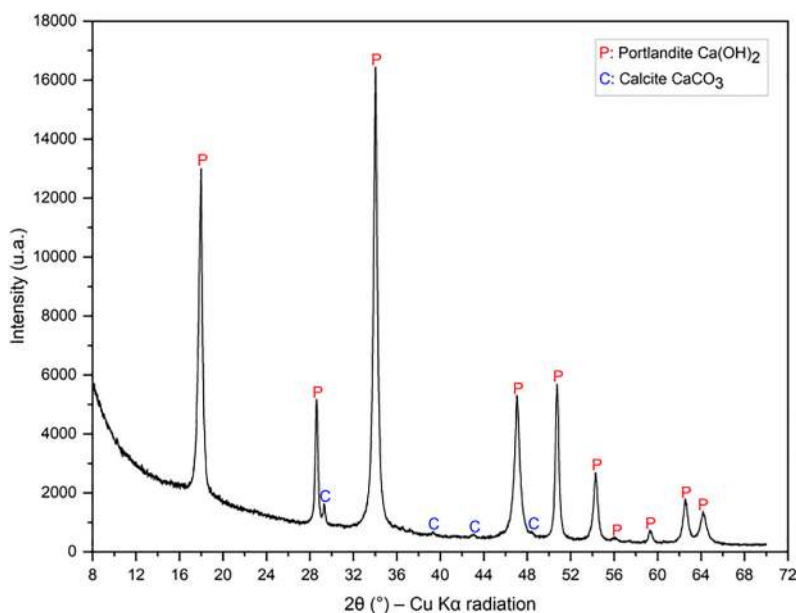


Fig. 10. XRD patterns of hydrated aerial lime.

However, SEM micrographs of the hydrated aerial lime (Fig. 9b) show well-defined hexagonal lamellae of portlandite crystals, characteristic of high-purity $\text{Ca}(\text{OH})_2$ phases and confirming its fine, homogeneous microstructure.

In addition, X ray diffraction (XRD) analysis performed on this aerial lime (Fig. 10) confirms the predominance of $\text{Ca}(\text{OH})_2$, in agreement with the COD database (96-152-9753). This phase, characterized by intense diffraction peaks, represents a content exceeding 90 % and is accompanied by minor peaks of calcite (CaCO_3) (COD 96-900-0968), indicating slight carbonation due to atmospheric exposure.

On the other hand, the heterogeneity observed for the TWP is also reflected in its mineralogical composition determined by XRD (Fig. 11). The diffractogram, indexed by matching with the ICDD databases (01-078-1252; 01-079-1741; 01-089-0951) and COD (96-155-7000; 96-900-0702; 96-900-9666), indicates a semi-crystalline state dominated by quartz (SiO_2), which is consistent with the high silica content of fired clay bricks. Other phases were also identified, notably hematite (Fe_2O_3), traces of magnetite (Fe_3O_4), feldspars, primarily albite ($\text{NaAlSi}_3\text{O}_8$) and microcline (KAlSi_3O_8), as well as illite [$\text{K}_{0.7}(\text{Al}, \text{Mg}, \text{Fe})_2(\text{Si}, \text{Al})_4\text{O}_{10}(\text{OH})_2$]. These minerals originate

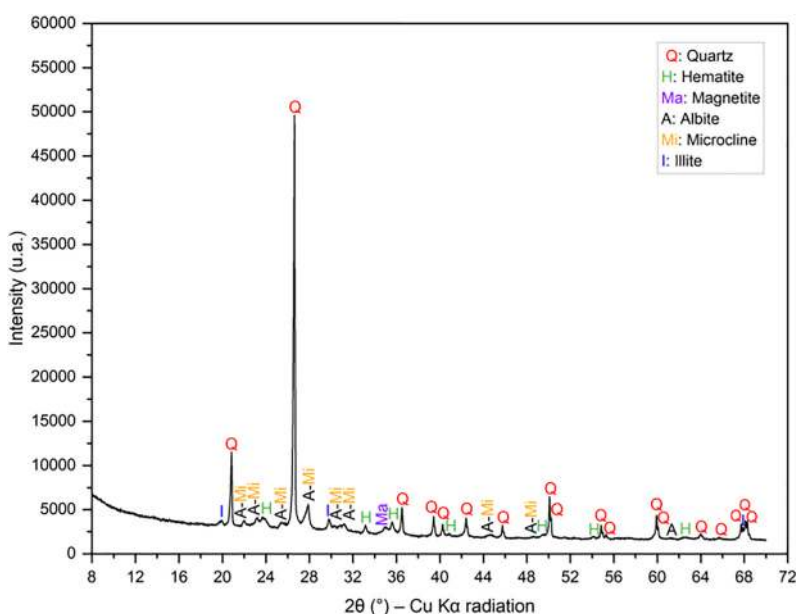


Fig. 11. XRD patterns of terracotta waste powder (TWP).

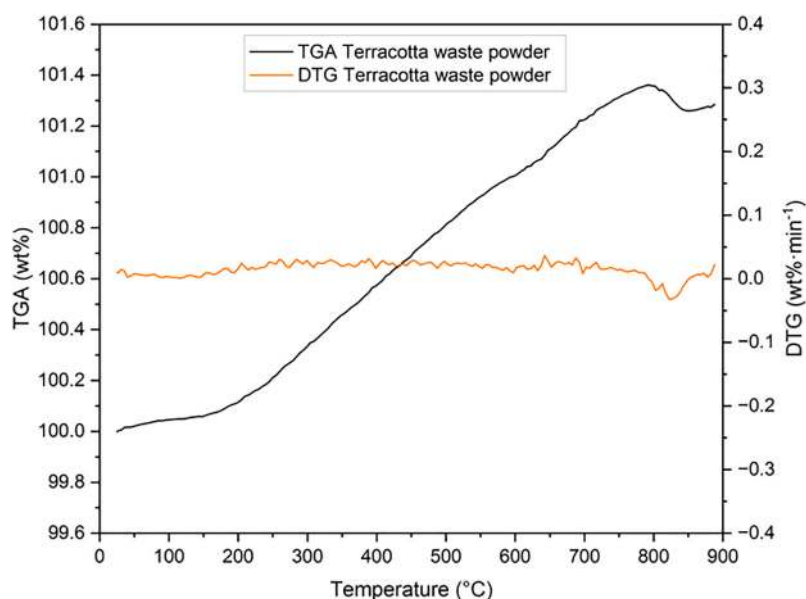


Fig. 12. TGA/DTG curves of terracotta waste powder (TWP).

from the clayey raw material and fluxes used in brick manufacturing and are partly preserved after firing. The observed peak positions and relative intensities are generally consistent with the ICDD/COD reference patterns, confirming a clay-silicate composition typical of a calcined terracotta material, in agreement with results reported in several previous studies [41,66,67].

Furthermore, the TGA and derivative thermogravimetric (DTG) curves of the TWP (Fig. 12) confirm its overall thermal stability within the investigated range, with a negligible mass change corresponding to a slight gain (approximately +1.4 %), likely linked to the progressive oxidation of metallic oxides, particularly the transition from magnetite to hematite, as reported by Soultana et al. [67] and Lamaa et al. [68], and in agreement with the oxidation mechanisms of magnetite described by Zheng et al. [69]. Overall, these findings confirm that the TWP is thermally and chemically stable, showing no decomposition or impurity-related reactions within the studied range, which is typical of already fired ceramic materials, and they support its applicability as a stable pozzolanic additive in lime-based binders.

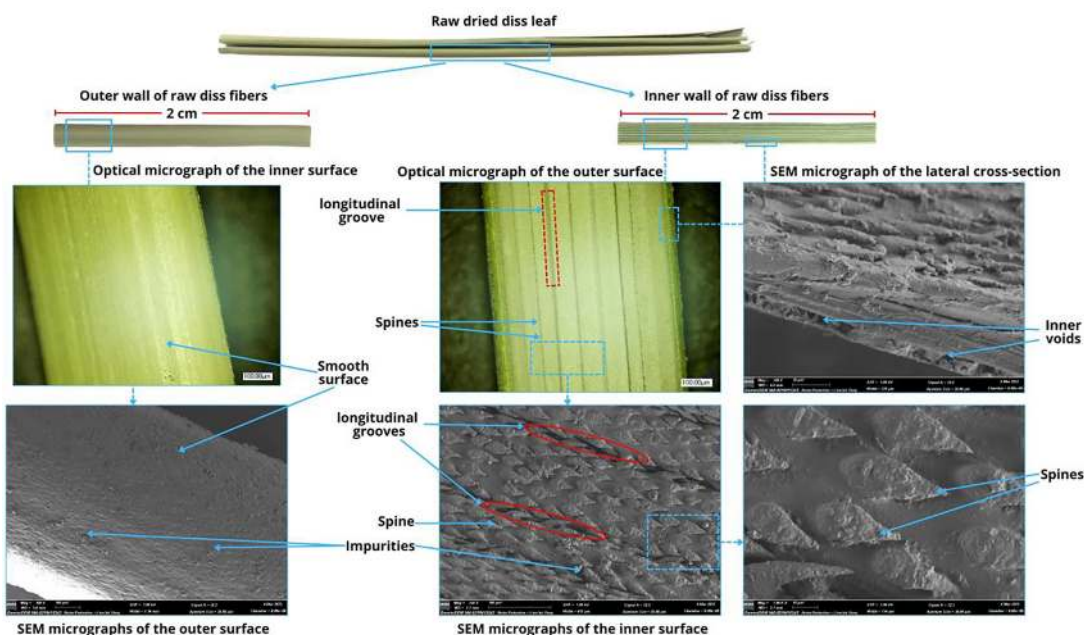


Fig. 13. Morphological characterization of raw diss fibers by SEM and Optical Microscopy.

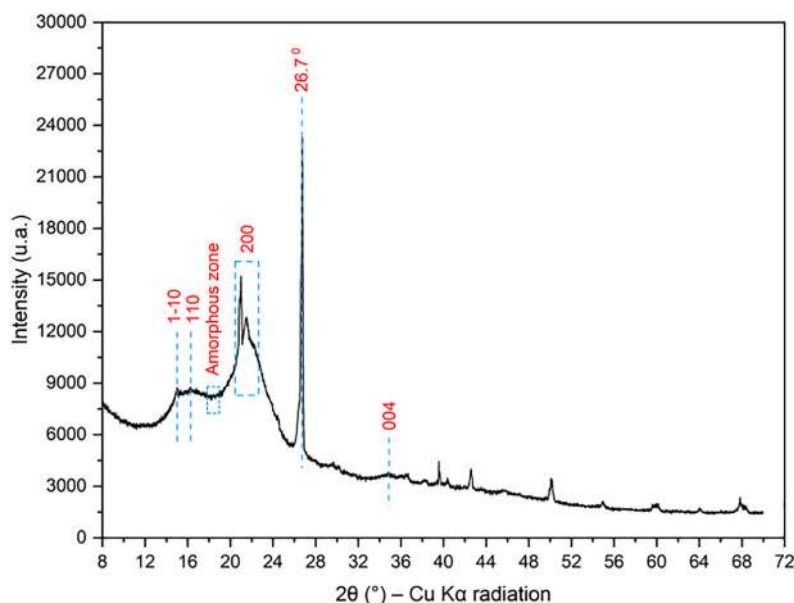


Fig. 14. XRD diffractogram of raw diss fiber.

3.1.2. Raw diss fibers

The micrographs of the leaves and fibers of diss dried in the raw state (Fig. 13) reveal the constitution and organization of the cell walls. The leaf exhibits a natural curling, resulting in closed or semi-closed curvatures. In longitudinal section, the outer surface appears smooth, while the inner surface shows a corrugated arrangement associated with a rough texture, punctuated by small sharp structures resembling spines. These are distributed along parallel reliefs several hundred micrometers in length, delimiting longitudinal grooves that confer an organized surface topography. Moreover, residual impurities, attributable to natural waxes [13], were observed and appear to be responsible for the glossy appearance noted on the inner and outer surfaces. In cross section, the lateral faces reveal the presence of internal voids organized in bundles.

The XRD diffractogram (Fig. 14) of the raw diss fibers highlights the profile of a partially crystalline lignocellulosic material. Indeed, the peaks observed at 14.98° (1-10), 16.31° (110), $21\text{--}23^\circ$ (200) and 34.86° (004) are assigned to the characteristic reflections of cellulose I, a polymorph generally considered predominant in plant fibers [70,71]. In addition, an amorphous halo between 18 and 19° , with a minimum at 18.28° , attests to the coexistence of crystalline and amorphous regions. These observations are consistent with those described for other natural fibers [72]. However, the spectrum reveals a peak at approximately 26.7° , attributable to quartz (SiO_2), a crystalline mineral often reported in raw plant fibers. The presence of silica in diss fibers has already been mentioned Assia et al. [15] and Megna et al. [21], and similar results have been observed for other untreated lignocellulosic agro-resources [73] [74]. Furthermore, the crystallinity index of the analyzed samples was estimated at 47.06 %, a value comparable to those reported in other studies on raw diss fibers [17,75,76].

3.2. Multiphysical characterization of eco-bio-based composites of TWP and diss fibers

3.2.1. Dry density

The dry densities of mixes F1–F6 are shown in Fig. 15. The values exhibit only minor variations between specimen geometries, confirming good repeatability. The reference mix F1, consisting of 85 % lime and 15 % TWP, exhibits the highest dry density ($1025.54 \pm 15.19 \text{ kg m}^{-3}$), reflecting a compact matrix without fibers. By increasing the diss fiber content from 0 % to 35 %, the dry density progressively decreases to $794.73 \pm 28.41 \text{ kg m}^{-3}$ for F6, corresponding to a reduction of about 22.5 % compared to F1. This behavior is governed by the intrinsic properties of the fibers, characterized by their low density ($\approx 140 \text{ kg m}^{-3}$) and high porosity, which contribute to a lighter composite material. At a 15 % replacement level of lime by TWP, the comparison between F2 and F3 does not indicate any significant influence on bulk density, with only a slight increase of around 2.0 % being observed, which is consistent with the higher solid density of TWP compared with lime. Likewise, the comparison between F2 and F4 shows that reducing the fiber length results in a small increase in density, on the order of 1.07 %, suggesting a slightly more compact internal structure.

3.2.2. Open porosity

Fig. 16a presents the open porosity results for formulations F1 to F6. The influence of the main formulation parameters was examined through three key comparisons.

The comparison between F2 and F3 indicates that replacing 15 % of lime with TWP has no marked effect on open porosity, aside from a modest increase of about 1.6 %. This slight variation can likely be related to an incomplete pozzolanic reaction that temporarily

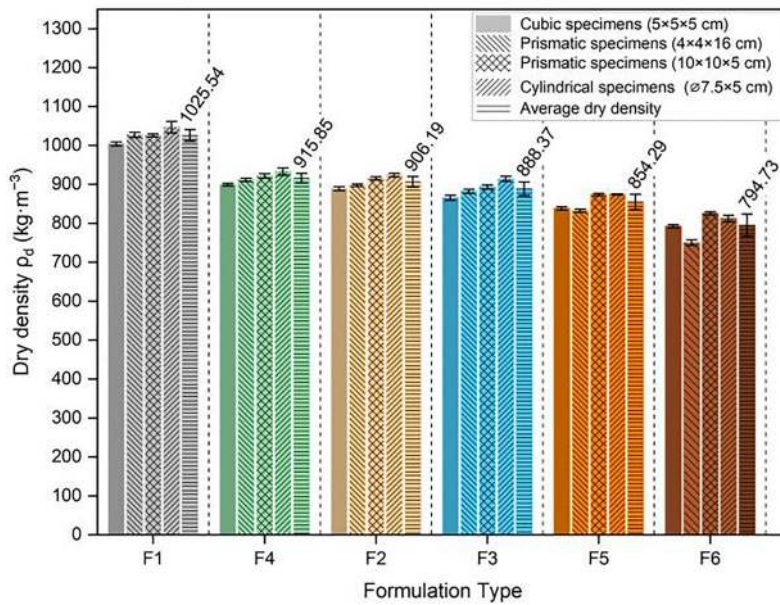


Fig. 15. Dry density ($\text{kg}\cdot\text{m}^{-3}$) of eco-bio-based composites of TWP and diss fibers (F1–F6).

limits microstructural densification, consistent with early-age behavior reported for lime-pozzolan systems [42,77], where slow reaction kinetics compete with carbonation at early stages, delaying densification before progressive pore-network refinement at later ages.

Similarly, reducing fiber size when contrasting F2 with F4 leads to a slight decrease in open porosity (about 1.41 %). Although limited, this effect may be related to denser packing and more uniform fiber coating, which can slightly increase binder demand, in agreement with Brzyski et al. [78].

By contrast, fiber content has a dominant influence, as fiber incorporation leads to a significant increase in porosity, rising from 49.40 ± 0.62 % for F1 (without fibers) to 59.24 ± 1.06 % for F6 (35 % fibers). This result confirms the structural sensitivity of the composite to the fiber-to-binder ratio (D/B). The observed increase can be attributed to the high intrinsic porosity of the fibers and their irregular morphology, which may disturb particle packing, increase the void volume, and promote fiber-matrix interfacial zones that are often more porous than the matrix [7,79]. Therefore, open porosity varies inversely with density, as shown in Fig. 16b, where the dry density of diss-based composites decreases linearly with open porosity, with a strong correlation ($R^2 > 0.95$). This trend illustrates that the bulk density of the formulations is primarily governed by the development of the open pore network induced by fiber incorporation and suggests that the open-pore fraction evolves consistently with the overall composite structure.

The composites developed in this study are generally lighter and more porous than the cementitious composites reported by Sellami et al. [19], which contained 0–30 % diss fibers by binder mass and exhibited densities ranging from 965 to 1440 $\text{kg}\cdot\text{m}^{-3}$ and porosities between 41.5 % and 51.7 %. However, they are of the same order of magnitude as the lime-alfa fiber composites reported by Garrouri et al. [7] which showed densities from 860 to 1020 $\text{kg}\cdot\text{m}^{-3}$ and porosities varying from 45 ± 2 % to 68 ± 8 %.

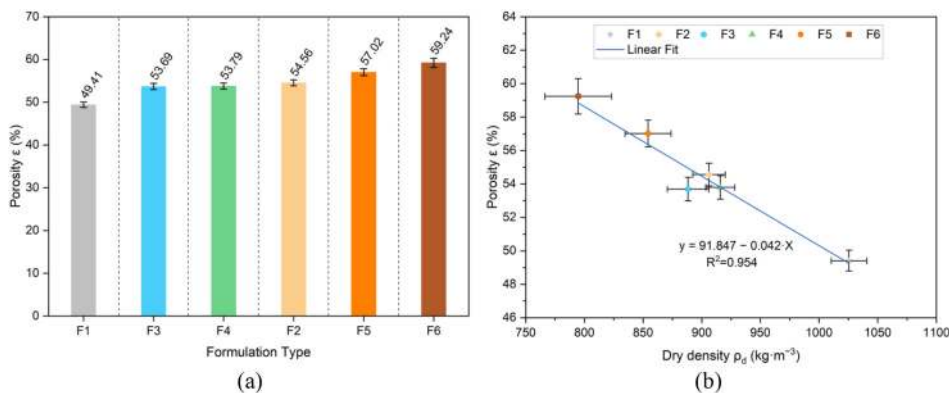


Fig. 16. (a) Open porosity (%) of eco-bio-based composites of TWP and diss fibers (F1–F6); (b) density-porosity relationship.

3.2.3. Thermal conductivity

The thermal conductivity of the diss-fiber composites was measured both parallel ($\lambda_{\parallel}$) and perpendicular ($\lambda_{\perp}$) to the manufacturing direction at $23 \pm 2^{\circ}\text{C}$ and $50 \pm 5\%$ RH (Fig. 17a). For all formulations, $\lambda_{\perp}$ was higher than $\lambda_{\parallel}$, with differences of 9–13 %, revealing thermal anisotropy. This anisotropy likely originates from the casting process, which promotes preferential fiber orientation in planes orthogonal to the manufacturing direction, thereby aligning most fibers parallel to the heat flux. In particular, the vibration-table compaction used in this study may have promoted fiber rearrangement and preferential alignment compared with manual hand-tamping. However, this comparison was not directly quantified and may depend on the applied vibration duration and intensity. Similar orientation-dependent behavior has been reported for other bio-based materials, in which compaction conditions affect particle orientation and consequently thermal anisotropy [24,80,81].

Such orientation effects influence the heat-transfer mechanisms within the material. When fibers align parallel to the flux, heat transfer follows continuous conductive paths along their longitudinal axes and along the binder-fiber interfaces, increasing the effective conductivity ($\lambda_{\parallel}$). Conversely, a series-type arrangement, where fibers are perpendicular to the flux, interrupts the continuity of the conductive network and introduces interfaces that enhance thermal resistance. The slightly higher perpendicular conductivity thus reflects partial percolation of the binder phase, as also emphasized by Frantz et al. [80].

Within the studied range (15–35 % fiber), the magnitude of anisotropy remained approximately constant, with $\lambda_{\perp}-\lambda_{\parallel}$ differences of about 11–13 % for F2, F5, and F6, regardless of fiber dosage. This implies that fiber content modifies the absolute conductivity, though not necessarily its directional dependence. However, for identical fiber fractions, anisotropy increased slightly with fiber size, reaching 11.5 % for long fibers (F2) compared to 9.6 % for short fibers (F4), indicating that larger and more elongated fibers contribute to fiber orientation and anisotropic heat transfer, as observed by Brzyski et al. [81].

Moisture influence. The effect of moisture on thermal conductivity was evaluated for $\lambda_{\parallel}$ under three relative-humidity conditions (dry, 50 %, and 85 % RH) at a constant temperature of 23°C (Fig. 17b). Regardless of composition, λ increased with humidity. The average rise was 4–8 % between the dry and 50 % RH states, and 12–16 % between the dry and 85 % RH states, confirming that water uptake partially replaces air in the pores, thereby increasing the effective conductivity. This trend is consistent with the quasi-linear relationships between λ and water content observed for all formulations (Fig. 17c) and previously reported for other lime-based

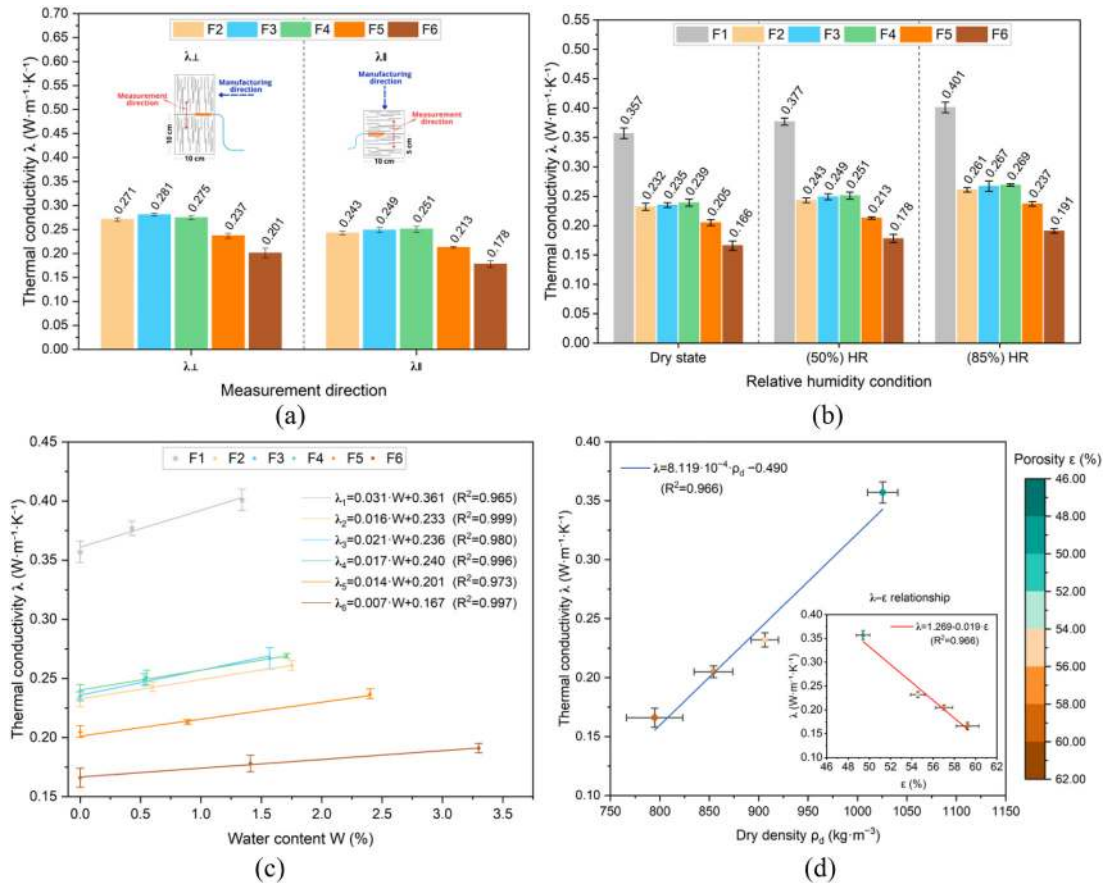


Fig. 17. (a) Thermal conductivity (λ) of formulations F1–F6 at 56 days, $23 \pm 2^{\circ}\text{C}$ and $50 \pm 5\%$ RH, measured in two testing directions; (b) $\lambda_{\parallel}$ as a function of relative humidity (0, 50, 85 %); (c) $\lambda_{\parallel}$ as a function of water content for each formulation (F1–F6); (d) $\lambda_{\parallel}$ as a function of dry density and porosity.

biocomposites [82,83].

Effect of fiber content (F1–F6). As expected, the incorporation of diss fibers significantly reduces thermal conductivity, with a more pronounced effect as the fiber-to-binder ratio increases. In the dry state, λ drops from $0.357 \text{ W m}^{-1} \text{ K}^{-1}$ for F1 (0 % fiber) to $0.166 \text{ W m}^{-1} \text{ K}^{-1}$ for F6 (35 % fibers), corresponding to a 53.5 % reduction. This decrease is mainly attributed to the intrinsic porosity of diss fibers and their low thermal conductivity ($0.059 \text{ W m}^{-1} \text{ K}^{-1}$) [20] compared with that of the binder. It also results from the overall increase in total porosity and the reduction in the apparent density of the composite, which generate additional interfaces that impede heat transfer through the matrix, thereby enhancing the material's effective thermal resistance. Similar behavior has been documented for other systems using plant fibers [9,84]. Moreover, Fig. 17d shows a marked linear relationship between thermal conductivity and the dry density of the diss composites, as well as an inverse dependence on their open porosity. These results confirm the presence of a key physical lever for optimizing thermal performance, consistent with trends reported in previous studies [85].

Effect of fiber size (F2 vs F4). Regarding fiber size, the effect on λ remains modest. When moving from F2 to F4, the reduction in fiber length is accompanied by a minor increase of about 3 %, likely related to the differences in density and porosity previously observed between these two formulations. According to the literature, particle size primarily influences thermal conductivity through the compactness and void volume of the composite, with finer particles tending to densify the structure and consequently increase thermal conductivity [78,81]. A consistent trend has been reported in ultra-lightweight concrete with miscanthus fibers, where the 2–4 mm fraction reduces λ more effectively than the 0–2 mm range, as it generates more voids and more distinct fiber-binder interfaces that limit conductive heat transfer paths [86].

Effect of TWP substitution (F2 vs F3). Under the tested conditions, replacing 15 % of lime with TWP in mixture F2 slightly reduced the thermal conductivity by about 1.3–2.5 % relative to mixture F3 (without TWP). This impact appears to be related to the ceramic nature and higher microporosity of the powder, which promote a more favorable void distribution and slightly reduce heat conduction. It can also be associated with the presence of low-reactivity particles that remain partly unreacted within the binder matrix, despite the mechanical strength improvement that will be discussed in the following sections. This finding suggests that a small insulation gain can be achieved without compromising mechanical performance. Comparable results have also been noted when brick powder is used as a binder substitute in lime-based renders [87] and in hemp-lime composites [46].

On the whole, the eco-bio-based composites developed in this study exhibit a thermal conductivity between 0.166 and $0.239 \text{ W m}^{-1} \text{ K}^{-1}$. These values are consistent with the λ value of about $0.21 \text{ W m}^{-1} \text{ K}^{-1}$ reported by Megna et al. [21] for diss-based composites formulated with a lime-glass powder binder for bulk densities of $900\text{--}950 \text{ kg m}^{-3}$, and with the range of $0.14\text{--}0.26 \text{ W m}^{-1} \text{ K}^{-1}$ obtained by Sellami et al. [19] for cementitious composites containing 20–30 % diss fibers.

According to the functional classification of lightweight concretes in RILEM LC2 [88], the various mixes produced can be classified as lightweight materials, as they have densities below 1100 kg m^{-3} . In particular, the composites containing 15 % diss fibers fall into class III insulating materials, given that they exhibit $\sigma_c > 0.5 \text{ MPa}$ and $\lambda < 0.30 \text{ W m}^{-1} \text{ K}^{-1}$.

3.2.4. Capillary water absorption

The capillary water absorption results of formulations F1–F6 are shown in Fig. 18a, which presents the absorbed mass per unit area M_w ($\text{kg} \cdot \text{m}^{-2}$), as a function of the square root of time $\sqrt{t}$ ($\text{h}^{1/2}$), and in Fig. 18b, which reports the capillary absorption coefficients C_A ($\text{kg} \cdot \text{m}^{-2} \text{ h}^{-1/2}$), measured at 5 min and 6 h, representative of the rapid and intermediate regimes.

Effect of fiber content (F1–F6). Increasing the diss fiber fraction raises capillary absorption in the formulated mixtures. After 48 h, formulation F6 (35 % fiber) reaches $26.13 \pm 0.27 \text{ kg m}^{-2}$, compared with $22.24 \pm 0.26 \text{ kg m}^{-2}$ for F1 (0 % fiber), consistent with the observed evolution of open porosity and density. This trend is in line with that reported for materials incorporating bio-aggregates, which is widely documented in the literature [79].

Regarding absorption kinetics, the initial phase (0–15 min) is characterized by steep slopes, indicating rapid water uptake, with C_A values ranging from 7.5 ± 0.1 to $13.7 \pm 0.4 \text{ kg m}^{-2} \text{ h}^{-1/2}$ as the bio-based fiber content increases from 15 % to 35 %, whereas the reference binder (F1) exhibits the lowest value ($6.3 \pm 0.2 \text{ kg m}^{-2} \text{ h}^{-1/2}$). This behavior reflects the higher surface sorptivity and pore

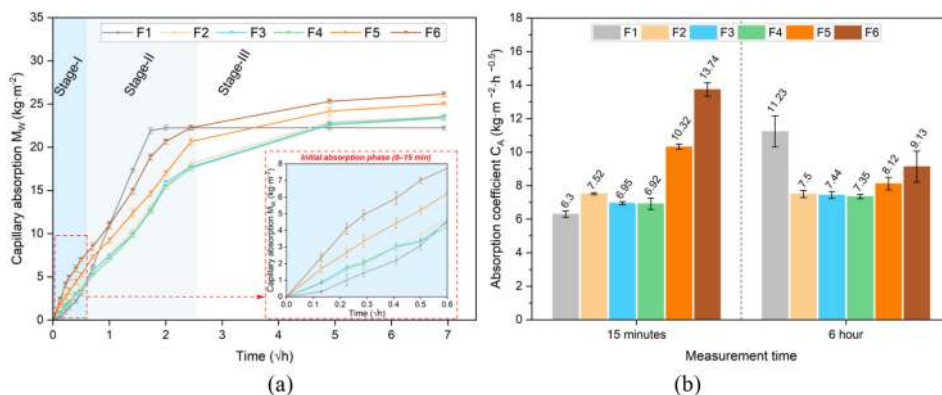


Fig. 18. (a) Capillary water absorption kinetics of formulations F1–F6 at 56 days; (b) Capillary absorption coefficient, C_A , for each formulation after 5 min and 6 h of testing.

connectivity induced by the porous, hydrophilic fibers.

The increased water uptake observed here for fiber-rich formulations can be explained both by the higher capillary porosity and by the development of a better-connected multiscale pore network consisting of the matrix microporosity and the intrinsic porosity of the fibers. In the early stages of imbibition, water is rapidly taken up near the exposed surface through the internal cavities of the fibers and the interfacial pores, possibly accompanied by slight fiber swelling, which temporarily accelerates capillary transport.

During the intermediate phase between 15 min and 6 h, the reference binder (F1) achieves saturation more rapidly, with a higher capillary absorption coefficient ($11.23 \pm 0.92 \text{ kg m}^{-2} \text{ h}^{-1/2}$), whereas the fiber-reinforced mixtures exhibit slower and more gradual kinetics. After 6 h, the absorption rate progressively decreases while approaching saturation, which is reached at around 48 h and marks the stabilization phase. This behavior likely reflects a redistribution of pores caused by fiber swelling, increased tortuosity, local blockage of capillary channels, which delays saturation. Similar mechanisms have been reported for other bio-based concretes [57].

Effect of fiber size (F2 vs F4). At the same fiber content (15 %), the short fibers used in mixture F4 slightly reduce water absorption, with a surface mass M_W about 0.8 % lower than that of mixture F2 containing longer fibers. This effect is more pronounced on the kinetics, with differences in the absorption coefficient of about 8 % at 15 min and 2 % at 6 h, indicating a moderate influence of fiber size, more marked at early times. This difference could be related to a denser packing of the short-fiber matrix, which likely increases tortuosity and reduces the continuity of capillary pathways, as well as to less extended fiber-matrix interfaces, thereby moderating the initial imbibition rate. Similar results have been reported for other biocomposites when smaller bio-aggregates were used [78].

Effect of TWP substitution (F2 vs F3). The replacement of 15 % of lime by TWP in mixture F2 does not significantly modify the capillary behavior, as only a slight increase in M_W of about 0.4 % at 48 h compared with mixture F3 without TWP was observed. However, in terms of kinetics, the absorption coefficient increased by approximately 8.2 % at 15 min and 0.8 % at 6 h, indicating that TWP primarily affects the pore-size distribution rather than pore connectivity under short-term exposure. This finding is consistent with the slight increase in open porosity previously observed and with a still limited development of the microstructure, probably due to the slow evolution of the pozzolanic reaction of TWP, which will be analyzed in more detail in the compressive strength results and confirmed by XRD, TGA, and SEM. Although only a slight effect is observed here, some studies have reported that the use of brick powder can reduce capillary uptake, for example in bio-based concretes [47] or restoration lime mortars, with an effect that remains dependent on curing age [39].

Overall, the capillary absorption coefficients measured in this study ($6\text{--}14 \text{ kg m}^{-2} \text{ h}^{-1/2}$) fall within the typical range reported for lime- and bio-aggregate-based composites [89,90]. The sensitivity analysis confirms that fiber content is the dominant parameter governing sorptivity, whereas fiber size and TWP substitution exert secondary yet measurable effects.

Nevertheless, the sensitivity of these composites to liquid water uptake should not be overlooked for envelope applications. This issue is particularly relevant under driving rain or accidental wetting, where increased material water content may affect thermal performance, favor mold growth, and compromise long-term durability. Therefore, mitigation strategies should be considered at both the envelope and material scales. At the envelope scale, protective renders or coatings can be applied to the wall surface or directly to the blocks, provided that they limit water absorption while maintaining sufficient water vapor permeability to allow drying [91]. Vapor-control measures, such as vapor retarders and ventilated air layers, may help limit condensation-related mold risks while preserving drying capacity [92], although they do not directly reduce capillary water uptake. At the material scale, formulation optimization may also be explored. Previous studies have shown that a moderate addition of hydraulic binder and water retainers can limit capillary absorption [89]. More recently, Liu et al. [93] reported that bio-based polymer additives, such as hydroxypropyl methylcellulose, can also reduce water absorption in lime-hemp composites while preserving their hygrothermal properties. In addition, bio-based antifungal systems, such as chitosan-encapsulated essential oils, have been proposed for lignocellulosic building materials and may therefore be explored to reduce fungal growth risk under prolonged high-moisture exposure [94]. Thus, further

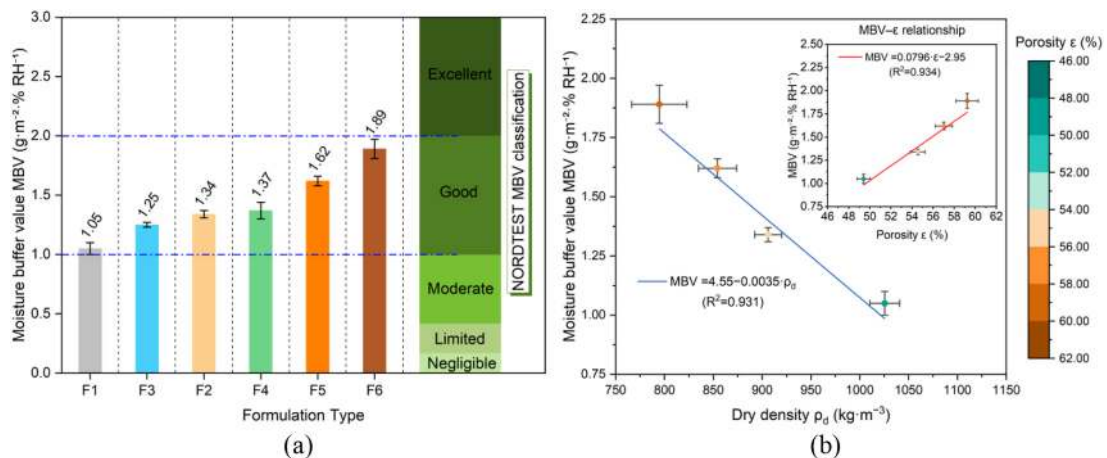


Fig. 19. Moisture Buffer Value (MBV) of eco-bio-based composites of TWP and diss fibers (F1–F6), according to the Nordtest protocol (a); relationship between MBV, dry density and porosity (b).

investigations are needed to assess the compatibility of these approaches with the developed composites, as well as their effects on material properties and long-term durability under real exposure conditions.

3.2.5. Moisture buffer value (MBV)

Fig. 19a shows the moisture buffer values (MBV) of the different formulations, determined according to the NordTest protocol and compares them with the corresponding classification.

Effect of fiber content (F1–F6). From the analysis of the data, it is clearly observed that increasing the diss fiber content markedly enhances the hygroscopic response. Formulation F6, containing 35 % fibers, exhibits the highest MBV, reaching $1.89 \pm 0.08 \text{ g m}^{-2} \cdot \% \text{ RH}^{-1}$, which corresponds to an increase of approximately 80 % compared with the F1 reference ($1.05 \pm 0.05 \text{ g m}^{-2} \cdot \% \text{ RH}^{-1}$). This rise reflects the hygroscopic character of the diss fibers, and emphasizes their crucial role in improving the hygric behavior of the composite. Moreover, Fig. 19b highlights a linear relationship, for TWP-diss composites with the same matrix, between MBV, porosity and dry density, underscoring the combined influence of these parameters on composite hygroscopicity. As porosity increases and density decreases, MBV increases, whereas the opposite trend is associated with a reduction in MBV. Similar observations have been reported in previous studies including Mazhoud et al. [82] and Juárez-Alvarado et al. [95].

Effect of fiber size (F2 vs F4). Reducing the size of diss fibers induces a slight increase in the mean mass change at equilibrium, leading to a modest improvement in moisture buffering capacity. The MBV increases from 1.34 ± 0.03 to $1.37 \pm 0.07 \text{ g m}^{-2} \cdot \% \text{ RH}^{-1}$, which is about 2.2 %, reflecting a small yet positive effect on the hygrometric regulation of the composite. Convergent observations have been reported in several similar systems. For hemp-based composites, finer particles reach hygrometric equilibrium more rapidly and promote better moisture control, as indicated by Mahmood et al. [24]. Likewise, Mazhoud et al. [96] reported a markedly higher MBV with fine shives, attributing this effect to increased moisture storage associated with hysteresis. This enhancement can also be associated to the larger specific surface area of finer particles, as also emphasized by Gbekou et al. [79].

Effect of TWP substitution (F2 vs F3). At identical fiber content and size, composite F2, whose binder phase includes 15 % TWP, exhibits higher performance than formulation F3 made with lime only. The MBV increases from 1.25 ± 0.02 to $1.34 \pm 0.03 \text{ g m}^{-2} \cdot \% \text{ RH}^{-1}$, corresponding to a gain of about 7.2 %, reflecting the beneficial effect of TWP. This progression suggests an enhanced hygroscopic affinity due to the contribution of TWP and, possibly, a pore structure more favorable to moisture storage and subsequent

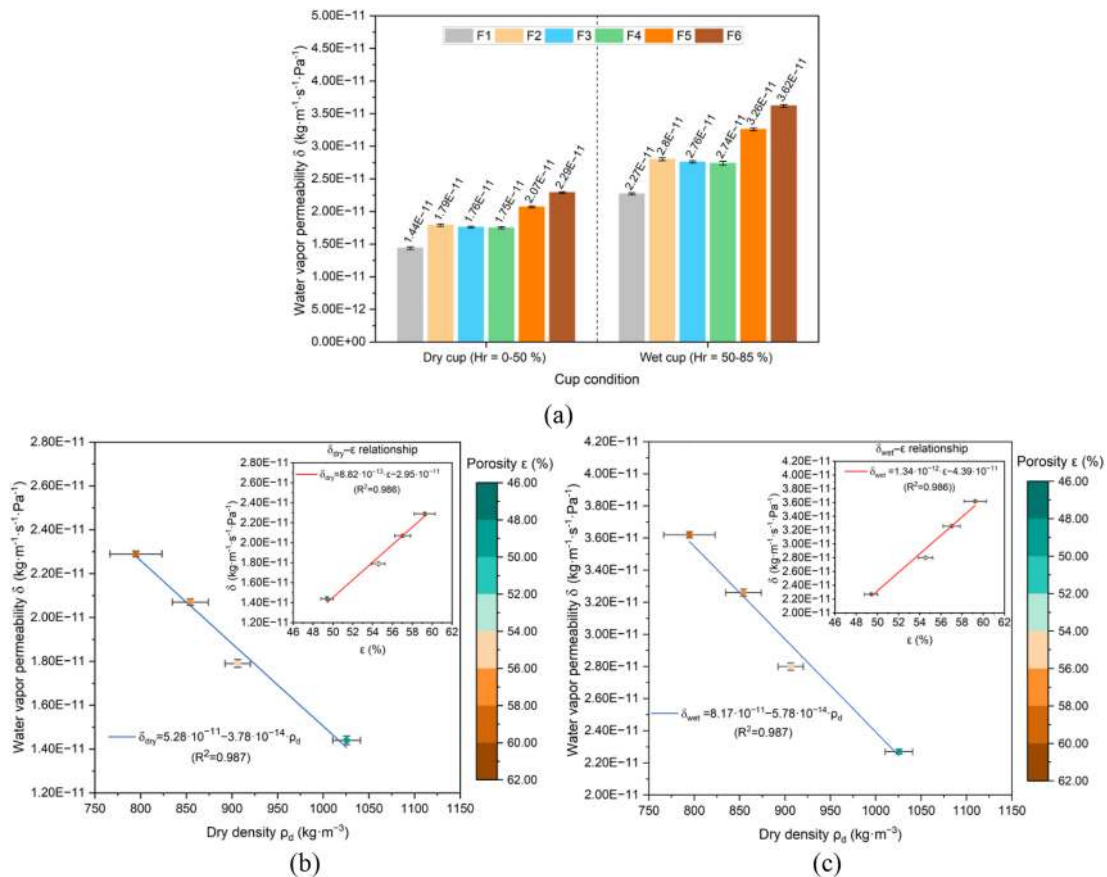


Fig. 20. (a) Water vapor permeability (δ) of TWP-diss formulations F1–F6 under dry-cup (0/50 % RH) and wet-cup (50/85 % RH) (b–c) effect of dry density and open porosity on δ_{dry} and δ_{wet} .

release within the composite. This result indicates a synergy between TWP and diss fibers, leading to improved hygric regulation. Comparable results have been reported for hemp-lime composites incorporating 10 % brick powder, which exhibit a higher buffering capacity than formulations containing metakaolin or based on hydraulic and hydrated lime [46].

For all formulations containing TWP and diss fibers, the mean MBV values range from 1.34 to 2.00 $\text{g m}^{-2} \cdot \% \text{RH}^{-1}$. According to the NORDTEST classification, these materials can be regarded as good moisture buffers. The measured MBV levels are higher than those typically reported for conventional building materials such as gypsum, ordinary concrete, and aerated concrete [59]. In some cases, they also exceed the values reported for diss-based composites by Zaid [28] (1.25–1.4 $\text{g m}^{-2} \cdot \% \text{RH}^{-1}$), while remaining within the range documented for certain bio-aggregate-based composites, such as miscanthus-fiber composites, which reach 1.38–2.05 $\text{g m}^{-2} \cdot \% \text{RH}^{-1}$ [79].

3.2.6. Water vapor permeability

Fig. 20a presents the water vapor permeabilities of the different formulations, determined under dry-cup (0/50 % RH) and wet-cup (50/85 % RH) configurations. Wet-cup values are overall higher than dry-cup values. This difference results from the larger humidity gradient, which induces liquid-phase transport within pores saturated by capillary condensation and, in turn, enhances vapor migration and diffusion at the material scale, consistent with previous results reported for other materials [96,97].

The analysis indicates that the reference formulation F1, representing the matrix alone and composed of 85 % lime and 15 % TWP, exhibits the lowest water vapor permeability among all mixes, with $1.44 \times 10^{-11} \text{ kg m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$ in the dry cup and $2.27 \times 10^{-11} \text{ kg m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$ in the wet cup. These levels are of the same order of magnitude as those reported for mortars based on lime [98] and for mortars incorporating brick waste [99].

Effect of fiber content (F1–F6). As the mass fraction of diss fibers relative to the binder increases, the permeability of the composite increases under both test conditions. Between 0 and 35 % fibers, the permeability increases by about 59 % for F6 compared with the reference F1, reaching $2.29 \times 10^{-11} \text{ kg m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$ in the dry configuration and $3.62 \times 10^{-11} \text{ kg m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$ in the wet configuration. This evolution can be explained by more efficient moisture transfer resulting from a more open and continuous pore network, as well as increased intergranular macroporosity due to diss fibers, whose morphology is naturally porous, as shown for other bio-based materials [89,100].

In line with these mechanisms, Fig. 20b at 20c clearly show that at both humidity levels the permeability varies linearly, increasing with open porosity and decreasing with dry density. Comparable trends are reported for materials based on straw, lime hemp concrete, as well as earth and limestone [93,101–103]. However, these studies report regressions that are sometimes linear, sometimes power law, and sometimes exponential, expressed either by the permeability δ or by the water vapor diffusion resistance factor μ . Differences between studies may arise from variability in materials, compositions, and manufacturing routes, which modify the microstructure and the macrostructure and, consequently, water vapor permeability. This interpretation is supported by analogy with the work of Collet et al. [25] for hemp concrete, showing an exponential response to relative humidity for sprayed and prefabricated elements, whereas a power law is observed for cast elements. Generally, the water vapor permeability values measured in this study, ranging from approximately 1×10^{-11} to $4 \times 10^{-11} \text{ kg m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$, remain comparable to those reported for other plant fiber composites in the literature [79,104].

Effect of fiber size (F2 vs F4). Reducing the fiber size in formulation F4 compared with F2 resulted in a slight decrease in permeability of about 2.2 % at both applied humidity levels. Although this influence is limited, it can be explained by the shorter fibers generating a more compact pore structure and thus greater resistance to vapor transport, in agreement with previous findings by Zhou

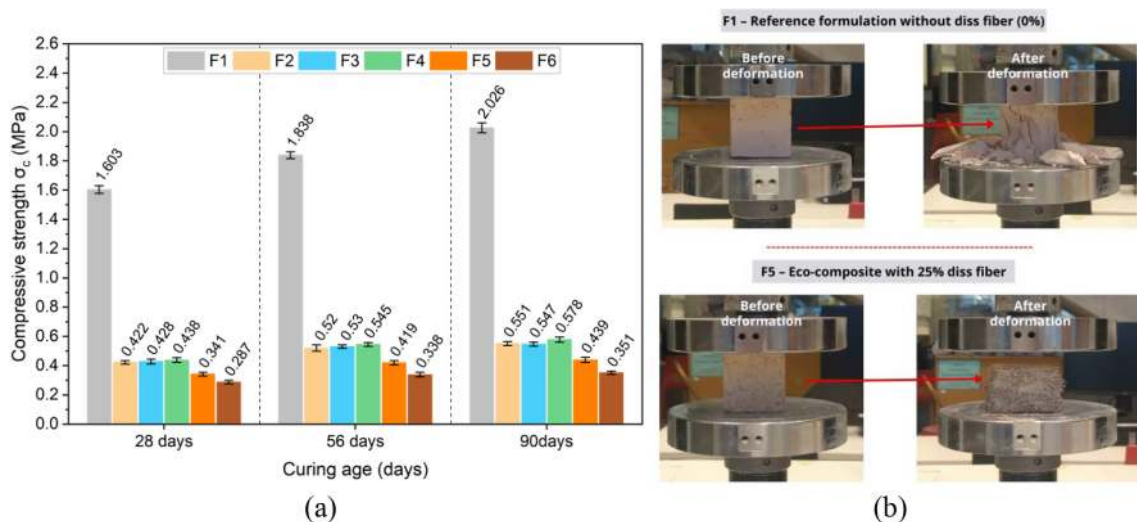


Fig. 21. (a) Compressive strength of formulations F1–F6 at 28, 56 and 90 days; (b) photographs of eco-bio-based composites F1 and F5 before and after the compression test.

et al. [105] and Mazhoud et al. [96]. However, at high relative humidity, some studies have shown that composites formulated with shorter bio-fractions can exhibit slightly higher permeability, whereas this effect remains negligible at low relative humidity [78].

Effect of TWP substitution (F2 vs F3). The replacement of 15 % of the lime with TWP in formulation F2 results in only a slight increase in water vapor permeability, on the order of 1.4–1.7 % depending on the humidity level, a variation of little significance. Under these conditions, the permeability appears to depend mainly on intergranular macroporosity rather than on the microporosity of the matrix, in agreement with the observations of Walker and Pavia [89]. Other studies have shown a marked influence of binder type on water vapor permeability, since the use of a hydraulic binder can significantly reduce it [33]. In general, controlling water vapor permeability is critical to prevent condensation in walls. Future research should quantify specific surface area, the distribution of micro- and macropores, and their connectivity, in order to better capture their effect on the hygrothermal behavior of bio-based composites. The study of fungal growth and long-term performance is also a priority for durability.

3.2.7. Compressive strength

The evolution of compressive strength for formulations F1–F6 at 28, 56, and 90 days is shown in Fig. 21a. In all cases, compressive strength increases with curing time, reflecting the progressive consolidation of the binder.

Effect of TWP substitution (F2 vs F3). Partial replacement of hydrated lime with 15 % TWP has no significant impact on mechanical performance. Compared with the reference lime-only formulation (F3), the mix containing 15 % TWP (F2) shows a slight decrease of about 1–2 % in compressive strength up to 56 days, followed by a slight gain (+0.7 %) at 90 days. The limited magnitude of these differences confirms that mechanical strength is essentially maintained and suggests only a marginal late-age improvement. This trend can be attributed to the slow kinetics of pozzolanic reactions between the aluminosilicate phases in TWP and portlandite, leading to the gradual formation of hydrates that progressively densify the matrix over time. This mechanism is consistent with the behavior reported for composites formulated with lime-pozzolan binders [46,51]. Under ambient conditions, carbonation may also accompany this process and support strength development over several months [53].

Effect of fiber size (F2 vs F4). Fiber size shows a slight influence on the compressive response. The use of short fibers (<12 mm) in mix F4 leads to a modest improvement of about 3.8–4.9 %, regardless of curing age, compared with composite F2 incorporating long fibers (<25 mm). This gain is likely due to a denser packing of short fibers, which reduces inter-fiber porosity, ensures a more homogeneous coverage of the matrix, and promotes more efficient load transfer. A similar trend has been reported for biocomposites incorporating shorter bio-aggregate particles [24,78]. This result indicates that fiber geometry and size play a secondary, yet still relevant, role in optimizing fiber arrangement and packing density, as well as in enhancing stress transfer within the composite.

Effect of fiber content (F1–F6). The results indicate that increasing the fiber-to-binder ratio (D/B) considerably reduces compressive strength, illustrating the trade-off between improved insulation efficiency and reduced mechanical compactness. Between F1 (0 % fiber) and F6 (35 % fiber), compressive strength decreases by about 82–83 % at all testing ages. This decline can be attributed to the increase in total porosity induced by the high fiber content, which modify the material structure and pore network, reduce the continuity of the binding phase, and lower the overall density, thereby limiting the efficiency of load transfer within the mineral skeleton. These findings are consistent with those reported by Daher et al. [34] and Garrouri et al. [7] for composites with increased bio-based fractions. Moreover, higher fiber contents also change the deformation mode, which shifts from a brittle failure of the binder to a more gradual crushing of the composite, characterized by height reduction and lateral swelling (Fig. 21b).

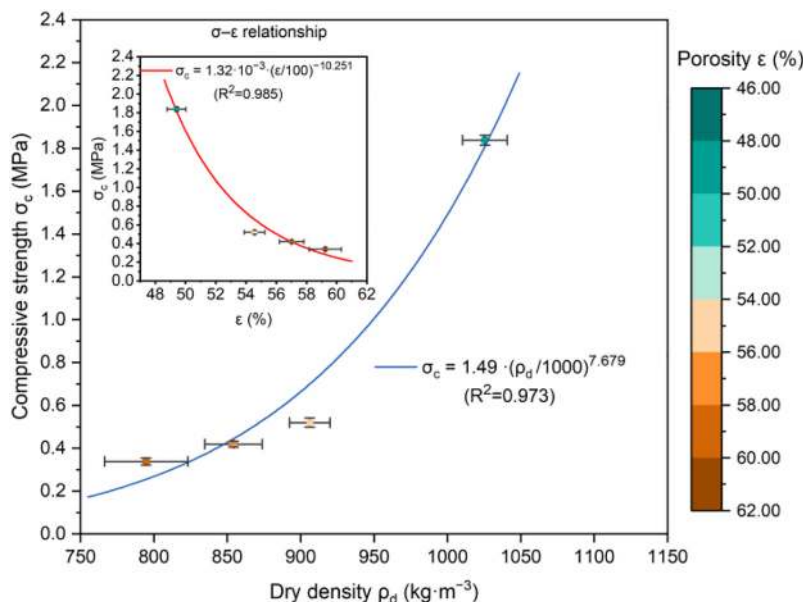


Fig. 22. Compressive strength as a function of dry density and porosity for eco-bio-based composites of TWP and diss fibers.

Correlations with structural parameters. Compressive strength exhibits a power-law relationship with dry density or open porosity, as shown in Fig. 22. Strength increases with density and decreases with porosity, with high correlation coefficients ($R^2 \geq 0.95$), confirming this strong dependence. This behavior is consistent with that reported for other porous composites such as wood concrete [106] and aerated concrete [107], and further evidences the strong structure-property coupling in this system.

In practical terms, the material appears promising. However, given its modest compressive strengths (≈ 0.3 – 0.6 MPa), it remains more suitable for non-structural applications, such as insulating or infill blocks, particularly in non-load-bearing vertical wall applications, in line with the recommendations of the French hemp concrete construction rules [108]. Further optimization pathways can be envisaged through fiber volume reduction, controlled addition of hydraulic binder to enhance matrix cohesion [109], and carefully managed compaction during casting, which may partially offset the strength loss associated with high bio-aggregate contents without increasing the binder dosage [110].

Overall, the measured mechanical performance remains lower than that reported by Sellami et al. [19] for a cementitious composite incorporating 30 % diss fibers, which achieved 0.93 MPa at a density of 965 kg m^{-3} . However, the obtained values are comparable to those reported for other bio-aggregate-based materials of similar density, such as the lime-date palm fiber mixes studied by Belakroum et al. [9], with compressive strengths of 0.2–0.6 MPa for densities of 680 – 968 kg m^{-3} .

3.2.8. Flexural strength

Since the failure of construction materials often initiates in tension, the flexural test, which induces an indirect tensile state, is a relevant indicator of cracking and in-service damage risk. In this study, it provides insight into the performance of the developed formulations and the influence of the main study parameters. Fig. 23a presents the flexural strength values measured for each mixture.

When the fiber content varies from 15 % to 35 %, a reduction in flexural strength of about 57 % to 79 % is observed compared with the reference mix (F1). This decrease is mainly related to the increase in total porosity and the decrease in binder fraction, which together reduce the effective load-bearing cross-section and the continuity of stress-transfer paths. Failure behavior is therefore governed mainly by fiber pull-out mechanisms rather than abrupt cohesive failure of the mineral matrix. The resulting composite exhibits a more ductile mechanical response, characterized by progressive crack propagation and crack-bridging phenomena that delay final rupture, while leading to a lower ultimate load (Fig. 23b). These mechanisms are consistent with those described in the literature on biocomposites with high plant-fiber contents [7,34].

Consistent with the compression results, substituting 15 % of lime with TWP does not significantly affect the flexural strength. A marginal improvement of about 1 % is observed between F2 (15 % TWP) and F3 (0 % TWP), likely linked to the slight densification of the matrix resulting from pozzolanic interactions between lime and TWP at later ages. However, at identical fiber content, the F2 composites containing long fibers exhibit about 9 % higher flexural strength than the F4 composites with short fibers, in agreement with previous observations [78]. This trend can be explained by the larger contact area of the longer fibers, which ensures better adherence with the binder and provides greater anchorage length, allowing improved stress transfer and tensile mobilization within the bending zone. Moreover, the particle-size homogeneity of the bio-based phase can modulate orientation and anchorage mechanisms and can therefore amplify or reduce flexural capacity, depending on dispersion and alignment [111].

3.2.9. X-ray diffraction analysis

The hydration and carbonation reactions of the mineral matrices of composites F1, F2 and F3 were identified by XRD at 56 and 90 days of curing. The corresponding diffractograms are presented in Fig. 24. For matrix F3 composed solely of hydrated lime the main mineralogical constituents are portlandite $\text{Ca}(\text{OH})_2$ (ICDD: 00-044-1481) and calcite CaCO_3 (ICDD: 01-072-1937). Between 56 and 90 days the decrease in portlandite peak intensities together with the increase in calcite indicates progressive partial carbonation under ambient CO_2 . In the matrices of composites F1 and F2 formulated with 85 % lime and 15 % TWP these two phases are also observed. The detection of quartz SiO_2 (ICDD: 01-088-2302) evidences the siliceous contribution of TWP. In addition reflections detected at

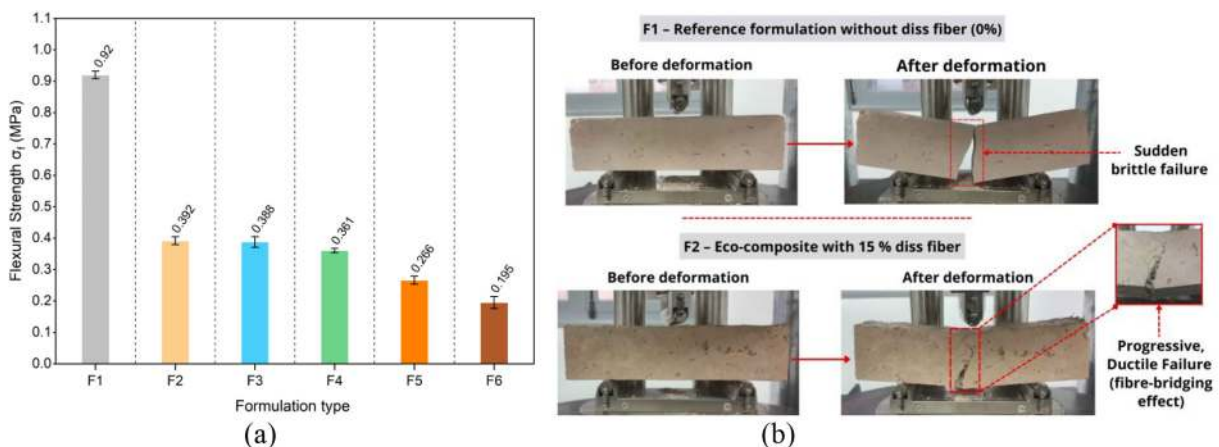


Fig. 23. (a) Flexural strength of formulations F1–F6 at 56 days; (b) photographs of F1 and F2 before and after the three-point bending test.

27.82°, 31.25° and 35.82°, as well as those located at 42.38° and 60.06° (2 θ , Cu K α), are consistent with positions reported for calcium silicate hydrate (C–S–H) (ICDD: 00-043-1488), as widely documented in the literature [112–114]. Moreover signals detected around 11.61°, 23.22°, 31.25° and 35.82° can be assigned to calcium aluminosilicate hydrate gels (C-(A)-S-H) (ICDD: 01-079-0677), in agreement with previous results [112,115,116].

Consequently, in the matrices of F1 and F2, the partial substitution of lime by TWP induces the formation of C–S–H and C–(A)–S–H gels, which are not detected in the pure lime matrix (F3). This confirms the pozzolanic reactivity of TWP through the reaction of its aluminosilicate phases with portlandite. The resulting hydrates enhance matrix densification and cohesion, ensure long-term microstructural stability, improve mechanical properties, and contribute to limiting the friability of the carbonated binder, particularly in hybrid lime-TWP matrices.

As curing progresses, the intensification of these gel signals is accompanied by a decrease in $\text{Ca}(\text{OH})_2$ and SiO_2 , indicating that the pozzolanic reaction remains active. Although XRD peak intensities may be affected by preferred orientation and sample heterogeneity, the consistent trend across formulations indicates an actual consumption of portlandite and a growth of secondary hydrates. This evolution refines the microstructure and results in a gain in mechanical strength, in agreement with the compressive strength results obtained for mixtures containing TWP. In parallel, the detection of calcite confirms that carbonation also occurs through reaction with ambient CO_2 . Portlandite reflections in F1 and F2 remain weaker than in F3, which is consistent with the combined action of pozzolanic and carbonation mechanisms.

Finally, it is essential to note that the presence of diss fibers likely has a limited effect on the development of pozzolanic activity, as it appears to reduce the intensity of certain hydrate reflections in F2 compared with F1, suggesting a slight delay in the reaction process.

3.2.10. Thermogravimetric analysis (TGA/DTG)

Thermogravimetric (TGA) and derivative (DTG) analyses were carried out on the mineral matrices of composites F1, F2, and F3 at 56 and 90 days to identify the main decomposition steps and to complement the XRD results. The tests also aimed to assess the evolution of the matrix with the addition of TWP, as well as its interaction with diss fibers. The corresponding curves are shown in [Fig. 25](#). On the whole, the effect of adding TWP results in increased portlandite consumption and the formation of new hydrates with curing age, confirming its pozzolanic reactivity and the continuation of the reaction over time. Fibers induce only a slight initial delay

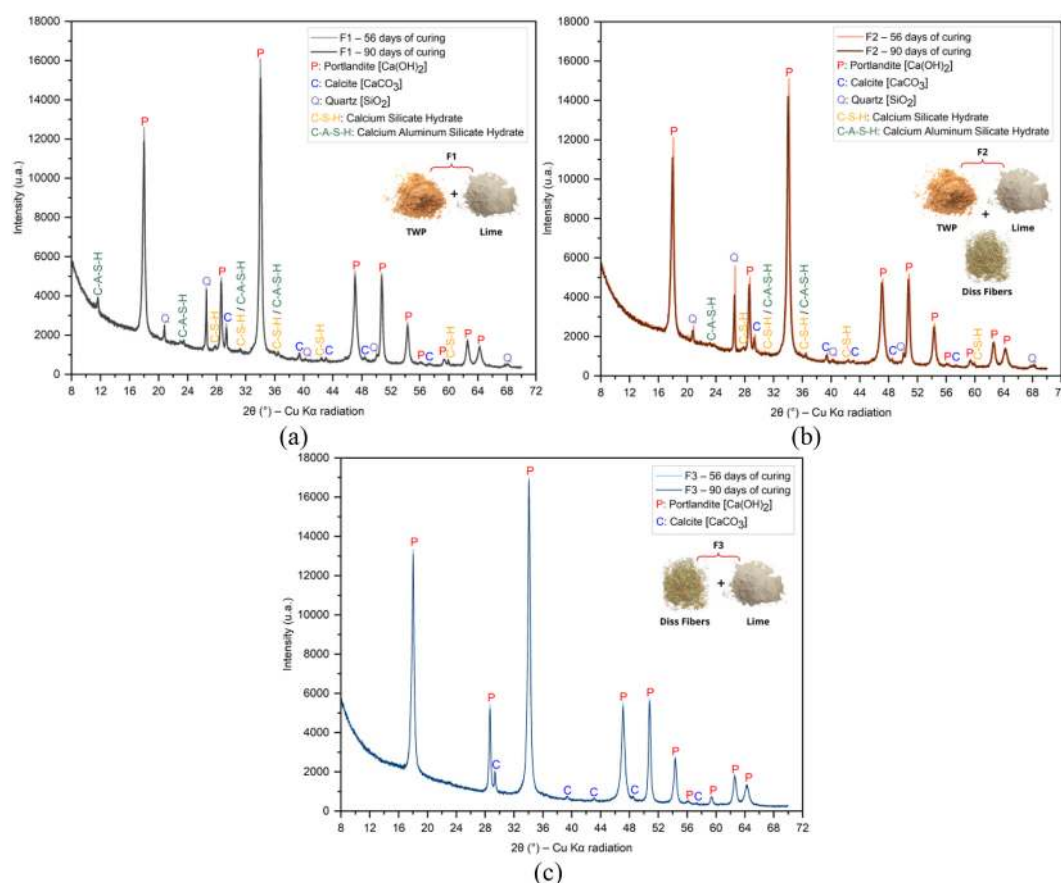


Fig. 24. X-ray diffraction patterns of the matrices of mixes F1 (a), F2 (b) and F3 (c) at 56 and 90 days.

in pozzolanic reactions.

Based on the TGA/DTG curves, four main stages can be distinguished in order to describe in detail the observed mass loss among the different tested matrices.

First stage (25–120 °C). A slight mass loss is observed in all tested matrices and is attributed to the release of evaporable water from the pore network [117,118]. In the pure lime matrix (F3), this loss is about 0.7 % at both ages and is essentially completed by 120 °C, whereas in the matrices F1 and F2, which contain 15 % TWP it continues beyond this temperature.

Second stage (120–370 °C). In this range, F1 and F2 exhibit a progressive additional mass loss, which can be primarily associated with the dehydration of pozzolanic hydrates (C–S–H and C–(A)–S–H) [119,120]. The total mass loss reaches 1.34–1.45 % for F1 and 0.92–1.32 % for F2, with values increasing with curing age, while F3 does not exhibit any pronounced additional loss. The progressive rise in mass loss over this range with curing time reveals the advancement of the pozzolanic reaction and the development of a more evolved microstructure in mixes containing TWP compared with the pure lime system, confirming the pozzolanic behavior of TWP.

Third stage (370–490 °C). Over this temperature interval, the lime matrix F3 exhibits mass losses of 17.2–18.8 % at 56 and 90 days, with a distinct DTG peak near 450 °C attributed to portlandite dehydroxylation to CaO and H₂O, as commonly reported in the literature [118,121]. These values exceed those of matrices containing TWP, namely F2 (14.2–15.7 %) and F1 (13.8–14.0 %), at the same ages, indicating greater portlandite consumption in F1 and F2 due both to the pozzolanic reaction with TWP and to simultaneous carbonation with ambient CO₂.

Fourth stage (550–880 °C). The degree of carbonation appears in this temperature range as a DTG peak assigned to calcite decarbonation CaCO₃ [121,122]. The associated loss indicates calcite contents of about 5.3–7.2 % for F3, 5.4–7.9 % for F2, and 3.89–5.2 % for F1 at 56 and 90 days, respectively. This difference is likely linked to the presence of diss fibers in F2 and F3, whose alkaline degradation can release CO₂ within the matrix and, over time, promote the formation of calcium carbonates, as reported for other bio-aggregates [123].

A negligible mass gain, sometimes detected around 570–610 °C in samples F3 and F1, is probably related to transient carbonation of CaO by CO₂ uptake, which may briefly and rapidly prevail over the calcination of CaCO₃ [124]. It may also arise from slight oxidation of unburnt carbon or trace iron-bearing phases [125,126].

Effect of diss fibers on the pozzolanic reaction (F1 vs F2 in the 120–370 °C range). At 56 days, the F2 matrix taken from the mixture containing diss fibers shows a lower amount of hydrates than the fiber-free reference matrix F1, whereas this gap narrows and becomes less pronounced at 90 days. This evolution indicates that these lignocellulosic species induce a slight initial delay in the pozzolanic reaction kinetics and hydrate formation, with a limited effect at later ages. This observation is consistent with the previously discussed XRD results and with reports on other bio-aggregates, notably lavender straw incorporated in a mineral pozzolanic binder [123]. Although a pozzolanic matrix was used, inhibition of binder hydration by organic compounds already reported in the literature [13, 16], does not appear to be fully mitigated, particularly at early ages.

Accordingly, this delay may be related to the release of soluble fiber extractives into the pore solution, where alkaline conditions may remain sufficient during the initial reaction period to promote their dissolution. Previous studies also show that the interaction with mineral binders may depend on the nature of the bio-aggregate, mainly due to differences in the content of soluble extractives [127]. These extractives may include simple sugars, polysaccharidic compounds, such as pectins and hemicelluloses, organic acids, such as acetic and uronic acids, and phenolic compounds [13,123]. Once released, these compounds can interfere with early binder reactions through adsorption, complexation, or precipitation mechanisms. In particular, pectins and organic acids are relevant because their carboxylate groups can interact with Ca²⁺ ions and form calcium-organic complexes. This may reduce calcium availability and delay the nucleation and growth of C–S–H and C–(A)–S–H hydrates, as reported in other bio-composites using lime or lime-pozzolan binders [128,129]. Therefore, the initial delay is most likely related to the combined effect of soluble fiber extractives, with the contribution of each extractive depending on its presence and relative content in the fiber composition.

Further investigations should be carried out on the same matrix using other agricultural resources commonly employed in bio-composites and reputed to be weakly perturbing, such as hemp, in order to compare the magnitude of the influence. Additional tests are also advisable using fiber pre-treatments designed to remove extractives without compromising hygrothermal properties.

3.2.11. Scanning electron microscope (SEM) observation and optical microscopy

At the microstructural level, analysis of the SEM micrographs in Fig. 26 corroborates the XRD and TGA findings for the matrices of F1, F2 and F3. Portlandite Ca(OH)₂ is clearly observed as hexagonal platelets and calcite CaCO₃ occurs as rhombohedral crystals, consistent with observations reported in the literature [130]. The increasing amount of calcite attests to progressive carbonation during curing and is most pronounced in F2 and F3 containing diss fibers.

The matrices of F1 and F2 composed of 85 % lime and 15 % TWP differ from F3 100 % lime through the emergence of new flocculent gel particles together with fibrillar textures and needle-like forms of variable length, sometimes interconnected and sometimes appearing as foil like plates. These morphologies are attributed to amorphous C–S–H and C–(A)–S–H gels, which are difficult to distinguish by imaging alone, in line with numerous studies [119,131,132]. Taken together, the observations confirm the pozzolanic reaction between portlandite and the aluminosilicate phases of TWP and indicate progressive densification of the microstructure over time. These constituents are also visible on the surface of diss fibers in F2 with a gel coating and a remarkable matrix fiber cohesion that strengthens during curing.

Optical microscopy observations were carried out on several surface areas of the specimens, in particular on composite F2. The corresponding images are presented in Fig. 27. The material exhibits a heterogeneous morphology, consisting of diss fibers coated and bonded by a mineral matrix composed of 85 % lime and 15 % terracotta waste powder. The fiber matrix bond is remarkable. Internal matrix pores and intergranular pores of different sizes are present, sometimes filled with air, as well as a few voids sometimes located at

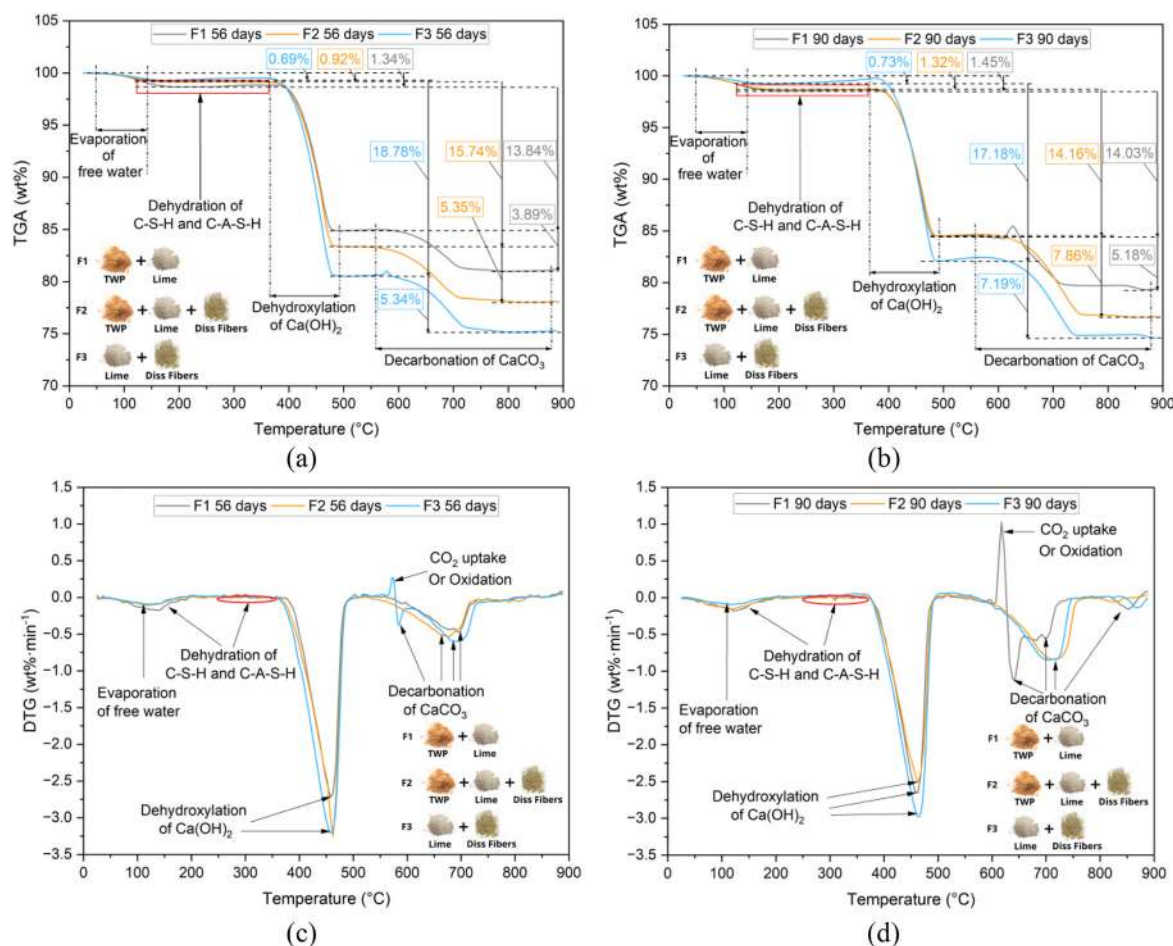


Fig. 25. (a-b) TGA and (c-d) DTG of the matrices of mixes F1, F2 and F3 at 56 and 90 days.

the fiber matrix contact. The isolated fiber extracted from the composite is covered with binder, which fills the longitudinal grooves and the interstices between the surface spines, indicating effective coating, good anchorage, and good cohesion with the matrix.

3.2.12. Impact analysis and synthesis of study results

Fig. 28 synthesizes the combined influence of diss fiber content and fiber length, as well as the partial substitution of lime with terracotta waste powder, on the physical, mechanical, thermal and hygrothermal properties of the composite discussed in this work for the different formulations developed. A detailed analysis of the individual effects of each parameter is presented in the following sections.

Effect of fiber content (F1–F6). The diss fiber content is the dominant parameter among those investigated, with a marked impact on all measured properties. Increasing the D/B ratio from 0 to 30 % leads to a decrease in dry density (−22.5 %) and an increase in open porosity (+19.9 %), which strongly enhances hygrothermal performances, particularly moisture buffering (+80 %), thermal insulation (+53.5 %), and water vapor permeability (+59 %). This high sensitivity is consistent with the intrinsic porosity and hygroscopicity of the fibers, combined with the increase in inter-particle voids, which together promote both air entrapment and vapor-water exchanges. In return, mechanical performances drop sharply (−78.8 % to −82.6 %), due to the reduced binder compactness and increased porosity, which is consistent with trends commonly reported for composites with high bio-aggregate content. Capillary absorption also increases (+17.5 %), with a strongly accelerated initial kinetics (up to +118 %), indicating the development of a more continuous and open pore network.

Effect of fiber size (F2 vs F4). The influence of fiber size remains globally moderate for all properties, with differences generally limited to a few percent, although still revealing exploitable trends. Short fibers induce a slightly more compact structure, with lower open porosity and slightly higher dry density (≈ 1.1 – 1.4 %), reflecting better packing and a more homogeneous coating by the paste. This increased compactness translates into a slight decrease in water vapor permeability (≈ 2.2 %), suggesting a tighter pore network, while thermal conductivity increases slightly (≈ 3 %), in line with the reduction in void volume and the improved continuity of the conductive phase. In terms of hygric behavior, moisture buffering improves slightly ($MBV \approx +2.2$ %), possibly related to the higher specific surface area and enhanced hygroscopic exchange capacity of short fibers. Mechanically, compressive strength increases

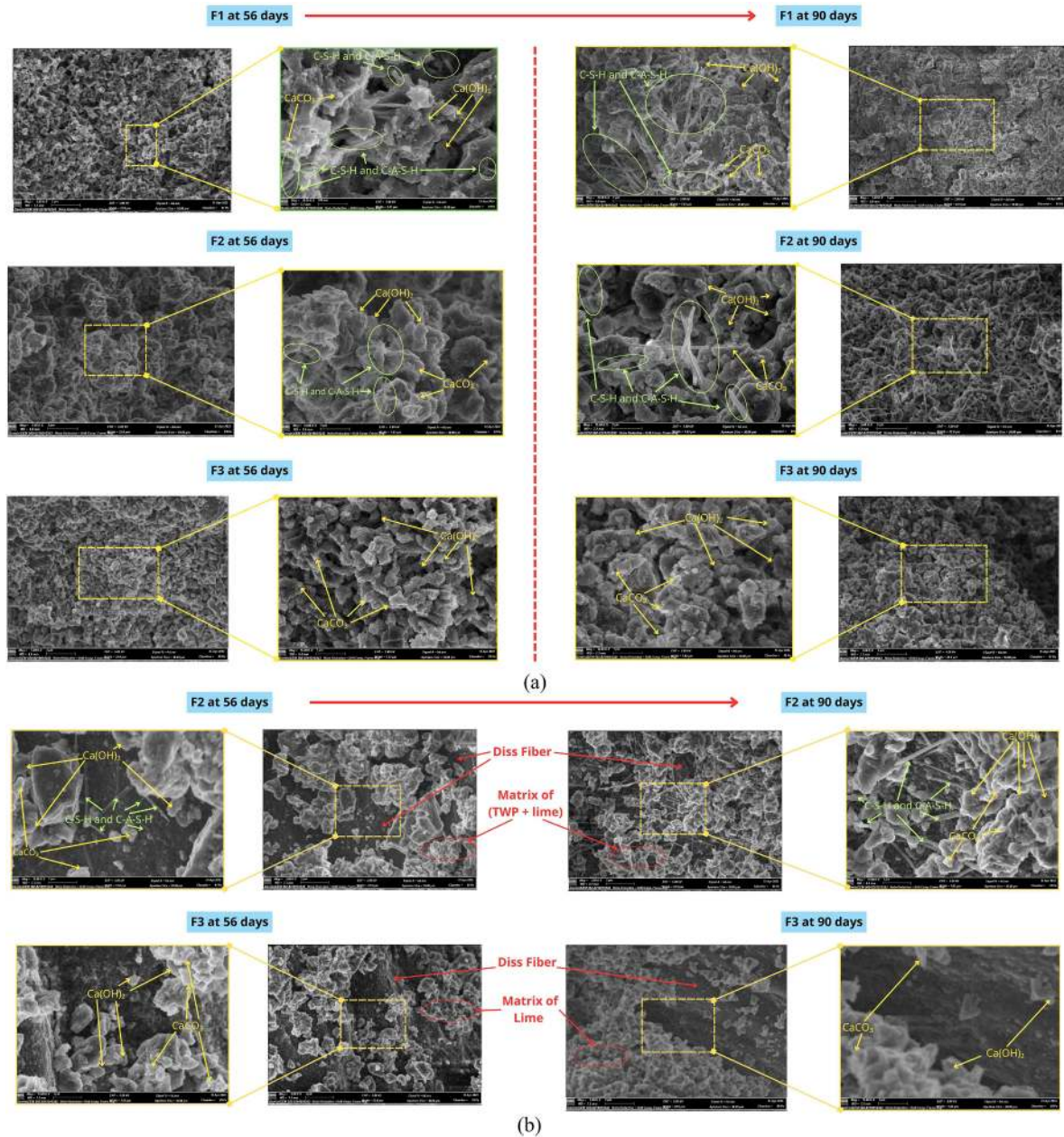


Fig. 26. SEM images at 56 and 90 days: (a) Matrices of mixes F1 and F2 composed of 85 % lime and 15 % TWP, and F3 composed of 100 % lime; (b) Regions highlighting the areas where diss fiber surfaces appear in F2 and F3 at the same ages.

slightly ($\approx +4.9\%$), consistent with a somewhat denser matrix, whereas flexural strength decreases ($\approx -7.9\%$), highlighting the anchorage role of long fibers, which are more favorable to tensile load transfer. For capillary absorption, the effect on the equilibrium absorbed mass remains marginal ($M_w \approx -0.8\%$), while the initial kinetics is more affected ($\approx +8\%$ on the absorption coefficient), suggesting increased tortuosity and reduced continuity of capillary pathways in the more compact matrix. Overall, fiber size affects the organization of the pore network (fiber orientation, pore distribution and connectivity), as well as the continuity and tortuosity of transport paths, which can modulate the hygrothermal and mechanical performances of the composite.

Effect of TWP substitution (F2 vs F3). The incorporation of 15 % TWP induces overall slight changes in the physical and mechanical properties, while providing targeted improvements at longer curing ages. Mechanical performance is generally maintained at early ages and slightly improves at later ages, with a gain of about 1 %, confirming the progressive pozzolanic reactivity of TWP and the gradual densification of the microstructure through hydrate formation, in agreement with XRD, TGA, and SEM observations. In parallel, density and open porosity exhibit only small variations (on the order of 1.6–2 %). As a result, capillary behavior remains

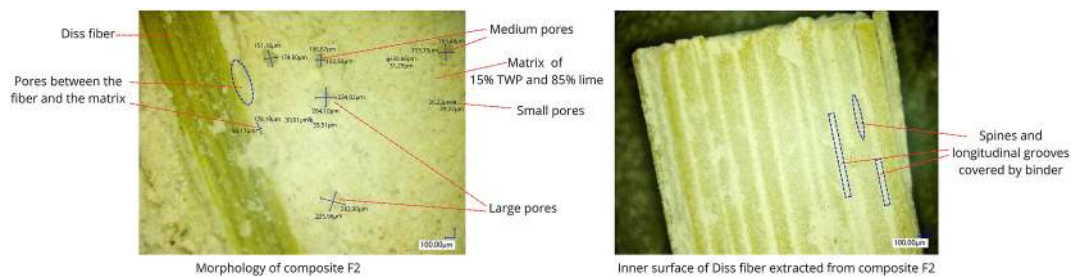


Fig. 27. Microscopic views of composite F2 morphology and of an isolated diss fiber taken from the same composite.

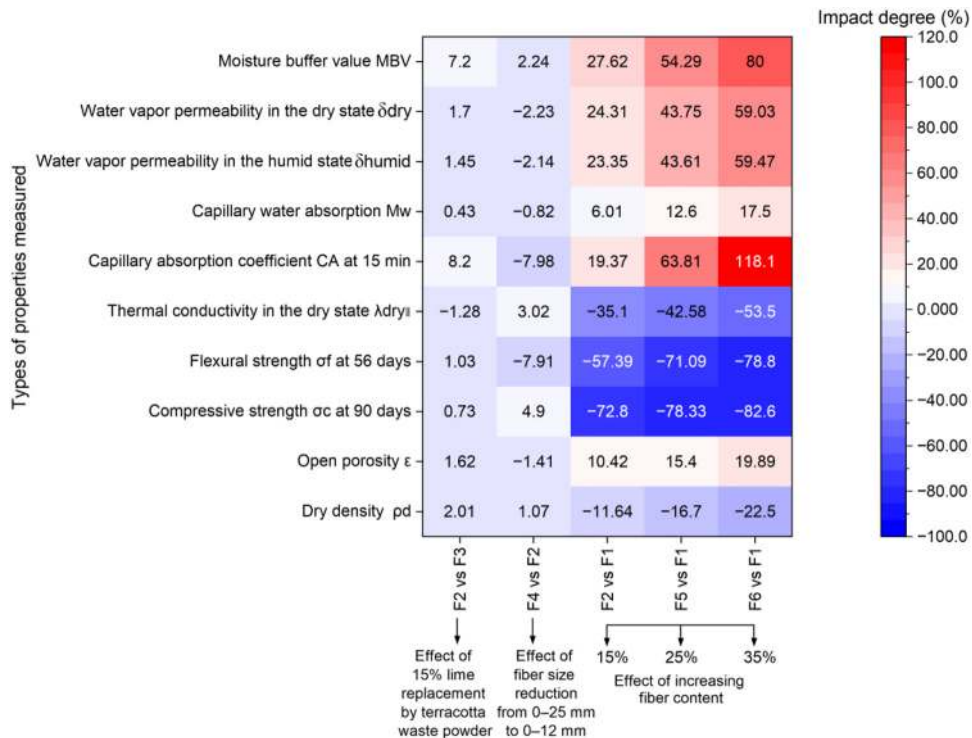


Fig. 28. Summary of the impact of diss fiber content and fiber size, as well as 15% lime substitution by terracotta waste powder, on the main properties of the developed composite as a function of mixture formulation.

almost unchanged, with only a slight increase in M_w of about 0.4 %, whereas the initial absorption kinetics is more sensitive, with an increase of approximately 8.2 % in the absorption coefficient. This trend suggests that TWP acts primarily on the pore-size distribution rather than on the overall open porosity, likely linked to a microstructure still under development at early ages. From a thermal standpoint, TWP results in a slight gain in insulation of about 1.3 %, indicating that marginal improvements in thermal performance can be achieved without compromising mechanical strength. Finally, a more pronounced enhancement of hygric performance is obtained, with an increase in MBV close to 7.2 %, reflecting the beneficial effect of TWP on the moisture buffering capacity of the material. This confirms the existence of a synergy between TWP and diss fibers, leading to improved hygric regulation.

The evolution of MBV observed under the combined effects of short fibers and TWP indicates that MBV depends not only on open porosity, whereas it relies more strongly on the hygroscopic nature of the constituents and on the architecture of the pore network, through pore size, shape, and interconnection, which govern the mechanisms of moisture storage and release.

4. Conclusion

In this study, an innovative eco-bio-based composites combining a lime-terracotta waste powder (TWP) binder and untreated diss fibers was developed as an alternative material for thermal insulation and hygrothermal control of building walls, while valorizing two local resources within a sustainable construction perspective. Its performance was assessed through detailed multiphysical characterization, analyzing the effect of three formulation parameters: substitution of 15 % of lime with TWP, diss fiber content, and fiber

size.

The characterization results indicate that increasing diss fiber content markedly enhances hygrothermal performance, primarily by increasing the moisture buffer value (MBV), while significantly reducing thermal conductivity and thus improving insulation performance. These benefits are accompanied by higher capillary absorption and increased water vapor permeability. At the same time, higher fiber contents systematically increase open porosity and reduce dry density, which lowers compressive and flexural strengths while promoting a more ductile response. On the whole, the material therefore exhibits the typical behavior of highly porous bio-composites, able to contribute simultaneously to thermal insulation and indoor hygrometric regulation. The F4 composite, containing 15 % short fibers, provides a good balance between mechanical and hygrothermal performance and falls within the category of lightweight insulating materials. The composite F6, incorporating 35 % fibers by binder mass, shows the best thermal and hygrothermal behavior. It combines low density ($\rho_s = 795 \text{ kg m}^{-3}$), very low dry thermal conductivity ($\lambda_{\text{dry}} = 0.166 \text{ W m}^{-1} \text{ K}^{-1}$), a high MBV ($1.89 \pm 0.08 \text{ g m}^{-2} \cdot \% \text{RH}^{-1}$), and increased water vapor permeability under humid conditions equal to $3.62 \times 10^{-11} \text{ kg m}^{-1} \text{ s}^{-1} \text{ Pa}^{-1}$, while with reduced compressive strength equal to 0.35 MPa at 90 days, which remains suitable for non-load-bearing vertical walls.

Long fibers generally amplify these effects and enhance flexural strength, whereas short fibers prove more effective for moisture buffering (MBV) and compressive strength. The F4 composite, containing 15 % short fibers, therefore emerges as a suitable compromise, exhibiting the highest compressive strength (0.58 MPa at 90 days), a moderate dry density (916 kg m^{-3}), a thermal conductivity of $0.239 \text{ W m}^{-1} \text{ K}^{-1}$, and an MBV of $1.37 \pm 0.07 \text{ g m}^{-2} \cdot \% \text{RH}^{-1}$, which places it among lightweight materials and within class III insulating materials.

Replacing 15 % of lime with TWP has an overall beneficial and moderate impact: it does not significantly affect density or porosity, only slightly modifies capillarity and permeability, while slightly reducing thermal conductivity, improving MBV, and maintaining or even enhancing long-term compressive strength. XRD, TGA, and SEM analyses confirm its pozzolanic reactivity, responsible for the progressive densification of the matrix through the formation of C–S–H and C–A–S–H phases. The presence of plant fibers causes only a slight initial delay in these reactions, with no significant long-term effect. Effective fiber coating, mechanical anchorage, and rough surface morphology indicate satisfactory fiber-matrix adhesion and overall composite cohesion. Correlations among open porosity, dry density, and the main hygrothermal, and mechanical properties were also identified, providing an interesting basis for developing predictive numerical models.

Overall, the developed material appears to be a promising candidate, ensuring both thermal insulation and moisture regulation while combining lightness, good hygrothermal performance, and the use of low-carbon local raw materials. However, optimization strategies remain necessary to improve the mechanical performance of the composite while preserving its thermal and hygrothermal qualities, in order to further enhance its application potential. A controlled addition of hydraulic binder or improved compaction during casting could help strengthen the matrix. The chemical interaction between fibers and binder should also be further explored to better understand its influence on mechanical behavior. Developing fiber treatments that limit early-age interactions with the matrix, while remaining environmentally compatible and without altering thermal and hygrothermal performance, appears particularly relevant.

Furthermore, controlling air and water transfer and understanding the pore architecture of the material remain essential. Additional investigations are required to quantify specific surface area, micro- and macropore distribution and connectivity, and to develop numerical models dedicated to the hygrothermal behavior of these eco-bio-based composites. Durability tests including fungal growth, long-term ageing, real-condition behavior under hygrothermal cycles, coupling with other wall materials and a life cycle assessment will help validate and better position this composite for large-scale applications in building envelopes.

CRedit authorship contribution statement

Mohammed Ksouri: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Software, Validation, Visualization, Writing – original draft, Writing – review & editing. **Kamilia Abahri:** Conceptualization, Data curation, Formal analysis, Funding acquisition, Methodology, Project administration, Resources, Software, Supervision, Validation, Visualization, Writing – original draft, Writing – review & editing. **Abdelmadjid Lasledj:** Conceptualization, Funding acquisition, Project administration, Resources, Supervision, Validation, Visualization, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

No data was used for the research described in the article.

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