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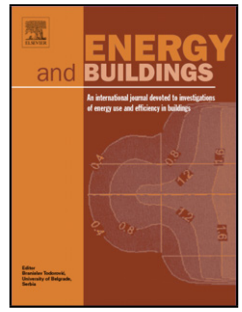

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EXPERIMENTAL STUDY ON THE PHYSICAL-MECHANICAL DURABILITY OF INNOVATIVE HEMP-BASED COMPOSITES FOR THE BUILDING INDUSTRY

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ABSTRACT

For reducing the environmental impact of the building sector, novel sustainable composites have recently been developed, by bonding hemp hurds with a new hybrid organic-inorganic binder. These composites, designed as substitutes for traditional insulating materials or as substitutes for formaldehyde-bonded wood particle boards, exhibit very promising properties. To ensure that the panel performance is maintained during the building operation phase, durability needs to be specifically evaluated as well. Therefore, in this study three composite types with low, medium and high density (LD, MD and HD, respectively) were subjected to accelerated ageing and the alterations in their physical-mechanical properties were evaluated. Composite resistance to accelerated ageing is strongly correlated with bulk density. HD composites, the only ones actually designed to be directly exposed to rainfall, exhibited almost negligible decreases in mechanical properties and hence a substantially satisfactory behavior. MD and LD composites, designed to provide thermal insulation and hence to be sheltered by HD panels, were affected to a larger extent by accelerated ageing, which however was definitely more severe than the real exposure conditions of the composites during their service life. Further studies are currently in progress to optimize the composites formulation and physical-mechanical durability.

KEYWORDS

Green materials; Natural fibres; Sustainability; Weathering; Durability; Accelerated ageing; Mechanical properties; Fibre-matrix interface; Thermal insulation; Formaldehyde-free materials

HIGHLIGHTS

- Novel hemp-based composites with promising properties have recently been proposed
- Their physical-mechanical durability after accelerated ageing was here evaluated
- Accelerated ageing consisted in cycles of immersion in water, freezing and thawing
- Composite resistance to accelerated ageing is strongly correlated with bulk density

1. INTRODUCTION

In developed countries, the energy consumption in the building sector accounts for about 20-40% of the total energy use, thus exceeding industry and transport, and it is predicted to significantly increase in the future [1]. Such an enormous energy consumption makes the development of innovative strategies for energy efficiency a priority in the building sector. Two main strategies have been followed: (i) reducing energy consumption during the building *service life*, for instance by adopting the "Zero Energy Building" approach [2]; (ii) reducing energy consumption also in the building *pre-service life*, including raw materials extraction and manufacturing [3], for instance by developing new building materials based on renewable resources (such as hemp) and/or involving less energy-consuming manufacturing processes [4-7].

Following this latter approach, novel sustainable composites for the building industry have recently been proposed by the authors [8-10]. These composites, obtained by bonding hemp hurds with a new hybrid organic-inorganic binder [10], were designed for different applications:

- low density composites (LD, 330 kg/m^3, thickness 50 mm) were developed for building thermal insulation, as substitutes for traditional materials such as glass/rock wool, extruded/expanded polystyrene or polyurethane foam;
- medium density (MD, 640 kg/m^3, thickness 30 mm) and high density composites (HD, 1210 kg/m^3, thickness 10 mm) were developed for substitution of formaldehyde-bonded wood particle boards (currently used in the building and furniture industry), for creation of exterior insulation and finishing systems and to be used as structural walls for one-storey social housing in developing Countries.

With respect to alternative traditional materials, the new composites exhibit: (i) a better environmental impact (both in the manufacturing and disposal phase) and a lower production cost, thanks to renewability of hemp hurds and the vegetable fraction of the binder; on the contrary, traditional insulating materials (such as extruded/expanded polystyrene, glass/rock wool and polyurethane foam) require petroleum-derived raw materials, high processing temperatures and/or cannot be recycled [11,12]; (ii) a better impact on human health, as they are free from any toxic organic binder (such as formaldehyde), unlike building materials such as mineral wool and wood particle boards [13,14].

The new composites also exhibit very promising thermal, physical and mechanical properties [10], comparable to those of hemp-based composites bonded with cement [15] and in some cases even superior to those of alternative natural fiber-based products available in the market [16,17]:

- LD composites have a low thermal conductivity (λ = 0.078 W/mK, in line with values reported for composites of similar density based on wood wool, wood fibers and wood chips [16]), a quite good reaction to fire (class "C - s2 - d0") and relatively good mechanical properties (flexural strength ~1 MPa). For comparison's sake, commercial cement-bonded wood wool composites with comparable bulk density (360 kg/m^3) exhibit slightly lower thermal conductivity (λ = 0.063 W/mK) but about half as much flexural strength (~0.5 MPa) [17].
- MD and HD composites, although having a higher thermal conductivity (λ = 0.138 W/mK for MD), are characterized by very good reaction to fire (class "B - s2 - d0" for MD, at least the same class for HD) and mechanical properties (flexural strength ~7 MPa for MD and ~16 MPa for HD) [10]. For comparison's sake, commercial cement-bonded wood chipboards with 1000 kg/m^3 bulk density exhibit flexural strength ~9 MPa [17].

The above-reported characteristics make the novel composites highly promising for substituting commonly used materials with higher environmental impact. In particular, among other applications, the possible use of the new composites for creating a multi-layer wall plug for concrete, steel or wood structures (where the inner LD layers provide thermal insulation and the external MD/HD layers provide resistance against physical-mechanical actions and protection against fire) has been proposed [10].

In any case, before the composites can be introduced into the market and actually employed in buildings, their durability needs to be specifically evaluated, to ensure that the panel performance is maintained during the building service life. Durability actually involves resistance to a multitude of weathering factors that may threaten the panel performance, e.g. moisture, freeze-thaw or bio-organisms. Which weathering factors may be active in a specific situation actually depends on the panel operation conditions (indoor/outdoor, possible exposure to direct rainfall, possible contact with the ground, possible presence of rendering layers, etc.), which, in turn, depend on the panel function.

As a first step towards a 360-degree assessment of the new composites durability during their service life, in this paper the resistance against water-related deterioration was experimentally evaluated, by determining the alterations in physical-mechanical properties after accelerated ageing. In the lack of an international standard describing a specific accelerated weathering method for hemp-based panels, the European Standard for wood-based panels EN 321 [18] was followed. The weathering conditions involved by this standard (i.e. cycles of immersion in water, freezing and thawing) can be considered as definitely severe, because it is extremely unlikely that, in their service life, the new composites will be subject to complete saturation with water. Indeed, the multi-layer wall plug made of hemp-based panels (similarly to wall plugs made of wood-based panels, currently available in the market) is designed to be protected from rising damp from the ground (by using an impermeable basement element), so that the only direct source of liquid water is expected to be rainfall. However, among the different composite types, the only panels that are expected to be subjected to direct contact with rainfall are HD panels, designed as the external layer of the multi-layer wall plug, over which also a traditional rendering layer may be present.

Consequently, the weathering conditions adopted in this study are actually relevant *only* for HD composites. On the contrary, in the case of MD and LD composites, the investigated weathering conditions are *not* representative of the real conditions that the composites will have to face in their service life. Different deterioration processes (such as fungi growth, possibly favored by interstitial humidity condensation) are expected to be more relevant for these kinds of composites. However, investigating how does mechanical resistance to accelerated ageing change as a function of the composite physical-microstructural properties is very interesting from a materials science point of view, hence also MD and LD composites were subjected to the same weathering tests, although not representative of the composites exposure conditions during their service life. Additional tests to evaluate the composite resistance against bio-deterioration (fungi, termites, etc.) will be addressed in future steps of research. Anyway, considering the basic environment of the composites (as detailed later in the paper), a good durability to bio-deterioration is expected.

2. MATERIALS AND METHODS

2.1 Hemp-based composites

Three composite types with low, medium and high bulk density (LD, MD and HD, respectively) were produced according to the procedure described in detail in [10]. In brief, the composites were obtained by bonding hemp hurds with a novel hybrid organic-inorganic binder, comprising a vegetable fraction and a magnesium-based mineral fraction [10]. For each composite, the hemp hurds size, the binder/hemp hurds ratio and the press compression ratio are reported in Table 1. Notably, with respect to MD and LD composites, HD panels were obtained by using hemp hurds with smaller size (length 2-5 mm instead of 10-30 mm, diameter 1-2 mm instead of 2-6 mm) and by adopting a lower binder/hemp hurds ratio (1:1.25

instead of 1:1). At the end of the manufacturing process, involving hot pressing at 80 °C for 3 minutes, panels with 150 × 50 cm^2 dimensions and thickness (t) ranging from 10 to 50 mm (depending on composite type, Table 1) were obtained. These panels were then sawn to obtain specimens for the different physical-mechanical tests performed to assess the composite durability. In particular, composite characteristics were determined before and after exposure to accelerated ageing, carried out as described in the following paragraph.

2.2 Accelerated ageing procedure for durability evaluation

The accelerated ageing procedure described in the European Standard EN 321 for wood-based boards [18] was adopted, in the lack of a specific international standard for hemp-based panels. This procedure involves repeated cycles of: (1) immersion in water at 20±1 °C for 70 h; (2) freezing at -20±5 °C for 24 h; (3) drying at 70±2 °C for 70 h; (4) conditioning in laboratory (T=23±2 °C and RH = 50±5%) for 4 h. In total, three cycles were carried out. At the end of the tests, the alterations in physical-mechanical properties were evaluated. In particular, unweathered and artificially weathered samples were tested after at least 28 days since the end of the ageing procedure, to allow weathered samples to equilibrate with the environment. For each physical-mechanical property, all the samples (unweathered and weathered) were tested on the same day, for ensuring comparability.

2.3 Physical-mechanical characterization

For each composite type, the physical-mechanical properties described in the following were determined on 5 replicate samples. It is noteworthy that, for composites having different density and hence different function, different physical-mechanical properties are particularly important. For instance, the resistance to axial withdrawal of screws is a fundamental property for MD and HD panels (forming the external part of the multi-layer wall plug), but the same property is secondary for LD panels (designed as the insulating internal layer of the wall). Nonetheless, the physical-mechanical properties described in the following were determined for all the panel types (whenever possible) to gain information on the dependence of composite properties on their bulk density.

2.3.1 Compressive strength

Compressive strength corresponding to a strain ε = 10% (σ_c) was determined for LD and MD composites, while for HD composite the limited thickness (t = 10 mm) makes σ_c determination scarcely reliable [10]. According to European standard EN 826 [19], 5 × 5 × t

cm^3 specimens were subjected to axial compressive test using an Amsler-Wolpert loading machine (maximum load 10 kN) at a constant displacement rate of 5 and 3 mm/min for LD and MD panels, respectively (as a function of sample thickness).

2.3.2 Flexural strength

Flexural strength (σ_f) was measured by three points bending test according to European standard EN 12089 [20] on 26 × 12 × *t* cm^3 specimens, using an Amsler-Wolpert loading machine (maximum load 5 kN) at a constant displacement rate of 10 mm/min.

2.3.3 Tensile strength perpendicular to the plane

Tensile strength perpendicular to the plane of the panel (σ_t) was determined according to European standard EN 1607 [21] on 5 × 5 × *t* cm^3 specimens, making use of the pulling apparatus illustrated in Figure 1*a*. In brief, samples were glued to 2 steel plates (20 mm thickness) using an epoxy bi-component resin and, after curing for 24 hours, the so-prepared specimens were inserted into the pulling apparatus, which has a self-aligning attachment to the loading machine for ensuring even load distribution in the sample [10]. The tensile load was then applied using an Amsler-Wolpert loading machine (maximum load 10 kN) at a constant displacement rate of 10 mm/min.

2.3.4 Resistance to axial withdrawal of screws

For LD and MD composites, the panel resistance to axial withdrawal of screws was determined both in the direction perpendicular ($R^{\perp}$) and parallel ($R^{//}$) to the panel plane, while for HD composite only $R^{\perp}$ was measured (because of sample thickness). According to the European standard EN 320 [22], describing the test method for particleboards and fiberboards and here adopted in the lack of a specific standard for hemp-based panels, 7.5 × 7.5 × *t* cm^3 specimens were used. The test was performed making use of the pulling apparatus illustrated in Figure 1*b* (for $R^{\perp}$) and Figure 1*c* (for $R^{//}$). As required by EN 320 [22], the screwing length (*s*) was different for different panels, as a function of panel thickness: $s = 15$ mm for LD and MD (having thickness $t > 15$ mm); $s = t$ for HD (having thickness $t = 10$ mm). For comparing $R^{\perp}$ values among different panels, the specific resistance to axial withdrawal of screws ($R_s^{\perp}$) was calculated as $R_s^{\perp} = R^{\perp}/s$. Once screws had been inserted into the samples, specimens were inserted in the pulling apparatus (Figure 1, *b* and *c*), made of a steel plate with a central boring (for restraining the screw) and an underlying metal jig (for restraining the panel). By making use of the same self-aligning pulling apparatus used for determining tensile strength perpendicular to the plane, the screw and the panel were then pulled apart by using an Amsler-Wolpert loading machine (maximum load 5 kN) at a constant displacement rate of 10 mm/min, until screw withdrawal at maximum tensile load.

2.3.5 Weight loss

Weight loss after accelerated ageing (Δw) was determined on $5 \times 5 \times t$ cm^3 samples according to the formula:

$$\Delta w = (w_i - w_f)/w_i \cdot 100,$$

where w_i and w_f are the initial and final weight, respectively, after equilibration in laboratory conditions.

2.3.6 Swelling in thickness

Swelling in thickness after accelerated ageing (G) was measured on $5 \times 5 \times t$ cm^3 specimens according to the European standard EN 317 [23], describing the test method for particleboards/fiberboards and here adopted in the lack of a specific standard for hemp-based panels.

2.3.7 Composite microstructure

The alterations in composite microstructure induced by accelerated ageing were evaluated by observing samples fracture surfaces, with a stereo-optical microscope (SOM, Olympus SZX10) and a scanning electron microscope (SEM, Philips XL20).

2.3.8 Analysis of water after panel immersion during accelerated ageing

For evaluating the possible causes of alterations in composite properties, the water in which the panels were immersed during accelerated ageing was analyzed. For each composite type, one specimen (dimensions $26 \times 12 \times t$ cm^3) was placed in a plastic container and immersed in 2.5 l de-ionized water for 70 h (corresponding to the duration of immersion in water for each cycle). The panels were then extracted and the pH of the water remaining in the plastic containers was determined by a pH-meter Cyberscan pH310. The same instrument was also used for measuring the pH of the fresh binder, right at the end of its preparation and before hemp hurds addition (for the detailed description of composite preparation see reference [10]). The same water was then further analyzed to determine the chemical composition of the fraction that had been dissolved in this water (causing a color change from transparent to light yellow). After filtering the water through a paper filter, the filter and the retained deposit were let dry in laboratory conditions. The dry deposit was then made conductive by using gold and finally analyzed by dispersive X-ray spectroscopy (EDS), using a EDX 9800 microanalysis device mounted on the Philips XL20 SEM.

3. RESULTS AND DISCUSSION

After accelerated ageing, the three composite types exhibited significantly different alterations in mechanical properties, as illustrated in Figure 2.

HD composites, the only ones that will actually be directly exposed to rainfall, underwent minor alterations in flexural strength ($\Delta\sigma_f$ = -6%, Figure 2*b*). This behavior is in line with (and in some cases better than) the behavior reported in the literature for hemp-based composites and, in general, natural fiber-based composites subjected to weathering tests [24-26]. For instance, in the case of hemp/unsaturated polyester [24] and kenaf/unsaturated polyester composites [26] significant decreases in flexural strength and tensile strength (reaching -40%) have been found after immersion in water, as a consequence of fiber swelling, responsible for fiber-matrix debonding and consequent mechanical properties decrease. Fiber swelling after immersion in water and consequent degradation of the matrix/fiber interface are usually identified as the main cause of natural fibers-based composites deterioration when exposed to water [24,26-28]. A significant decrease in flexural strength, owing to fiber-matrix interface damage induced by fiber swelling, has been reported also for composites based on vegetable fibers and blast furnace slag, which had been exposed for 16 months to natural weathering (in this case, however, other weathering factors such as matrix carbonation and fiber mineralization contributed to the panel mechanical deterioration) [25]. HD composites investigated in this study also underwent negligible decreases in resistance to axial withdrawal of screws ($\Delta R_s^{\perp}$ = -2%, Figure 2*d*). Differently, significant decreases were registered in tensile strength ($\Delta\sigma_t$ = -38%, Figure 2*c*), which is thought to derive from the fact that this property is strongly dependent on the composite internal cohesion, that was diminished by some deterioration of the fiber/matrix interface and some binder dissolution (as described in the following where SEM results are discussed).

Although not designed to be directly exposed to rainfall, also MD and LD composites were subjected to accelerated ageing by immersion in water, freezing and thawing. As expected, MD composites and, to a higher extent, LD composites underwent more pronounced alterations in mechanical properties compared to HD composites, as can be seen in Figure 2. MD panels exhibited significant decreases in σ_c, $R_s^{\perp}$ and $R^{//}$ (-31%, -20% and -

41%, Figures 2*a*, 2*d* and 2*e*, respectively) and higher decreases in σ_f and σ_t (-49% and -61%, Figures 2*b* and 2*c*, respectively). LD panels exhibited high decreases in basically all mechanical properties, ranging from -51% in $R_s^{\perp}$ (Figure 2*d*) to -89% in σ_t (Figure 2*c*). However, especially in the case of LD composites, the panel mechanical properties were rather limited even *before* accelerated ageing, essentially because these panels are not designed to provide a very high mechanical performance, but rather to have good insulating properties. The different resistance to weathering of HD composites, on the one hand, and MD and LD composites, on the other hand, is a consequence of the different water absorption (21.2, 39.3 and 118.4 wt%, respectively [10]) and mechanical properties (especially tensile strength, amounting to 1.79, 0.49 and 0.18 MPa, respectively [10]) of the composites. These features, in turn, are a consequence of the different characteristics of hemp hurds and binder/hemp hurds ratio, as well as of the compression ratio (Table 1).

After weathering cycles, all the composites exhibited some swelling in thickness (Table 2), similar for the three composite types and substantially in line with values found in a previous study after immersion in water for 24 hours [10]. The measured values are also in line with values reported in the literature for other natural fiber-based composites, e.g. ~3.5% swelling for kenaf/unsaturated polyester composites after immersion in water for 200 hours (in the present study, panels had been immersed for a total of 210 hours) [26]. With respect to swelling in thickness reported in technical data sheets of commercial cement-based woodchip boards [17], values measured for the hemp composites result higher (~3.5% instead of ~1.5%), but the immersion in water lasted much longer for the hemp composites than commercial woodchip boards (210 hours instead of 24 hours). Swelling measured in the present study can be ascribed to swelling of hemp hurds when water penetrates into them, which causes partial hemp hurds/matrix debonding and, consequently, mechanical properties deterioration, according to what has been reported in the literature for other hemp-based composites [24].

After weathering cycles, the composites also exhibited some weight loss (Table 2). Similarly to mechanical properties, also in terms of weight loss HD composites resisted weathering better than MD and LD composites, as expected. Especially in the case of these latter composites, water color shifted from transparent to light yellow and small pieces of hemp hurds detached from the composites during immersion in water, which induces to attribute the measured weight losses (as well as the registered decrease in mechanical properties) to partial dissolution of the binding fraction.

According to SOM and SEM results, in the case of the unweathered composites (Figures 3 and 4), a good adhesion between the binder and the hemp hurds can be observed. This is particularly evident in the SEM micrographs of the HD samples (where a good continuity between hemp hurds and the binder is present) and of the MD samples (where a good adhesion is present as well, even if the binding matrix is more porous). After accelerated ageing (Figures 3 and 4), some differences in panel microstructure can be noticed. More cracks and voids can be observed at the binder-hurds interface, where however the adhesion seems to be still rather good. In addition to some deterioration of the matrix-hemp hurds interface, an increase in the matrix porosity can be observed, presumably due to some dissolution of the binding phase.

Partial dissolution of the binding matrix is also suggested by results of pH measurements carried out on water in which the panels had been immersed for 70 h. For all the panel types, water resulted having a pH = 9.9-10.0, hence significantly higher than the original pH of de-ionized water used for the test (pH = 5.6). This increase in pH is very likely to be ascribed to binder partial dissolution in water. Indeed, the fresh binder has pH = 9.9, hence the dissolution of some binder component is thought to be responsible for increasing the pH of water where the panels had been immersed.

To have a first indication of which component of the binder is responsible for its partial solubility in water, EDS was performed on the deposit obtained by filtering and drying the water where the panels had been immersed during the ageing cycles. EDS detected only C and O (contained in the vegetable fraction) and not Mg (contained in the mineral fraction). Therefore, the partial solubility of the binding matrix seems to be ascribable to the vegetable fraction, rather than to the magnesium-based compounds.

Further studies are currently in progress to optimize the binder formulation, so as to make the binding matrix less soluble in water and hence to enhance the physical-mechanical durability of the composite panels.

4. CONCLUSIONS

The results of this study, aimed at evaluating the physical-mechanical durability of innovative hemp-based composites for the building sector, allow to derive the following conclusions:

- For the HD composites, designed to be the external layers of a multi-layer wall plug and hence the only composites actually designed to be directly exposed to rainfall, almost negligible decreases in mechanical properties, fully in line with values reported in the literature for alternative hemp-based composites, were found after accelerated ageing in severe conditions. This allows to regard the physical-mechanical durability of these composites as substantially satisfactory.

- For the MD and LD composites, designed as internal thermal insulating layers of the multi-layer wall plug, higher decreases in mechanical properties and weight loss were registered after accelerated ageing. However, since the tested weathering conditions are far from being representative of the real exposure conditions of these types of composites, the suitability of LD and MD composites as internal layers of the wall plug seems to be not altered.
- Results on how physical-mechanical durability of the composites changes depending on the composites physical-microstructural properties (which in turn depend on the process conditions) and results on the partial dissolution of the binding matrix will be used to further optimize the binder formulation, so as to lower its solubility in water and thus enhance the panel physical-mechanical durability. Such work is currently in progress

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FIGURE CAPTIONS

Figure 1. Purpose-built apparatus for measuring the tensile strength perpendicular to the plane of the panel (a) and the resistance to axial withdrawal of screws in the direction perpendicular (b) and parallel (c) to the plane of the panel.

Figure 2. Durability evaluation of the investigated hemp-based composites: mechanical properties. (a) σ_c = compressive strength; (b) σ_f = flexural strength; (c) σ_t = tensile strength perpendicular to the plane of the panel; (d) $R_s^{\perp}$ =specific resistance to axial withdrawal of screws in the direction perpendicular to the plane of the panel; (e) $R^{//}$ = resistance to axial withdrawal of screws in the direction parallel to the plane of the panel. Results are averages for 5 values, bars indicate standard deviation.

Figure 3. Durability evaluation of the investigated hemp-based composites: microstructure of fracture surfaces (SOM).

Figure 4. Durability evaluation of the investigated hemp-based composites: microstructure of fracture surfaces (SEM).

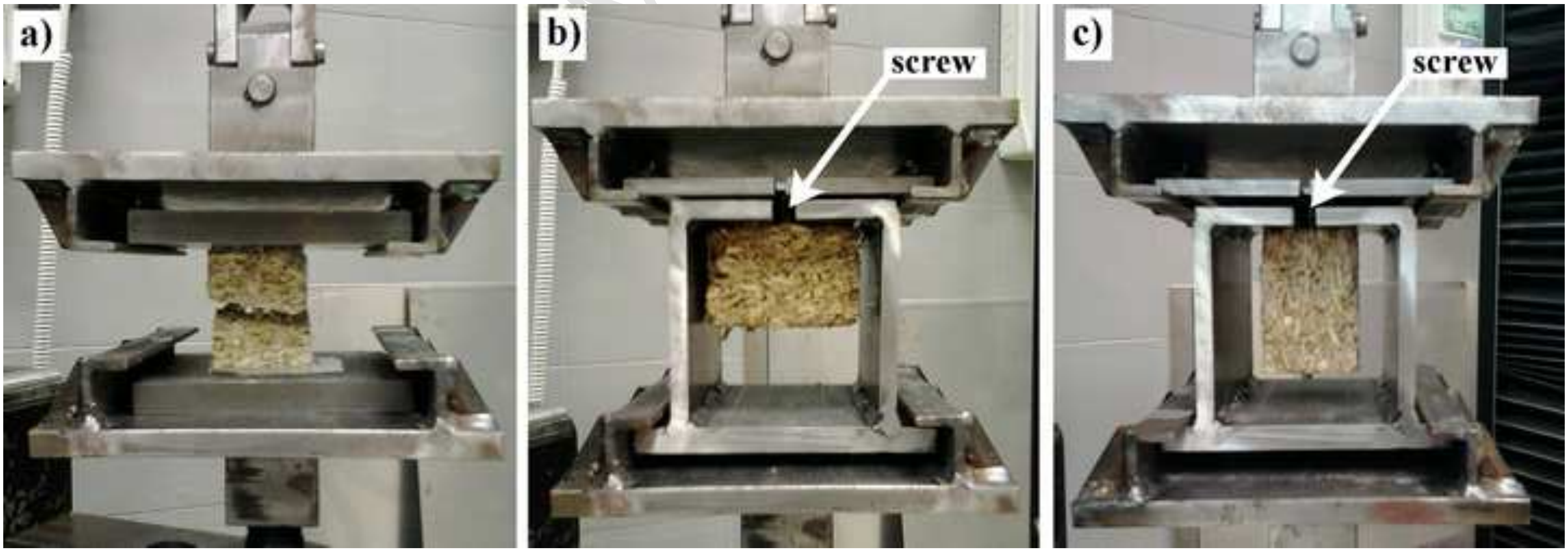
Table 1. Characteristics and manufacturing parameters of the investigated hemp-based composites.

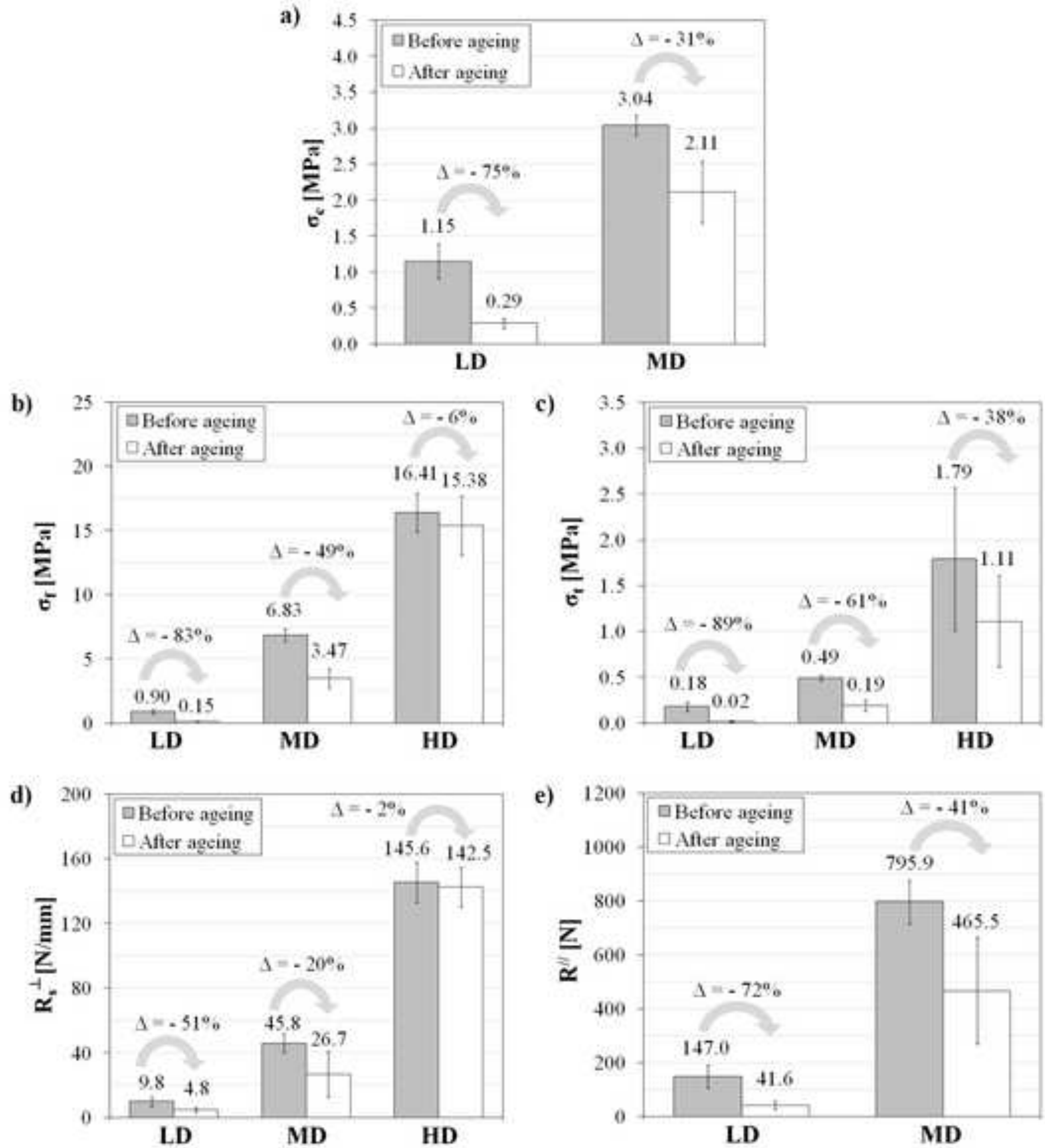
	Low density (LD)	Medium density (MD)	High density (HD)
Density [kg/m^3]	330	640	1210
Thickness (t) [mm]	50	30	10
Hemp hurds length [mm]	10-30	10-30	2-5
Hemp hurds diameter [mm]	2-6	2-6	1-2
Binder/hemp hurds ratio [wt./wt.]	1:1	1:1	1:1.25
Compression ratio	1:1.7	1:3.3	1:4

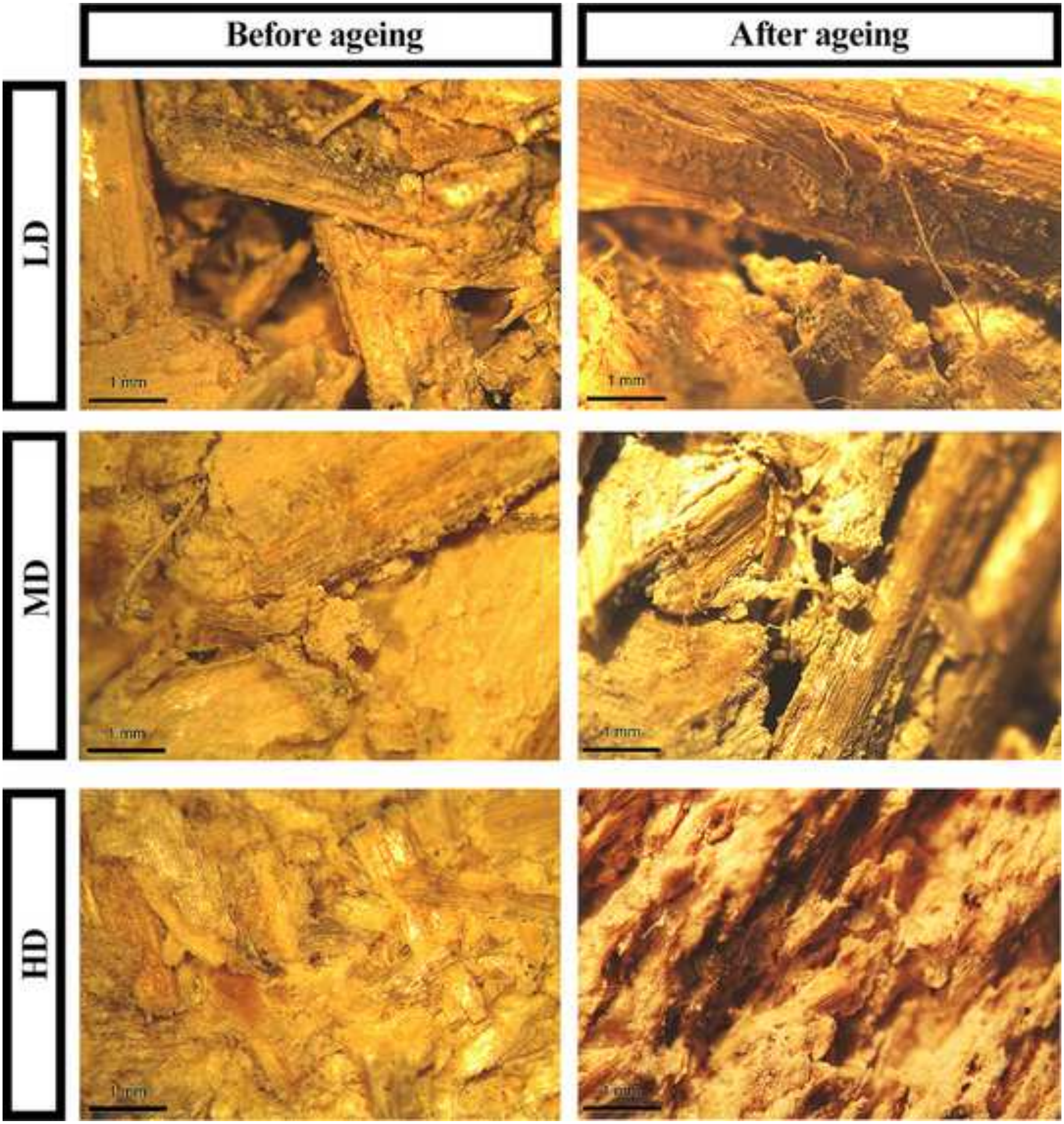
Table 2. Durability evaluation of the investigated hemp-based composites: physical properties (weight loss (Δw) and swelling in thickness (G) after accelerated ageing). Results are averages for 5 values, standard deviations in brackets.

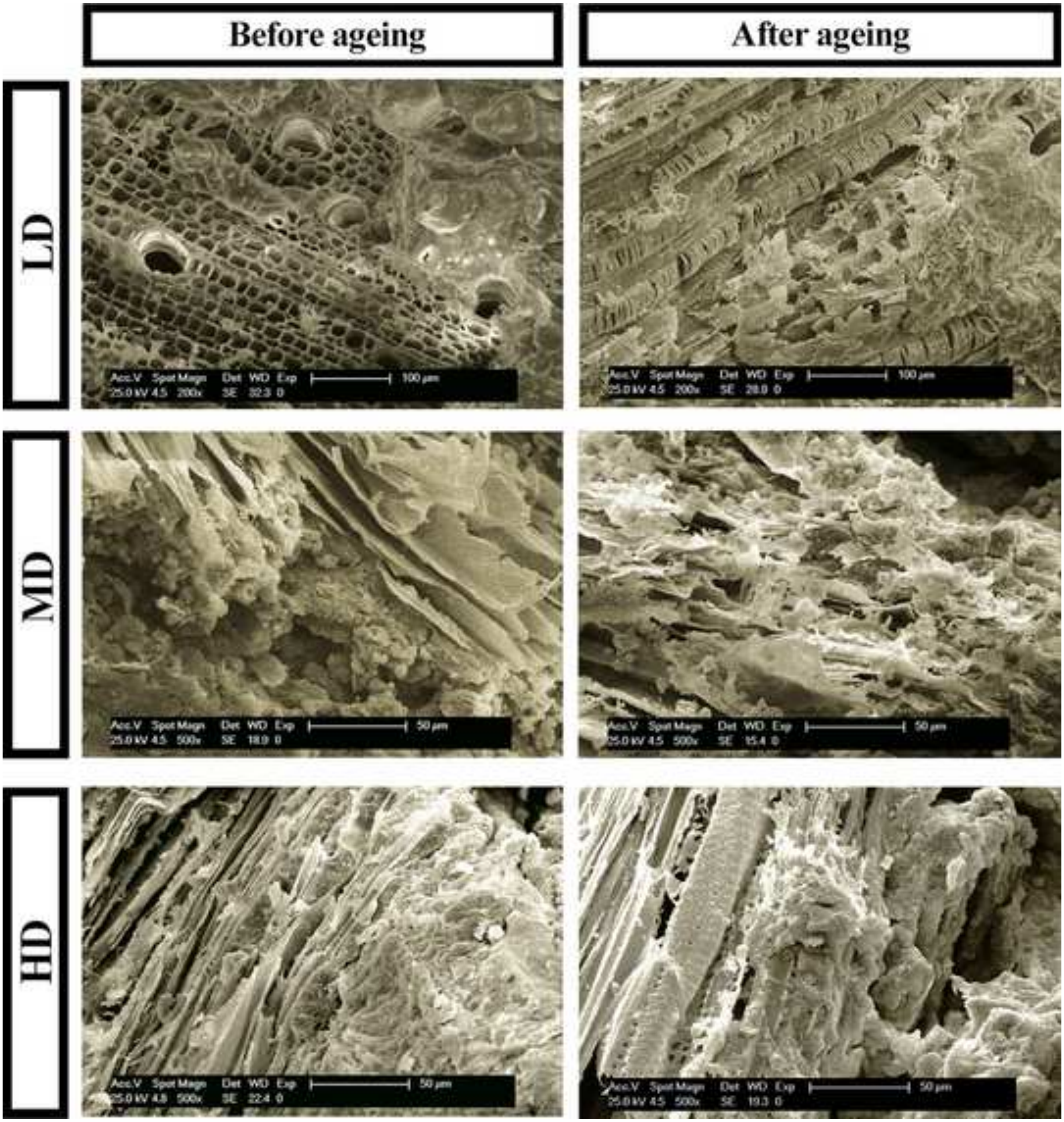
	LD	MD	HD
Δw [wt.%]	14.6 (±0.3)	11.3 (±1.0)	8.3 (±0.4)
G [%]	3.5 (±0.9)	3.2 (±1.0)	3.9 (±1.2)

Figure 1




a)
σc [MPa]
Before ageing
After ageing
Δ = - 75%
Δ = - 31%
1.15
0.29
3.04
2.11
LD
MD
b)
σf [MPa]
Before ageing
After ageing
Δ = - 83%
Δ = - 49%
Δ = - 6%
0.90
0.15
6.83
3.47
16.41
15.38
LD
MD
HD
c)
σt [MPa]
Before ageing
After ageing
Δ = - 89%
Δ = - 61%
Δ = - 38%
0.18
0.02
0.49
0.19
1.79
1.11
LD
MD
HD
d)
Rs⊥ [N/mm]
Before ageing
After ageing
Δ = - 51%
Δ = - 20%
Δ = - 2%
9.8
4.8
45.8
26.7
145.6
142.5
LD
MD
HD
e)
R// [N]
Before ageing
After ageing
Δ = - 72%
Δ = - 41%
147.0
41.6
795.9
465.5
LD
MD


Before ageing
After ageing
LD
MD
HD
1 mm
1 mm
1 mm
1 mm
1 mm
1 mm


Before ageing
After ageing
LD
MD
HD
100 µm
50 µm