

## Article

# Gypsum Mortars with *Acacia dealbata* Biomass Waste Additions: Effect of Different Fractions and Contents

Alessandra Ranesi <sup>1,2,\*</sup> , Paulina Faria <sup>1</sup> , Ricardo Correia <sup>3</sup> , Maria Teresa Freire <sup>4</sup>, Rosário Veiga <sup>2</sup>   
and Margarida Gonçalves <sup>3,5</sup> 

- <sup>1</sup> CERIS—Civil Engineering Research and Innovation for Sustainability, NOVA School of Science and Technology, NOVA University of Lisbon, Quinta da Torre, 2829-516 Caparica, Portugal; paulina.faria@fct.unl.pt  
<sup>2</sup> National Laboratory for Civil Engineering, Avenida do Brasil 101, 1700-066 Lisbon, Portugal; rveiga@lnec.pt  
<sup>3</sup> METRICs, Department of Science and Technology of the Biomass, NOVA University of Lisbon, Quinta da Torre, 2829-516 Caparica, Portugal; rjc07189@campus.fct.unl.pt (R.C.); mmpg@fct.unl.pt (M.G.)  
<sup>4</sup> CERIS—Civil Engineering Research and Innovation for Sustainability, SIVAL—Gessos Especiais Lda, Rua Emídio Oliveira Faria, 2425-879 Souto da Carpalhosa, Portugal; inov-ge@sival.pt  
<sup>5</sup> VALORIZA, Polytechnic Institute of Portalegre, Campus Politécnico, 7300-555 Portalegre, Portugal  
\* Correspondence: a.ranesi@campus.fct.unl.pt; Tel.: +39-389-9975619

**Abstract:** In recent decades, interest in the eco-efficiency of building materials has led to numerous research projects focused on the replacement of raw materials with mineral and biomass wastes, and on the production of mortars with low-energy-consuming binders, such as gypsum. In this context, five different fractions (bark, wood, branchlets, leaves, and flowers) of *Acacia dealbata*—an invasive species—were evaluated as fillers for premixed gypsum mortars, at 5% and 10% (vol.) addition levels and fixed water content. Although these biomass fractions had different bulk densities (>50% of variation), all the mortars were workable, although presenting different consistencies. As expected, dry density decreased with biomass addition, but, while mortars with addition at 5% presented a slight shrinkage, a slight expansion occurred with those with 10% addition. Generally, the mechanical properties decreased with the biomass additions even if this was not always proportional to the added content. The wood fraction showed the most positive mechanical results but flexural and compressive strengths of all the tested mortars were found to be higher than the lower standard limit, justifying further studies.

**Keywords:** bio-based mortars; invasive species; biomass additions; bio-composites; by-products; agro-industrial wastes; density; dimensional variation; mechanical properties; pore structure



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## 1. Introduction

In recent years many findings have been made on the effects of indoor relative humidity and temperature on human health [1–3]. It is common knowledge that an intermediate range of relative humidity (RH) can prevent airway and ocular irritations in various diseases [4,5] and is often related to thermal comfort in free-running buildings [6] when adaptive models are considered [7].

Plastering mortars usually cover large indoor surfaces and, thus, can contribute to passively equilibrating indoor relative humidity improving occupants' comfort and, in some cases, health. To provide that contribution, they have to be highly hygroscopic, adsorbing and desorbing moisture from and to the indoor air.

Gypsum plasters are broadly used to coat (plaster) indoor walls and ceilings as they appear to be an appropriate option not only in new construction but also in many restoration interventions [8]. Moreover, hemihydrate gypsum binder is produced at around 120–180 °C, having a much lower firing temperature and milling energy for production than other binders, i.e., cement (around 1500 °C) or air lime (around 900 °C). Thus, the associated low embodied energy makes gypsum plasters a sustainable solution. However,

among common plastering mortars, gypsum-based mortars present very low hygroscopicity [9–11]. Although not studied in the present work, it is probable that the moisture buffer capacity of gypsum plasters may be improved with the addition of hygroscopic materials.

Nonetheless, the eco-efficiency of building products can be increased by addition of wastes and the replacement of raw materials [12,13]. Among these wastes, agro-industrial wastes can be used in the production of eco-products for construction [14] with the purpose of enhancing relevant physical and chemical properties, such as bulk density, thermal conductivity, and the hygric and hygroscopic behavior of those products, while creating useful applications for various biomass wastes [15–19]. Actually, as biomass is usually hygroscopic, it is expected that they may improve gypsum plasters hygroscopicity. However, in comparison to cement-based mortars [20–22], studies on the effects of the incorporation of agro-industrial wastes in gypsum-based mortars are rare.

Acacia species mostly originated in Australia but have spread all over the world and have become invasive due to their high capacity for growth, seed production, and seed germination, which can be active for several years. Their selective removal is not economically viable if added-value applications are not found for the collected biomass. The use of biomass collected in forest environments contributes to reducing the danger of forest fires [23,24] and, in the case of invasive species, may constitute a method of propagation control since it disrupts the reproductive cycle, namely by preventing seed formation [25].

Use of Acacia wastes in plaster formulations was not found in literature although applications of Acacia biomass in composite materials have been described by some authors [26,27]. Also, some fractions of the plant have already been used in other sectors, namely bark as a source of tannins for the leather industry [28] or flowers used to produce absolute oils for the perfume industry [29]. Other fractions of Acacia biomass have traditionally been used as a source of bioactive components for folk medicines [30]. After recovery of these functional extractives, the biomass still retains most of its lignocellulosic components, keeping its potential to be used in energy production or material applications.

The aim of the present study was to assess if the addition of different fractions and contents of *Acacia dealbata* biomass to gypsum plastering mortars jeopardizes the common fresh-state properties of the mortars or their mechanical properties in order to discard formulations which do not meet the requirements for further studies related to hygroscopicity. Hence, five different fractions of the same plant (*Acacia dealbata*) were selected and added to a gypsum premixed mortar (5% and 10% vol.) after the recovery of extractives for other applications. Although the premixed product is based on a low embodied energy binder, such as hemihydrate gypsum, the addition of biomass reduces the consumption of raw materials needed to produce it, reducing the environmental impact of the plasters. Nevertheless, it is important to confirm if the addition require a higher consumption of water to present adequate workability and comply with the mechanical requirements for gypsum-based plastering mortars.

## 2. Materials and Methods

### 2.1. Materials

A premixed industrial powder product (GP), Sival Reabilita, produced by the company Sival, in Portugal was selected for the study. This product based on gypsum, mineral fillers, and admixtures and is ready to mix with water for manual application in interior walls and ceilings. It complies with EN 13279-1 [31], type B1/20/2 and can be used to plaster old indoor old walls as well as new ones.

Five different *Acacia dealbata* fractions were selected to be incorporated in the mortar formulations: flowers (Fl), leaves (Le), branchlets (Br), wood (Wo), and bark (Ba). The biomasses were collected in the regions of Alcobaça and Caparica (Central Portugal), from at least ten different trees at each location. The fractions were milled, sieved to 2 mm, and macerated in acetone, at 25 °C, to recover extractives that could be valorized in various applications, such as nutraceutical products. The extracted biomasses were air-dried at

25 °C for 48 h, and characterized for loose bulk density and color coordinates according to the CIELab color system before use in the mortar preparations. Instrumental color was determined using a colorimeter (CHROMA METER CR-410, Tokyo, Japan), calibrated using a standard white reflector plate. The visual observation of the *A. dealbata* fractions at the time of the mortar production is presented in Figure 1, and Table 1 presents their loose bulk density and color parameters.



**Figure 1.** *Acacia dealbata* fractions: (a) wood, (b) branchlets, (c) leaves, (d) flowers, and (e) bark.

**Table 1.** Loose bulk density and color coordinates of *Acacia dealbata* fractions.

<i>A. dealbata</i> Fraction	Loose Bulk Density (g/cm <sup>3</sup> )	Color (CIELB Coordinates)		
		L*	a*	b*
Wood	0.211	65.58	0.68	18.94
Branchlets	0.378	59.11	1.31	17.51
Leaves	0.415	34.97	0.59	11.04
Flowers	0.217	35.33	8.50	11.39
Bark	0.435	43.19	7.58	12.85

A decrease in the luminosity parameter L\* observed for leaves and flowers indicates darker colors. Although fresh *Acacia* flowers had a light yellow color, the process of solvent extraction and drying, promotes oxidation reactions that result in a brownish darker color. Low values of a\* indicate an increase of the green component, an effect that was more evident for leaves and wood. The yellow component was higher for the biomass fraction with higher b\* values, namely wood and branchlets. These different colors of the biomasses can influence the color of the mortars and are important if the plasters have no finishing layer. The loose bulk density of the factions varied up to 51% from the denser bark fraction to the lighter wood one.

## 2.2. Mix Design

The selected fractions of *A. dealbata* were added to the GP, at 0% (reference mortar), 5%, and 10% by volume, and manually homogenized. Additions of constant volumes of all the different fractions were made so that the physical changes were comparable. The mass for exact incorporation of each fraction (Table 2) was calculated according to the loose bulk density of each biomass (Table 1).

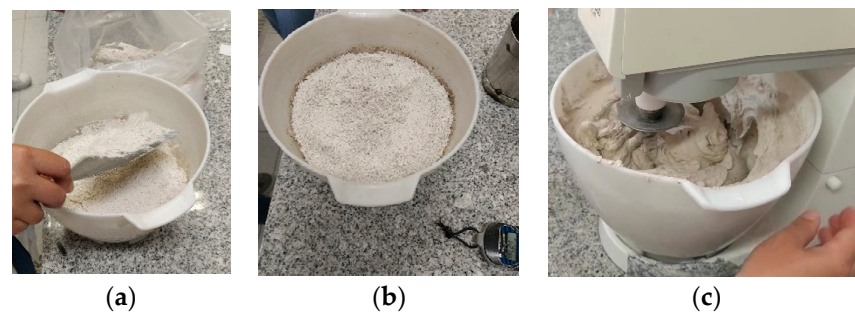
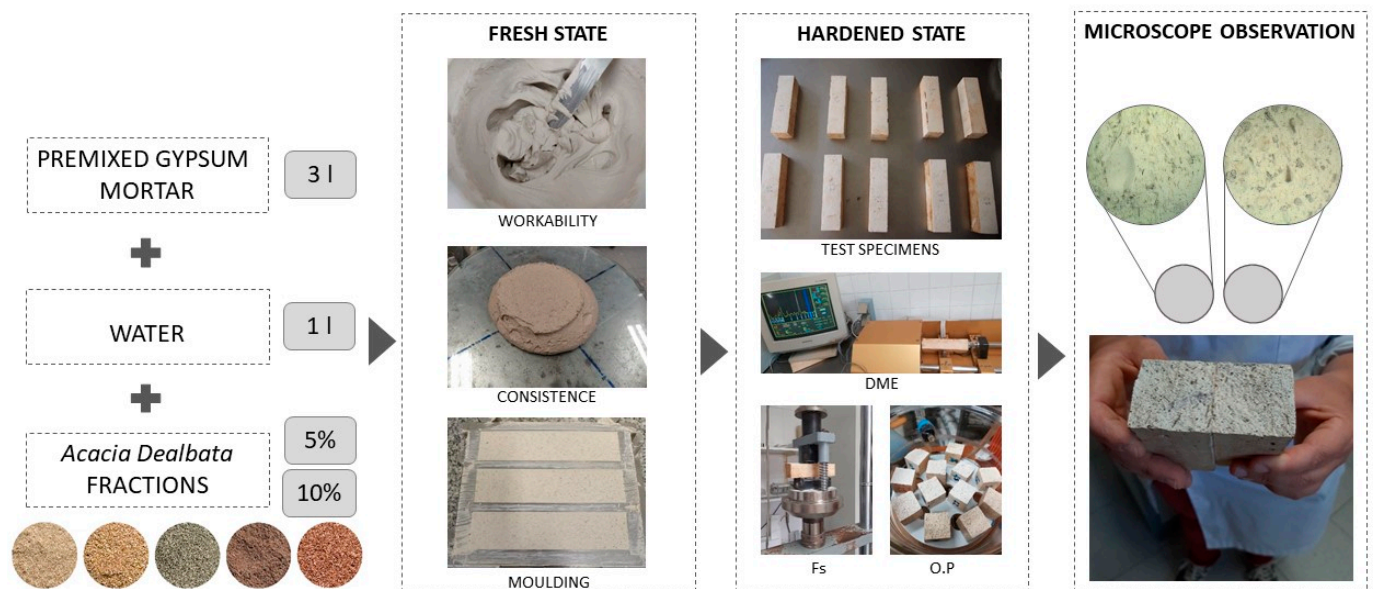
The powder mix was added to water for 1 min using the sprinkling method, left to soak for another minute, and then mechanically mixed for 1 min (Figure 2). A volumetric ratio of 1:3 (water:GP) corresponding to a ratio by mass of 0.45, was kept for all the mortars (Table 2).

## 2.3. Specimens and Methods

In Figure 3 a flowchart is presented to resume the experimental steps.

**Table 2.** Composition of the mortars and flow table consistence.

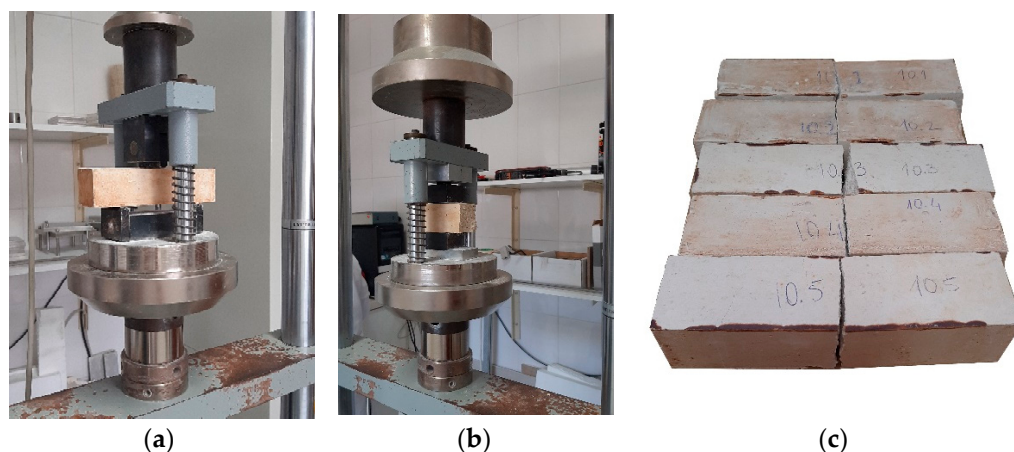
Mortar	Acacia Fraction	Addition (vol, %)	Addition (Mass, g)	Flow (mm)
REF	–	–	–	163.0
Fl5	Flowers	5	32.6	163.0
Le5	Leaves	5	62.2	168.5
Br5	Branchlets	5	56.7	171.3
Wo5	Wood	5	31.7	160.3
Ba5	Bark	5	65.2	175.5
Fl10	Flowers	10	65.3	165.5
Le10	Leaves	10	124.4	156.5
Br10	Branchlets	10	113.5	171.0
Wo10	Wood	10	63.3	165.3
Ba10	Bark	10	130.4	165.0

**Figure 2.** Mortar preparation: (a) sprinkling method, (b) soaking, and (c) mechanical mix.**Figure 3.** Experimental flowchart. DME, dynamic modulus of elasticity; Fs, flexural strength test; O.P., open porosity by vacuum and hydrostatic weighing.

The fresh mortars were tested for consistency using the flow-table method, determined based on EN 13279-2 [32] at a fixed amount of water. The fresh-state density was determined based on EN 1015-6 [33]. For the hardened mortars, a minimum of five standardized prismatic specimens (160 mm × 40 mm × 40 mm) were produced for each formulation and the following properties were determined:

- Volumetric shrinkage—determined using a digital caliper.

- Apparent bulk density—based on EN 1015-10 [34]—geometrically determined using a digital caliper and a balance with 0.001 g resolution.
- Flexural and compressive strengths—based on EN 1015-11 [35]—using an electromechanical testing device from Microtest, model EM1/100/FR. The loading rates were adjusted so that failure occurred within a period of 10–25 s for flexural strength and 30–90 s for compressive strength. Load cells of 2 kN and 200 kN were used, depending on the mechanical strength of the material (Figure 4).
- Dynamic modulus of elasticity—based on EN 14146 [36]—by resonance frequency using a Zeus ZRM equipment.
- Optical microscope observation—using an Olympus SZH-10 optical microscope.
- Open porosity—based on EN 1936 [37]—by vacuum and hydrostatic weighing.

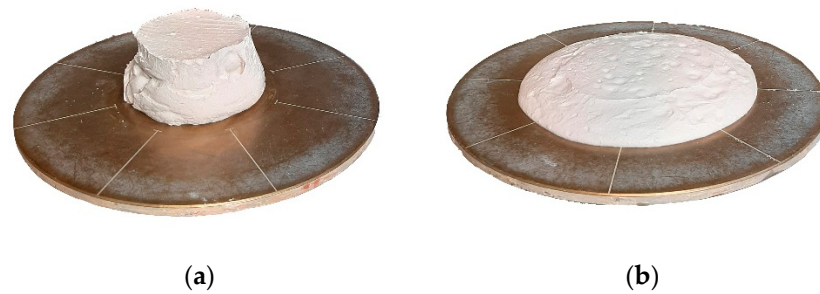


**Figure 4.** Flexural (a) and compressive (b) strength tests, and Wo10 specimens after the flexural tests (c).

### 3. Results and Discussion

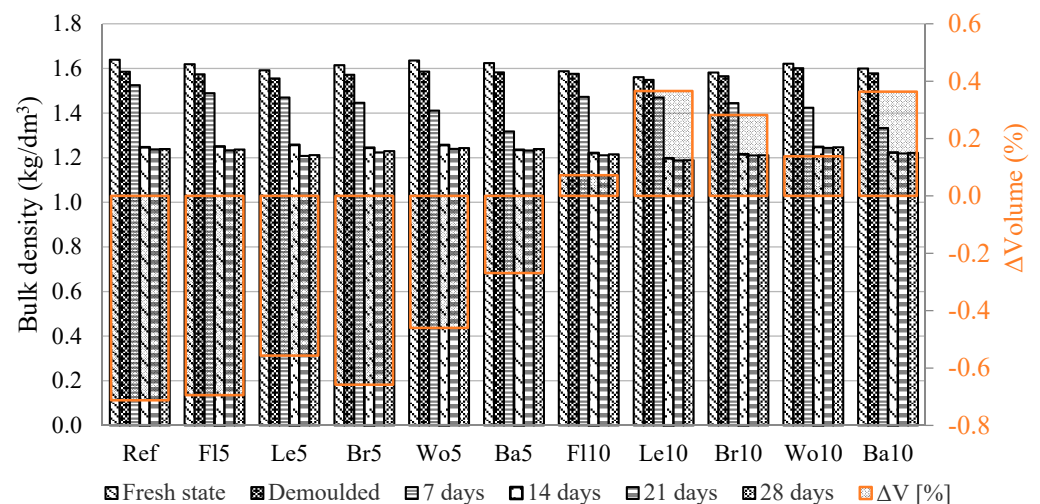
#### 3.1. Flow-Table Consistency, Density, and Drying Shrinkage

The consistency of the mortars (Figure 5) was determined using the flow-table method and the results obtained are presented in Table 2. All the mortars showed adequate spreading values, coherent with a good workability. Except for wood, the addition of 5% of biomasses slightly improved the flow of the mortars. The same happened for the 10% addition, except for the leaves. Therefore, the influence of the biomasses was in general very positive, as the additions did not required additional water to maintain workability. Moreover, it was expected that, for a fixed amount of water, an increase in the volume of powders would have led to lower values of flow-table consistency. This was not observed in the case of flowers and wood which, on the contrary, showed a flow increase. This may have been related to the combined effects of different particle size distribution, particle shapes, and hygroscopicity of the different biomass fractions. Hygroscopicity of a biomass is typically higher than that of biochars and affects the water available for hydration of other mortar components [38]. Unlike biochar particles or sand particles, biomass particles have irregular shapes that influence the mechanical interactions between themselves and other mortar components, thus affecting the flow behavior of the wet mortars [39]. These observations suggest that further tests using biomass particles of different granulometries may help to elucidate these effects.



**Figure 5.** Flow-table consistency at the removal of the cone of the reference mortar (a) and after jolting the flow table fifteen times (b).

Figure 6 shows the values of the bulk density of the mortars from the fresh state to the 28th curing day. It can be easily observed that during the first 14 days, the density decreased for all the formulations, and that the drying essentially occurred during that period. The addition of biomasses, in both volumetric percentages (5% and 10%), did not significantly modify the bulk density of the mortars which was around  $1.6 \text{ kg/dm}^3$  in the fresh state and  $1.2 \text{ kg/dm}^3$  from the 14th curing day onwards. Only for the plastering mortars with addition of bark did the first 7 days of curing show a higher decrease in bulk density, possibly caused by a faster evaporation of water. The bark particles had some differences in their contents of cellulose, hemicellulose, and lignin, relative to the other biomass fractions [40], which determines the surface groups of these particles and may have influenced their interactions with water molecules. Generally, the volume reduction was lower for the mortars with 5% biomass addition relative to the reference mortar, while all the formulations with 10% (vol.) biomass addition showed a slight volume increase up to one month of age. No cracks were observed in either the reference or the modified mortar samples. Therefore, the addition of biomass had a positive effect in the prevention of significant volume variation that could cause plaster cracking or lack of adherence to the support.



**Figure 6.** Bulk density of the tested mortars and, in orange, the volumetric drying shrinkage at 28 days.

### 3.2. Flexural and Compressive Strength

Flexural and compressive strength tests were performed after 30 days. All the mortars with added of bio-based wastes presented lower flexural and compressive strengths than the reference mortar. This was much less significant for those with addition of the wood fraction, especially at 5%. Indeed, a general decrease was noticed, namely between 40

and 55%. Moreover, a higher content of the same biomass usually corresponded with a lower flexural strength of the mortar, except for leaves and branchlets where the content presented insignificant differences. Although the latter showed a similar flexural strength at 5% and 10% of biomass addition, the compressive strength increased with an increase in biomass. In addition, bark showed this tendency to a small degree, whereas particles of flowers and wood reduced the compressive strength with increased contents. As mentioned previously, the different fractions of *A. dealbata* biomass have different amounts of cellulose, hemicellulose, and lignin. This affects their surface and their tenacity during milling, resulting in different particle shapes and different particle size distributions [40]. These different characteristics will also affect chemical and physical interactions with other mortar components, thus influencing mechanical properties. This tendency for a decrease in mechanical properties with the addition of biomass was already observed by other authors [41,42]. Morales-Conde [43] found that not exceeding 5% of sawdust incorporation on gypsum mortars led to an improvement of flexural strength, whereas all the percentages of sawdust additions decreased the compressive strength. The same authors related this phenomenon to a discontinuity introduced by the particles in the gypsum matrix which might have caused a reduction of strength. A lower hydration rate in the composites was also referenced by Chiki et al. [44], Panesar et al. [45] (2012), and Fatma et al. [42]. Nevertheless, all the mortars fulfilled the flexural and compressive strengths requirements of the EN 13279-1 [31] for gypsum plasters, as presented in Figure 7.

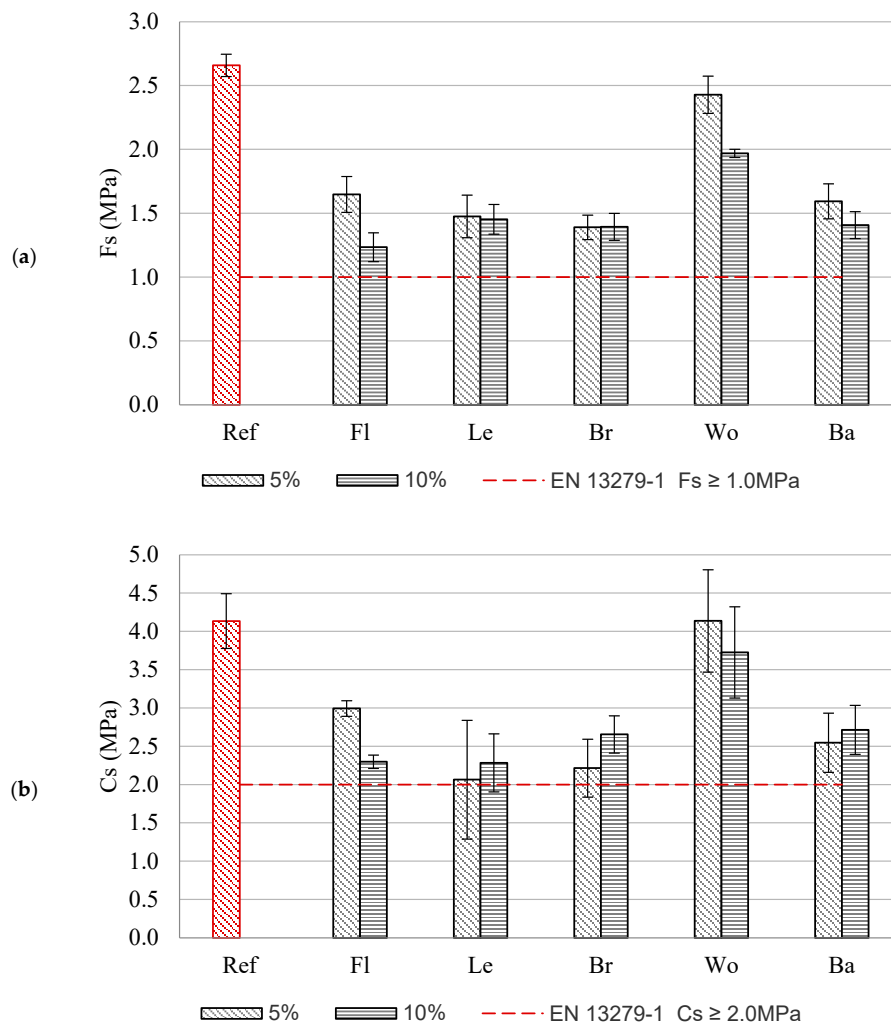
### 3.3. Dynamic Modulus of Elasticity (DME)

The dynamic modulus of elasticity (DME) generally showed a similar tendency to the flexural and compressive strengths. The incorporation of 5% and 10% (vol.) of different *A. dealbata* fractions introduced a decrease in the DME of the mortars (Figure 8). Moreover, the higher the volume of biomass added, the lower the modulus, as expected. These results can indicate a higher deformability of the mortars, which can lead to a lower susceptibility to cracking phenomena. Nevertheless, the particles of biomass could be responsible for the DME decrease by triggering new voids in the mortar matrix, as evidenced by the Olympus SZH-10 optical microscope observation (Figure 9). A poor interface between the gypsum matrix and the sawdust particles, and a high water absorption of the sawdust were found to be responsible for the poorer mechanical behavior of the gypsum–sawdust composites studied by Dai et al. [46]. Some differences on the hydrophilic nature of the fractions of *A. dealbata* may have led to a high absorption of the mixing water and a consequent increase of volume of the biomass particles during the mixing process. Thus, less water available during the gypsum hydration process could have led to a lower hydration rate (with lower mechanical properties) of the modified mortars when compared to the reference one. Moreover, once dried, the particles of biomass could have lost their gained volume creating the big voids that were found (Figure 9). The biomass particles could also have physically replaced a corresponding volume of the gypsum paste, therefore, decreasing the mechanical properties of the mortar.

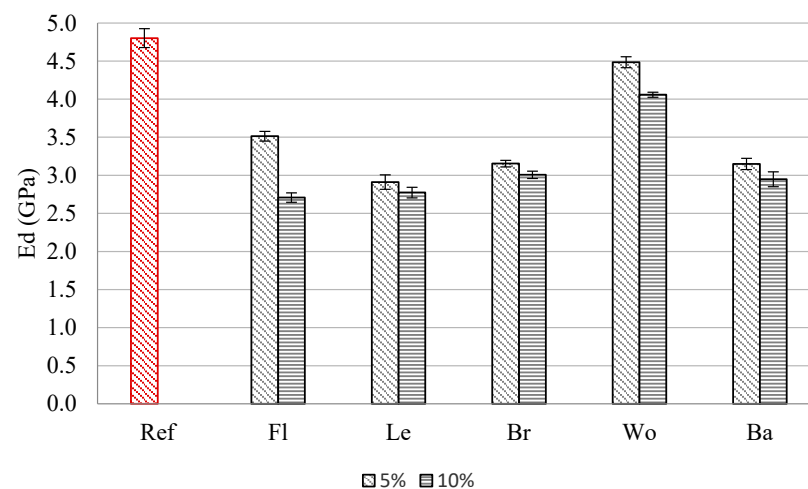
The color of the dry mortars was also assessed by naked eye and optical microscope observation. It could be seen that the differences in color due to the addition of the different biomasses and contents was not relevant when compared with the reference mortar.

### 3.4. Open Porosity

The values of open porosity of the eleven mortars were quite similar, although a small amount of variation was observed (Figure 10). All the mortars were highly porous, with values around 40%. The addition of flowers, leaves, and branchlets increased the porosity (the higher the percentage, the higher the effect). The addition of wood and bark, instead, kept the open porosity below the value of the reference mortar. The results agreed with the observed values of DME and flexural and compressive strengths, whereby the mortars with incorporation of *A. dealbata* wood particles showed the lowest open porosity and one of the highest mechanical properties.

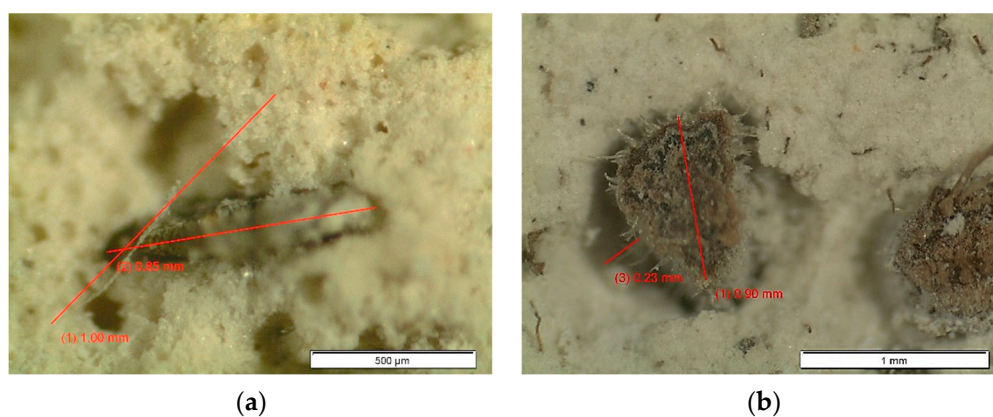


**Figure 7.** Flexural (a) and compressive (b) strengths of the mortars—average values and standard deviation. Dashed red lines represent the lower limits of the EN 13279-1 [31] for gypsum plasters (B1 to B6 class).

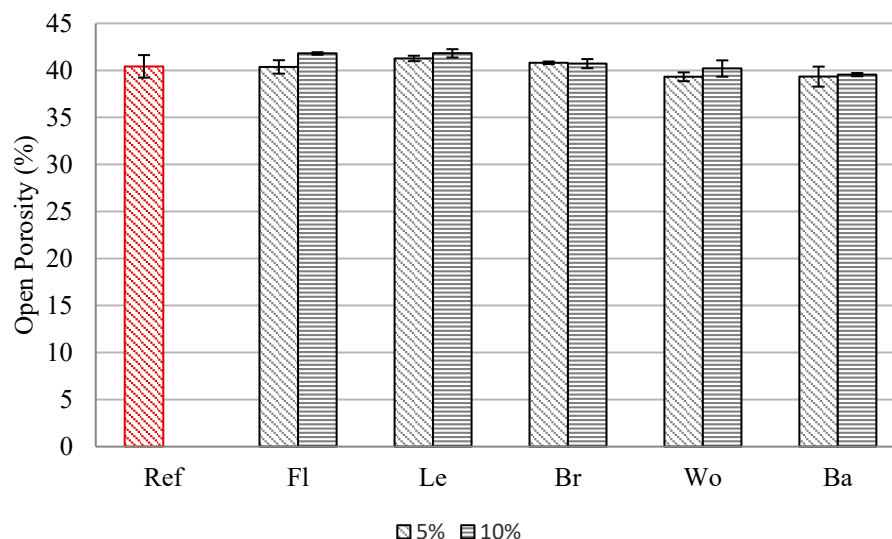


**Figure 8.** Dynamic modulus of elasticity of the tested mortars—average values and standard deviation.





**Figure 9.** Optical microscope observations showing voids introduced by the biomass in Le5 (a) and Fl10 (b) mortar.



**Figure 10.** Open porosity of tested mortars. The average values and the standard deviation were calculated from three specimens.

#### 4. Conclusions

After the removal of extracts that can be used in value-added chemical products, the wastes of five different fractions of *A. dealbata*, an invasive species in many countries, were added to a premixed gypsum plastering mortar in 5% and 10% volumes.

The study showed that the addition of *A. dealbata* biomasses did not significantly affect the workability of the mortars and their water requirements—seven mortars out of ten showed a higher flow-table consistency than the reference mortar despite a fixed amount of water being used for all. In addition, the bulk density, open porosity, and color were not appreciably modified by the additions, but a general decrease in mechanical properties was observed. However, mortars with all the studied fractions and contents presented flexural and compressive values that complied with the requirements of EN 13279-1 [31] for gypsum plasters. *Acacia dealbata* wood showed the closest mechanical properties to the reference mortar, having the best potential to be used as an addition to gypsum mortars, at least up to 10% volume incorporation rate, without causing relevant changes in their workability, density, or flexural or compressive strengths. Thus, this study allows us to conclude that the gypsum-based products with the inclusion of the studied additives are viable as plasters, according to EN 13279-1 [31].

Further studies will assess whether the addition of these biomasses, beyond their advantages in the reduction of incorporated energy, can improve the hygroscopicity of

gypsum plasters and, therefore, their passive contribution to comfort and health in indoor environments. Moreover, the biomass fractions which had more negative effects on the mechanical properties of the studied plasters, might still present a high relative humidity passive regulation performance and therefore be promising for this reason.

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