Article

Advancing the Circular Economy: Reusing Hybrid Bio-Waste-Based Gypsum for Sustainable Building Insulation

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Abstract: Finding eco-friendly products that are beneficial to the environment and serve as tools for sustainable development is a contemporary challenge. This work illustrates the recovery of bio-waste-based materials, which not only improve the hygrothermal properties of gypsum but also promote the paper and wood recycling processes in a circular economy approach. The samples were subjected to tests for density, water absorption, ultrasonic pulse velocity, flexural strength, compressive strength, and thermophysical property characterization. A statistical analysis of variance was used to study the impact of waste on the physico-mechanical behavior of gypsum, leading to the development of predictive models that can be used to predict and optimize the performance of bio-composites in various applications. The results revealed a reduction in mechanical strength with the addition of waste, but the samples still exhibit superior insulation properties, surpassing commonly used standard boards. By adding ouate and wood wastes to a mass of 20% in its natural state, the gypsum becomes lighter and acts as a better insulator with a reduced density, thermal conductivity, and ultrasound velocity of up to 50%, 57%, and 83%, respectively. These findings show the significant implication of reducing environmental impacts while contributing to the promotion of sustainable building practices, both in new construction projects and in building renovations.

Keywords: biobased material; gypsum; cellulose; sustainable development; waste recovery; statistical analysis ANOVA

1. Introduction

Considering the ambitious targets for reducing energy consumption and CO_2 emissions in the construction industry, which are accompanied by widespread pollution of all environmental components, including climate change [1], it is especially crucial to consider the building systems, materials, and initial passive house design to significantly reduce energy consumption [2,3]. Ceiling fans, for example, are an effective building technology that can cut cooling energy by 32% while increasing thermal comfort in hot, humid spaces [4]. In this context, the International Energy Agency (IEA) advises enhancing the building envelope, primarily relying on the enormous advances in materials research and industrialization, including the thorough characterization of natural solutions [5]. The development of new materials based on renewable raw resources with a low environmental impact has become a key issue for research and engineering. The use of recycled agricultural by-products or natural industrial waste in the manufacture of construction materials is a compelling strategy for addressing environmental challenges and enhancing reliability.

This approach not only contributes to resource conservation but also plays a key role in improving the reliability of construction materials [6]. Biobased construction materials can improve the hygrothermal performance of buildings. This is due to their unique ability to absorb and release moisture, facilitating natural ventilation. In addition, thanks to their superior thermal insulation capabilities, bio-based materials aim to create environments that naturally regulate temperature levels, thereby reducing reliance on heating and cooling systems. This dual-purpose strategy effectively addresses both issues, promoting energy efficiency, reliability, and overall comfort in the built environment. In this case, the correct management of waste derived from raw materials of natural origin in the paper and wood industries can be used as reinforcements in building materials. In particular, the amount of wood waste varies significantly depending on the typical structure or building typology of the analyzed country or region. Since wood is used in massive quantities in many different sectors and is part of our daily lives, the available quantities of sawdust and other recycled wood are also important. Wood waste can be made up of Sawdust particles or wood shavings that are left over after sawing wood into useful, uniform sizes. Recycling wood shavings is fascinating from a dependability standpoint [7]. About 30 million tons of recycled wood are produced in Europe each year [8], according to COST Action E31. It is particularly important in Nordic countries, such as Norway, where it accounts for 10.12% of total construction waste [9]. In addition, as in the case of paper waste, for example, global estimates indicate that more than fifty million tons of paper products are consumed, and those elements exceed the required limitations [10]. Given that significant amounts of waste are produced during the pulp manufacturing process, the paper and board industry faces several difficulties in this regard [11]. The production of paper has a detrimental effect on the environment.

In practice, according to the literature, to achieve acceptable mechanical properties and reliability, most developed biobased materials use lime [12,13] or cement [14,15] as their primary binders. Additionally, the use of these binders has a detrimental impact on the environment, for instance. Cement production is responsible for 8% of greenhouse gas emissions [16]. An amount of 900 kg of CO_2 is produced in one ton of cement production. The global cement industry is predicted to produce 2.34 billion tons of CO_2 emissions by 2050 if nothing is carried out to reverse this trend [17]. On the other hand, the lime industry also has a significant CO_2 emission rate. Taking into account the amount of CO_2 fixed during the carbonation of lime, the CO_2 emission rate is estimated to be between 335 and 415 kg/t [13]. Therefore, some researchers have pointed to the use of gypsum in the production of biobased composites because it is the least polluting building material due to its low energy consumption during production [18,19]. An amount of 140 kg of CO_2 is produced during the production of one ton of natural gypsum. When compared to Portland cement, emissions of carbon dioxide from natural gypsum as a raw material were found to be at least five times lower [20]. It is also worth noting that gypsum has a high specific heat, which improves thermal diffusivity. This property enables the material to better distribute and transfer heat, resulting in better thermal regulation in buildings. Furthermore, gypsum has fire-resistant advantages. Because of its crystalline structure, gypsum is a fireproof material that can retard the spread of flames and limit the spread of heat during a fire. As a result, it is a safe choice for construction applications where fire resistance is critical [21].

Gypsum was chosen as a binder for this work because it is a plentiful resource in Tunisia (the second-world producer). This natural Tunisian material is generally made of calcium sulfate hemihydrate, as in other countries, and is classified among the highest-quality gypsum in the world, with more than half of the production exported to Europe [22,23].

Given that the construction industry is responsible for consuming 40% of the world's granular materials and emitting 50% of CO_2, it is critical to prioritize the use of environmentally friendly building materials. Biobased materials offer a significant advantage via the use of renewable resources in construction instead of relying on nonrenewable minerals. The synthetic and chemical products commonly used in construction can be toxic

and non-recyclable, posing a threat to both human health and the environment. Biobased building materials provide a sustainable alternative via the utilization of materials that are dependable and do not harm the Earth's crust [24]. The literature reports several benefits of using natural aggregates to create materials with various use criteria. In terms of thermal behavior, the lowest thermal conductivity is the criterion for lowering building energy consumption. Studies have suggested insulation materials and demonstrated the benefits of natural waste/fibers such as date palm fibers [14], cellulose fibers [25,26], wood waste [7,27], wheat straw [28], Posidonia oceanica fibers [29], rice straw [18], and Sargassum muticum [30].

The wadding (ouate) is derived from natural paper pulp waste, and several studies have explored the potential for creating composites from paper residues. For instance, Oliveira et al. [25] reported on the development of biobased gypsum using recycled cellulose. The gypsum with a 60% cellulose content exhibited a thermal conductivity (0.32 W/m.K) of the composite material that was 40% less than that of the reference gypsum. Although the mechanical resistance of these gypsum composites is insufficient for certain applications, they are recommended for use in insulation boards to enhance the thermal performance of buildings. Additionally, Mandili et al. [12] made a similar observation when they confirmed that a composite material composed of paper pulp waste with lime as a binder had a significant thermal insulation capacity (varying between 0.097 and 0.12 W/m.K). This was attributed to the low values of thermal conductivity exhibited by the composite, which can be explained by the high porosity of the paper waste. Balti et al. [31] demonstrated that incorporating paper waste into gypsum resulted in a reduction in density and brittleness in the materials. Despite this, the composite exhibited sufficient mechanical properties, a 2.45 MPa flexural strength and 5.07 MPa compressive strength, for use in construction, particularly as interior insulation materials.

In the case of wood waste, it can be generated in the form of wood shavings or sawdust. These wastes are nevertheless used in construction applications; typically, they are added to cement- or gypsum-based mortars to reduce cracking and improve hygrothermal properties. For example, Pedreno-Rojas et al. [9,32] conducted a study to evaluate the mechanical and physical properties of new composites made from gypsum and wood waste. By adding recycled wood to a gypsum matrix, they were able to produce more eco-efficient composites. The new material improved its thermal and acoustic insulation capabilities compared to those of the reference material, while becoming lighter as the amount of wood waste increased. However, this improvement came at the expense of the mechanical strength of the new composites, which decreased as the amount of wood waste increased. When comparing composites based on the type of waste added, those containing wood shavings were consistently lighter and exhibited better thermal performance than those containing sawdust in the same percentage. Conversely, the composites containing sawdust had better mechanical behavior and acoustic absorption. In a similar study, Dai et al. [33] developed a composite made of gypsum and sawdust. The sawdust used in the composite contained tannins, acetic acid, hemicellulose, and lignin, which were identified through attenuated total reflectance–Fourier transform infrared (ATR-FTIR) analysis. The mechanical results of the study suggest that the decrease in gypsum performance observed may be attributed to water absorption from the sawdust. Corinaldesi et al. [34] investigated the flexural and compressive strengths, as well as the thermal conductivity, of cement mortars containing varying amounts of wood waste. The results indicated that sawdust-based mortars performed better than wood shavings-based mortars did, but a higher dosage (10% instead of 5%) strongly degraded the mechanical performance of the mortar. In fact, the compressive strength of mortars containing 10% wood waste never exceeded 5 MPa in compression. The addition of wood by-products had a less significant negative effect on flexural strength than it did on compressive strength, likely due to the fibrous structure of the wood shavings. Furthermore, there was a positive influence on the reduction in water vapor permeability and thermal conductivity, which decreased by 25% with the addition of 5% waste. However, capillary water absorption slightly increased.

Similarly to this, Aigbomian et al. [8] developed a new building material out of lime, wood, and waste paper. Similarly, the compressive value of the resulting wood concrete varied from 0.06 MPa to 0.80 MPa, suggesting that it can be used as a filler for hollow blocks and wall panels. Furthermore, the product had high thermal conductivity, with values varying from 0.046 to 0.069 W/m.K. According to the literature, it is generally supported that the incorporation of cellulose waste in building materials can lead to a reduction in their mechanical performance while improving their thermal performance.

Biobased building materials are long-lasting and have a positive impact on social wellbeing. It has been observed that this pragmatic material is an unrivaled alternative, not only because it reduces carbon and energy emissions but also because it provides thermal comfort with lower energy consumption for building operations when used in place of conventional materials [35].

The main aim of this paper is to utilize locally available cellulosic waste to propose a construction material that is durable, lightweight, and suitable for thermal and acoustic insulation while also being capable of supporting loads. We investigate the influence of waste from ouate (OW) and wood shavings (SW) on the physical properties (density, water absorption, and ultrasonic pulse velocity), mechanical properties (flexural and compressive strengths), and thermo-physical properties (thermal conductivity, diffusivity, and volumetric specific heat) of the biobased gypsum materials. Following the experimental phase, a meticulous statistical analysis was conducted to exhibit the efficacy of various waste materials. This methodology aims to ascertain the representativeness and robustness of the experimental data on one hand. On the other hand, it also facilitates the comprehensive comparison of the significance of the controlled variables in the experiment (mechanical and thermophysical properties) on the developed specimens. Moreover, this study not only aims to improve the characteristics of gypsum but also to valorize these waste materials and provide a solution for their recycling in building materials like gypsum.

2. Materials and Methods

2.1. Raw Materials

In this study, the matrix material is gypsum, manufactured by MEDGYP, a company in Tataouine, Tunisia. This gypsum satisfies all of the requirements of EN 13279-1 [36]. Table 1 lists its characteristics. The wood workshop generated shaving waste (SW) with particle sizes that ranged from 0 to 10 mm (Figure 1a). Furthermore, ouate waste (OW) generated by the "Tunisie-Ouate" company in Enfidha, Tunisia, was used. The ouate pulp was dried and crushed into particles with a size range of 0 to 5 mm (Figure 1b).

Table 1. Characteristics of the gypsum used.

Purity		>94%
Bulk density		>600 g/L
Flexural strength (MPa)	2 h	>1.5
	7 days	>3
Surface hardness (N/mm^2)	2 h	>4
	7 days	>10

2.2. Sample Preparation

For each formulation, as shown in Table 2, prismatic test specimens with $16 \times 4 \times 4$ cm^3 dimensions were prepared in accordance with the EN 13279-2 standard [37]. The percentages of each compound added are based on the weight of gypsum and were determined using data from the literature and preliminary tests. These results indicate that a lower percentage of gypsum was inadequate for achieving homogenization and the binding of the mixture, leading to the production of brittle samples. The following is the sample designation code: the reference sample (neat gypsum) is denoted by REF. O35W5 represents gypsum containing 35% ouate and 5% wood. O30W10 represents gypsum containing 30%

ouate and 10% wood. O25W15 represents gypsum containing 25% ouate and 15% wood. Lastly, O20W20 represents gypsum containing 20% ouate and 20% wood.
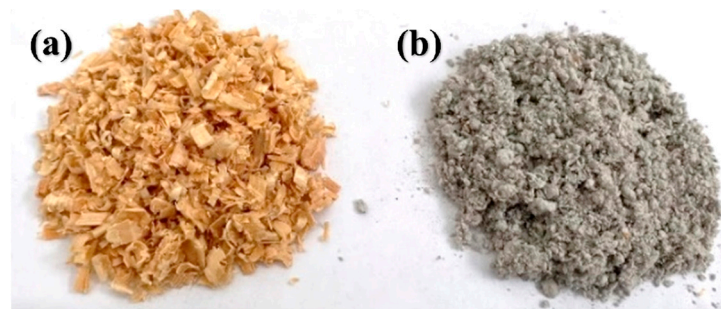

Figure 1. (**a**) Wood shavings and (**b**) ouate wastes.

Table 2. Summary of the compositions studied.

Sample Numbers	Sample Designation	Gypsum (wt. %)	Ouate Waste (wt. %)	Wood Shavings (wt. %)
	REF	100	0	0
1	O35W5		35	5
2	O30W10	60	30	10
3	O25W15		25	15
4	O20W20		20	20

O: ouate and W: wood.

For the best mixing performance, combine the gypsum with the waste material before adding it to the water [38]. As seen in Figure 2, a combination of gypsum, ouate (OW), and wood shavings waste (SW) was mixed with 350 mL of water in a mixer, homogenized with an additional 50 mL of water, and continuously mixed for 5 min. The final product was put into a mold. The specimens were kept in the laboratory after demolding at 25 °C and $50 \pm 1\%$ relative humidity. Three duplicate samples of the same mixture were created to estimate the standard deviation.
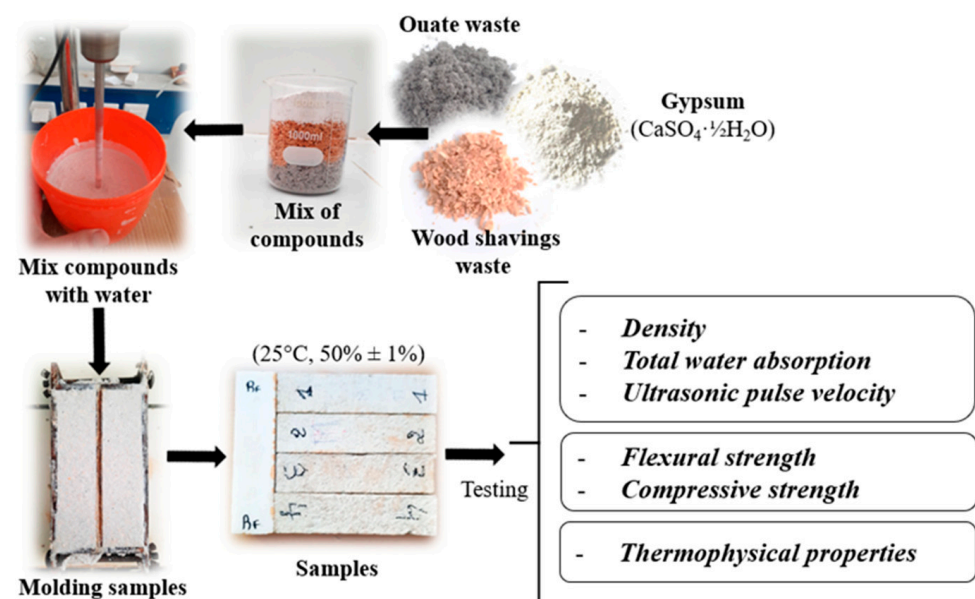

Figure 2. The process of preparing and characterizing biobased gypsum composites.

2.3. Statistical Analysis

Following the testing stage, the data underwent thorough statistical analysis with the following objectives: 1—to validate the model characterizing response variation within the experimental domain; 2—to confirm the representativeness and robustness of the experimental data; 3—to assess the significance of the variables under control in the experiment concerning both the thermophysical and mechanical properties of the specimens; 4—to evaluate the residual variance of a model based on the controlled variables, aiming to comprehend their collective impact on thermo-physical and mechanical properties.

As shown in Table 3, ouate waste (OW) and wood shavings waste (SW) percentages were chosen as variable factors X_1 and X_2, respectively. The lower and upper constraints of each component were chosen based on Table 2. For this statistical analysis, the response variables density, water absorption, ultrasound velocity, flexural strength, compressive strength, and thermal conductivity were assessed using a multiple linear regression model. To perfectly fit the two tested factors, the first-degree polynomial model presented in Equation (1) was used. The data in this paper were analyzed using the statistical and graphical analysis program NemrodW® (Version 9901). All experimental data were subjected to an analysis of variance based on the Fisher test (F-test) for data and model reliability [31,39]. Each analysis yielded an ANOVA table with the significance of each factor, the residual deviation of the model, the coefficient of determination (R^2), the adjusted coefficient of determination (R^2_{Adj}), and the effect size (the partial square root mean square (ηp^2)). When the estimated probability (*p*-value) is less than the specified probability (0.05), the coefficient is viewed as significant. The percentage achieved with the F-test is represented by the stars asterisk (*).

$$Y = b_0 + b_i \times X_i + b_j \times X_j + b_{ij} \times (X_i \times X_j) \tag{1}$$

where Y is the estimated response. X_i and X_j are the independent variables. b_0, b_i, b_j, and bij are the coefficients of the model.

Table 3. Independent variables with low and high levels.

Factor	Code	Unit	Levels	
			Lower Level	High Level
OW	X_1	wt. %	0	35
SW	X_2	wt. %	0	20

OW: ouate waste, SW: shavings waste.

2.4. Experimental Test Methods

2.4.1. Physical Properties

- Density

The density was calculated in accordance with EN 13279-2 [37]. The dry volume and weight of each sample were measured to determine its density, which is described as the ratio of weight (g) to volume (cm^3) of the sample. The average value of three measurements was used to calculate the density of the developed composite.

- Total water absorption

The amount of water that accessed the material was measured to determine water absorption. The sample was immersed in water for 24 h until saturation after being dried at 50 °C for weight stability. Equation (2) was used to calculate the water absorption (EN 1609 standard [40]).

$$W(\%) = \frac{m_w - m_d}{m_d} \times 100 \tag{2}$$

where m_w is the wet mass (g) and m_d is the sample's dry mass (g).

- Ultrasonic pulse velocity

To establish the relationship between the density and ultrasonic velocity of composite materials, a non-destructive test was conducted following EN 12504-4 [41]. The procedure involved measuring the fundamental resonance frequency of the specimens using the GrindoSonic MK7 instrument. The final value was calculated by averaging three longitudinal values of ultrasonic velocity obtained for each specimen.

2.4.2. Mechanical Properties

Mechanical characterization measurements were performed using the EZ multi test machine of 50 kN ((Lloyd Instruments SA, Montigny-Le-Bretonneux, France).

- Flexural strength

Samples of $4 \times 4 \times 16$ cm^3 dimensions were exposed to a three-point flexural test via EN 13279-2 [37]. The load was applied at a rate of 0.5 mm per minute at a similar distance from the supports until the material broke. The spacing between the supports was 140 mm (Figure 3a).
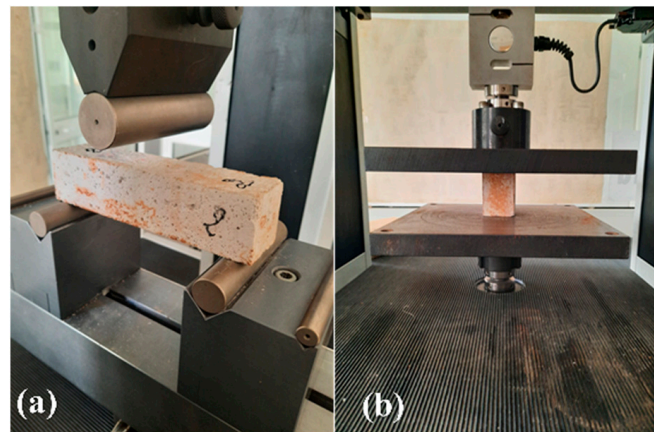

Figure 3. (**a**) Flexural and (**b**) compressive strength tests.

- Compressive strength

After performing the bending test, in accordance with the standard EN 13279-2 [37], the half prisms generated via flexural test fracture were sawed to $4 \times 4 \times 6.5$ cm^3 dimensions for the compression test (Figure 3b). A speed of 1 mm per minute was chosen for all the samples.

2.4.3. Thermophysical Properties

In this test, $4 \times 4 \times 16$ cm^3 samples were employed to measure the thermal diffusivity, thermal conductivity, and heat capacity of all prepared composites. A "TPS 2500 S" instrument from the company Hot Disk® (Gothenburg, Sweden), consisting of a sensor connected to a thermal constant analyzer, was used. The sensor was a "Kapton-isolated" model (ref A 5501) from Hot Disk® with a 6.4 mm radius and was chosen specifically for the sample dimensions. The Hot Disk technique is based on the theory of a transient planar source as described in ISO 22007-2 [42], where the probe is inserted between the sample and an insulating reference material with known thermophysical properties (polystyrene). The probe acts as a heat source to raise the sample's temperature and record the temperature rise over time. Power heating at 33 mW and an 80 s measurement time were the parameters used in the measurements. At room temperature, we made three measurements on three sides of the sample. An average of nine tests was used to calculate the experimental results and associated uncertainties for each sample. The experimental set-up shown in Figure 4 enables thermophysical properties to be measured simultaneously with, high accuracy and non-destructive.
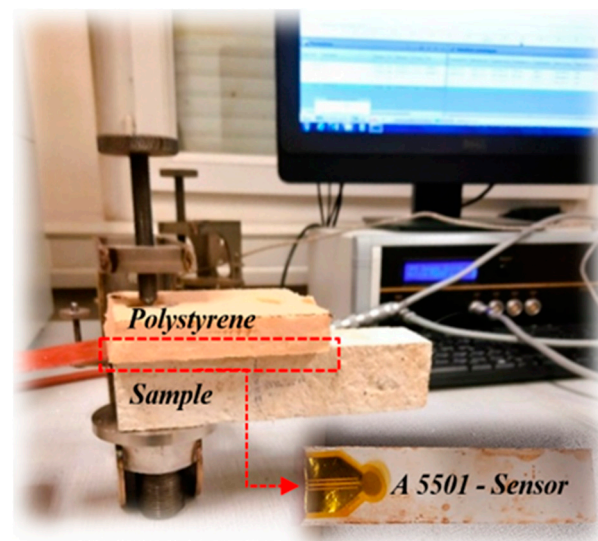
Figure 4. Sample thermophysical test with the Hot Disk setup.

3. Results and Discussion

3.1. Physical Properties

Figure 5 presents the mean density values for each composite, along with error bar intervals. As demonstrated, a considerable decrease in density ranging from 1.027 to 0.520 g/cm^3 is observed. This reduction can be attributed to the addition of ouate waste (OW), which has a porous structure and a lower density. The obtained result is in agreement with previous studies that have reported the impact of OW on the density of various building materials [25,43]. Furthermore, a noticeable decrease in density is observed as the proportion of SW increases and that of OW decreases. For instance, in comparison to the gypsum reference sample (REF), a reduction of approximately 0.35% and 50% was noticed for O35W5 and O20W20, respectively. This trend is in agreement with the results of Pedreño-Rojas et al. [9], who reported a value of 0.702 g/cm^3 with the addition of 40% of wood shavings, which is almost half the density of reference gypsum. In summary, it is clear from our results that mixed wood shavings and absorbent ouate waste can be used to create lightweight gypsum composites with densities of less than 1.00 g/cm^3.
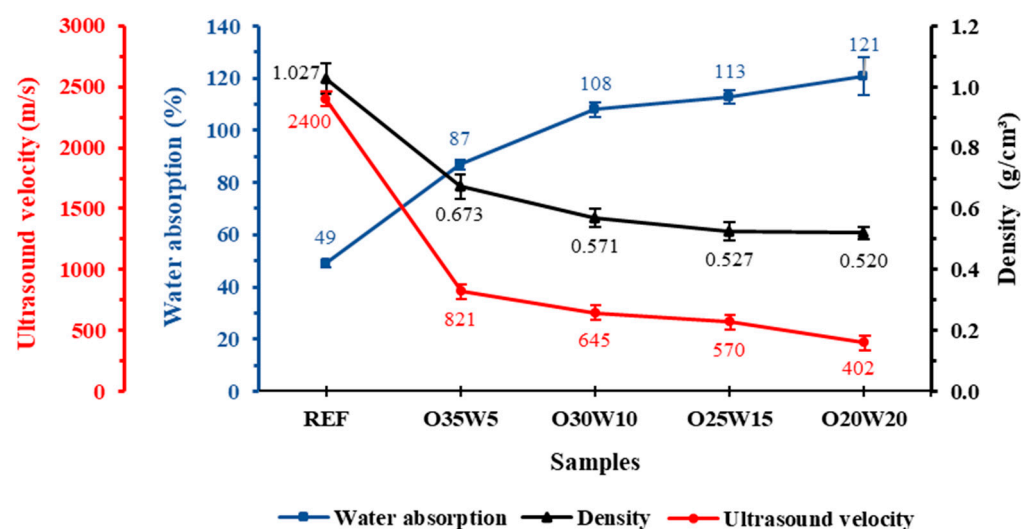

Figure 5. Physical property results: density, water absorption, and ultrasound velocity.

Figure 5 depicts the results of the water absorption test conducted on the biobased samples. The total water absorption of the samples varied from 49% for REF to 121% for

O20W20, indicating a substantial increase of 72%. This notable increase can be attributed to the inclusion of a hydrophilic natural waste source, which possesses a porous structure leading to high water absorption [44]. Moreover, to evaluate the water absorption capacity, the samples were subjected to prolonged immersion in water for a month, reaching a state of saturation until a weight equilibrium was reached. Following this total water absorption test, the resilience of these bio-composites in the presence of water became evident. Although it is logical to expect some degree of swelling in linear dimensions, no significant change was observed. Despite the reduction in material stiffness, further visual inspection revealed that the sample retained its structural stability.

Mandili et al. [12] reported that the addition of 60% cellulosic waste resulted in a water absorption value of 112% for gypsum. The water absorption values remain substantial due to the presence of cellulose pulp, which exhibits a water absorption coefficient exceeding 300%. Hence, we can deduce that the presence of ouate waste is the primary factor contributing to the significant increase in water absorption. Furthermore, Figure 5 demonstrates a gradual rise in water absorption as the content of SW increases and the content of OW decreases. Dai et al. [33] also found that the water content of gypsum composites increases with the gradual addition of wood waste, which aligns with the findings of this study. The progressive addition of wood waste leads to the increased water absorption of gypsum.

Figure 6 shows the exponential equation with $R^2 = 0.971$, which represents the relationship between the water absorption rate and the final density of the material. The water absorption is related to the variation in density, as the sample becomes denser as the water absorption of the material decreases.
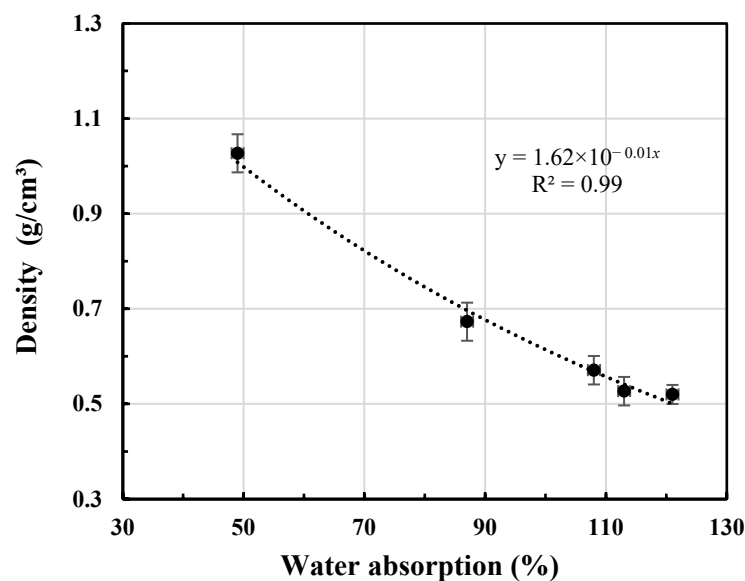

The equation shown in the figure is:
$$y = 1.62 \times 10^{-0.01x}$$
$$R^2 = 0.99$$

Figure 6. The relation between water absorption and density.

The variation in the ultrasonic pulse velocities (UPV) of gypsum with different proportions of ouate (OW) and wood shavings (SW) wastes is shown also in Figure 5. As expected, the ultrasonic velocity decreased with the addition of OW and SW to the gypsum. The UPV varied from 2400 m/s for the reference sample to 402 m/s for O20W20, so a decrease of 83% is observed. This significant decrease in UPV is related to the porosity of the materials, as the velocity generally decreases with increasing porosity [45], which in turn indirectly indicates poor compaction due to the larger pores in the gypsum mixtures. The pore structure of the material has a significant impact on this velocity, which offers information about the composite microstructure [12]. The prepared samples exhibited lower ultrasonic velocities as the SW content increased.

Extensive research has been conducted on gypsum to improve its thermo-mechanical properties through the incorporation of waste elements. However, the addition of these wastes can have an effect on the acoustic properties of the resulting composite materials. The introduction of cellulosic waste has been shown to significantly affect ultrasonic velocity, a significant acoustic characteristic. According to research, as the amount of waste in gypsum composites increases, the ultrasonic velocity decreases. The decline can be attributed to the difference in acoustic impedance between the waste material and the gypsum matrix. The composite's pores and microcracks are also contributing factors to the reduced ultrasonic velocity. Therefore, it is essential to consider how the decrease in ultrasonic velocity may affect the strength and stiffness of the composite when constructing gypsum composites with waste materials. In summary, the impact of incorporating waste materials on the ultrasonic velocity of gypsum composites highlights the importance of considering the acoustic properties of these materials when evaluating their suitability for building and construction applications [46,47]. Based on these results, we can affirm that this biobased composite material is a good sound absorber.

The composites were also subjected to the ultrasonic pulse velocity (UPV) test to assess the relationship between density and ultrasound velocity. Figure 5 clearly shows that the incorporation of the waste materials resulted in a linear decrease in the UPV. The ultrasonic velocities of the samples decreased with decreasing density. These results correlate with $R^2 = 0.99$. The results (Figure 7) exhibit a similar pattern to that in previous studies [26,47]. However, it is important to emphasize that, for data points representing aggregated values (four points between 0.55 and 0.70 g/cm^3), R^2 is likely to increase. A significant linear relationship was observed when comparing ultrasonic pulse velocity values with corresponding densities. This decrease in velocity can be linked to the presence of pores, which prevent the ultrasound waves from passing through the gypsum samples. As the number of voids increased, the weight of the gypsum composites decreased, leading to a reduction in density.
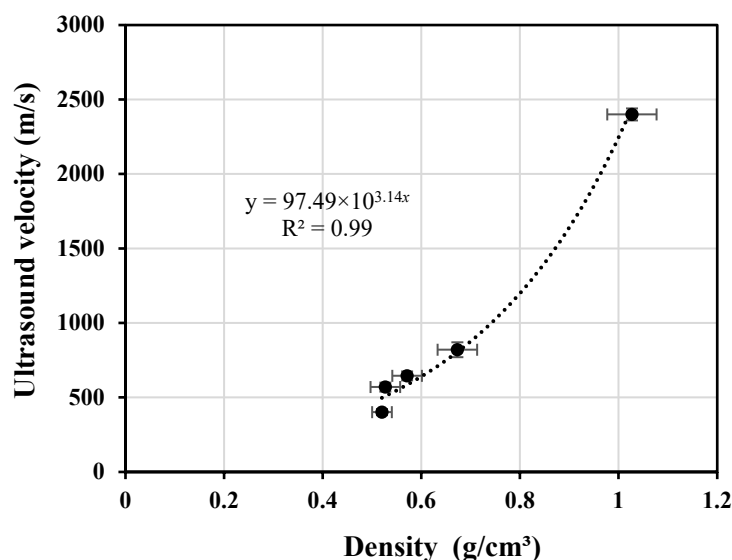

Figure 7. Relationship between UPV and density of samples.

3.2. Mechanical Properties

Figure 8 depicts the flexural and compressive strengths after. The resistance values of the composites are highly similar and of the same order of magnitude in both flexion and compression tests. The compression resistance is consistently higher than the flexion resistance for the reference gypsum (REF). A significant reduction in flexural and compressive strengths was observed with the incorporation of OW and SW. The values of mechanical strength decreased from 2.5 MPa to 0.2 MPa in flexural test and from 4.28 MPa to 0.26 MPa in compression. Sample O35W5 exhibits the highest compressive and flexural

values, measuring 0.88 MPa and 0.77 MPa, respectively. This decrease in resistance is clearly observed when the quantity of wood shavings increases at the expense of ouate content. These outcomes can be explained by the fact that OW and SW reduce the composite's strength and flexibility.
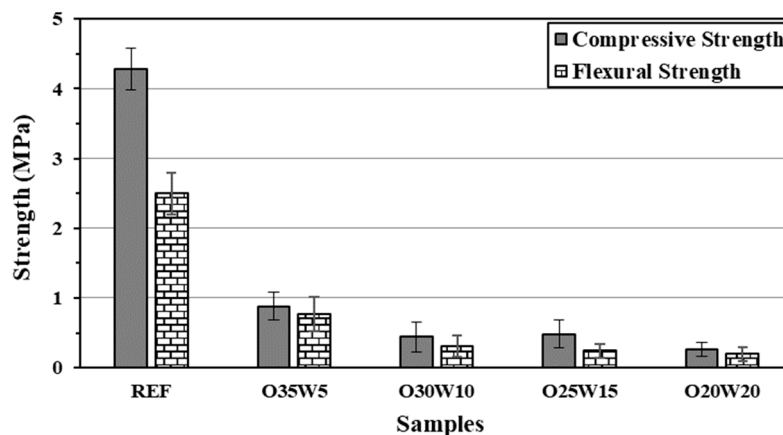

Figure 8. Flexural and compressive strengths of the samples.

The addition of cellulosic wastes, such as wood and ouate, to gypsum, can lead to a substantial decrease in its mechanical properties for several reasons. Firstly, cellulosic waste possesses a porous structure that can function as a stress concentration zone, consequently weakening the mechanical strength of the gypsum matrix. Additionally, the surface of the cellulosic waste may not fully adhere to the gypsum matrix, which can generate weak spots in the final composite material. Finally, the cellulosic waste could hold a high level of moisture, which can reduce the mechanical strength due to a weakening of the intermolecular bonds [9,12,18]. Morales-Conde et al. [48] have provided support for our concerns. It has been observed that resistance to gypsum bending decreases gradually when wood shavings are added in amounts more than 2.5%. Results from compression tests show that gypsum performed worse than the reference gypsum did, with a decline being in direct proportion to the addition of more wood waste. Dai et al. [33] have also found that the addition of 20% wood waste significantly reduces the mechanical performance of gypsum. Flexural and compressive strengths can be reduced by up to 64%. This reduction can be attributed to the addition of wood waste, which is the primary cause.

Figure 9 demonstrates the relationship between the average densities and mechanical properties of biobased composites. It is worth noting that the flexural and compressive strengths of prepared biobased samples decrease linearly with the decrease in gypsum composite densities. Therefore, a 50% reduction in density hurts gypsum's strength. The flexural strength dropped from 2.50 to 0.20 MPa, while the compressive strength decreased from 4.28 to 0.26 MPa and the density declined from 1.027 to 0.520 g/cm^3. These results are consistent with the expected behavior of strength decreasing with a drop in density. The study found a good correlation between bending and density (R^2 = 1.00), and compression and density (R^2 = 1.00). The addition of cellulosic waste may have contributed to the decrease in strength and the reduction in gypsum density.

The most prevalent kind of cracking in biobased gypsum is a ductile fracture (Figure 10), followed by shear cracks in the sample core. This demonstrates that developed composite gypsum has quasi-ductile behavior, as opposed to pure gypsum's sudden brittle failure.

Biobased materials, such as waste additives or natural fibers, often possess inherent ductile properties or bring additional flexibility to the composite structure. The inclusion of these materials in the gypsum matrix improves energy absorption and deformation capabilities, leading to a more ductile response when subjected to stress or strain. This ductile behavior allows the composite to undergo plastic deformation before fracture, resulting in the ductile fracture patterns observed. In addition, the interaction between biobased waste and the gypsum matrix can improve the overall toughness and resilience

of the composite, contributing to its quasi-ductile behavior. Overall, the incorporation of biobased materials into gypsum composites introduces ductile properties and improves the material's ability to resist stress and strain, leading to quasi-ductile behavior compared to that of pure gypsum.
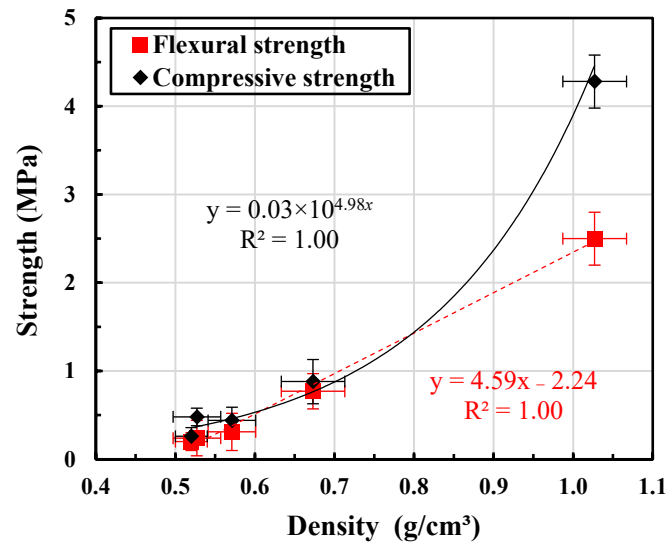

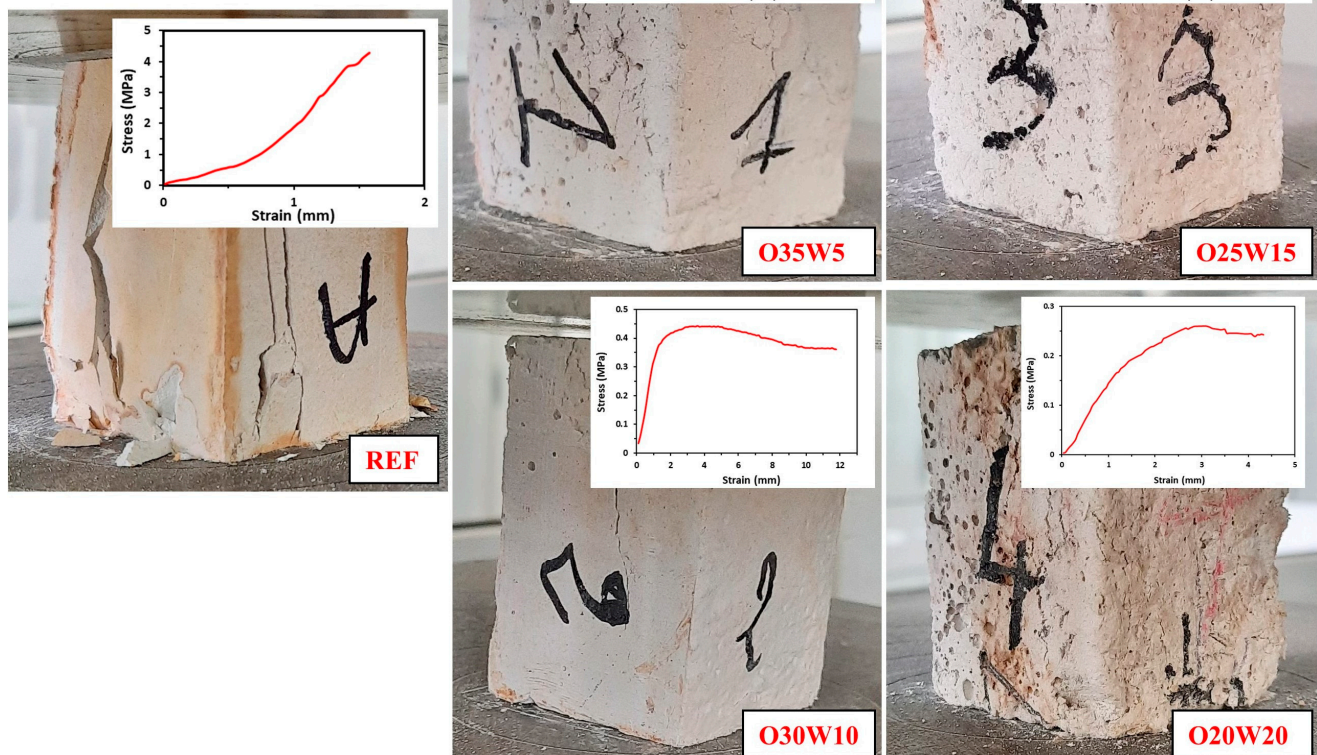**Figure 9.** Flexural and compressive strengths against density.

Figure 10. Behaviors of the samples after the compression tests.

Given that these lightweight materials, like other existing insulation materials, are intended to be used in conjunction with structural elements, this observed ductile behavior may be of interest because it improves the accommodation and fit between structural and non-structural elements, thereby improving the absorption of small dislodgments.

Due to their low mechanical strength values, the bio-composites studied are not suitable for use as structural materials, as expected. However, according to the EN 1992-1-1 standard [49], the materials meet the condition of bearing loads with their own weight. Also, according to the EN 998-1 standard [50], this product is applicable for use as a monolayer coating since it has compressive strengths superior to 0.4 MPa after 28 days under the CSI category. It is clearly demonstrated that the strength properties of biobased gypsum are not as good as those of normal gypsum but are comparable to those of lime–hemp or wood–Crete composites. For example, hemp concrete has a compressive strength of 0.4 MPa. The required compressive strength values for paper and wood-based materials with lime are between 0.21 and 0.49 MPa, and those for lime and cement are between 0.18 and 0.27 MPa [8]. The strength values of our composites, ranging from 0.26 to 0.88 MPa, are quite comparable to the compressive strength of the other composite developed for insulation. Thus, the compressive strength values are found to be higher than those of gypsum + 40% straw (0.031 MPa) [13], gypsum + 1.5% tragacanth + 80% polystyrene (0.35 MPa) [51], and gypsum + 0.5% vegetal protein (0.41 MPa) [52]. In conclusion, these biobased gypsum products ought to be utilized in buildings as fillers for hollow blocks and wall panels, as well as interior or insulating plaster.

3.3. Thermophysical Properties

As mentioned in Section 2.4.3, thermophysical measurements were performed using the Hot Disk method. Figure 11 shows the evolution of the thermal diffusivity, thermal conductivity, and heat capacity of the different samples with varying mass fractions of OW and SW. The error bars associated with the experimental data are also included.
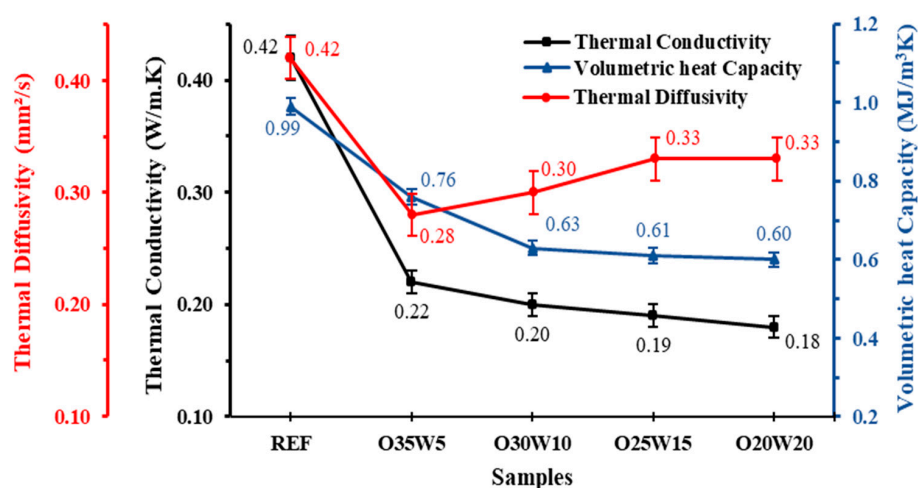

Figure 11. Thermophysical properties of the samples: thermal conductivity, heat capacity, and thermal diffusivity.

According to the findings, gypsum composites have low thermal conductivity values (Figure 11). Compared to the thermal conductivity of 0.42 ± 0.02 W/m.K measured for the pure reference gypsum (REF), the thermal conductivity decreases from 0.22 ± 0.01 W/m.K for sample O35W5 to 0.18 ± 0.01 W/m.K for sample O20W20, which is less than 50% and 57%, respectively. Therefore, the decrease in thermal conductivity between the less loaded (O35W5) and more loaded (O20W20) wood shavings is not significantly different (a variation of 7%). The presence of OW in the composite significantly reduced the material's density and contributed to the reduction in the thermal properties of the composite due to its low thermal conductivity (0.059 W/m.K).

Other studies have confirmed this, emphasizing the effect of cellulose on reducing the thermal conductivity of certain building materials [12,53]. The tendencies toward decreasing thermal conductivity are clearly observed when increasing the percentage of SW between 5% and 20% at the expense of OW. Corinaldesi et al. [34] showed the remarkably reduced thermal conductivity of concrete due to the addition of wood shavings, and Pedreno-Rojas et al. [9] showed that the thermal conductivity coefficient of gypsum boards is improved via the addition of wood shavings. The fact that a high percentage of wood waste was used makes this improvement even more evident (an improvement of 37.6% over the reference material with 20% wood shaving content).

Consequently, the findings of this study are consistent with those reported in the literature. In Figure 12, the thermal conductivity value of the studied bio-composite O20W20 is compared with those reported in the literature for other insulation materials based on gypsum and waste. As observed, all compounds containing residues exhibit lower thermal conductivity coefficients compared to those of the non-added material. Specifically, compounds incorporating rice husk and wood waste demonstrate significantly lower thermal conductivity coefficients that are very similar to those of O20W20. On the other hand, the bean- and hemp-based compounds show relatively smaller decreases in their thermal conductivity coefficients, with reductions of 12% and 13%, respectively. In contrast, the O20W20 compound exhibits a substantial decrease of 57% in its thermal conductivity coefficient, indicating comparatively lower energy efficiency compared to that of its reference material. These gypsum-based bio-composites possess low thermal conductivity, although there is some variation in density. Only the O20W20 sample and the gypsum composite containing 60% cellulose fail to exceed the specified threshold for lightweight gypsum (LWG). Notably, the developed gypsum O20W20 demonstrates a remarkable reduction in the thermal conductivity percentage while maintaining a lower density. Furthermore, it is evident that the combination of wood chips and wadding in sample O20W20 resulted in an optimized reduction in thermal conductivity of 57%. This is higher than the reductions achieved when using wood chips alone (40%) or cellulose alone (54%) in conjunction with gypsum. The bio-composite's insulating abilities are likely improved by the addition of wood chips and wadding, which lower thermal conductivity even more. A higher percentage reduction in temperature may be the result of a more effective barrier to heat transfer created by the unique properties and composition of the materials utilized in the O20W20 sample.
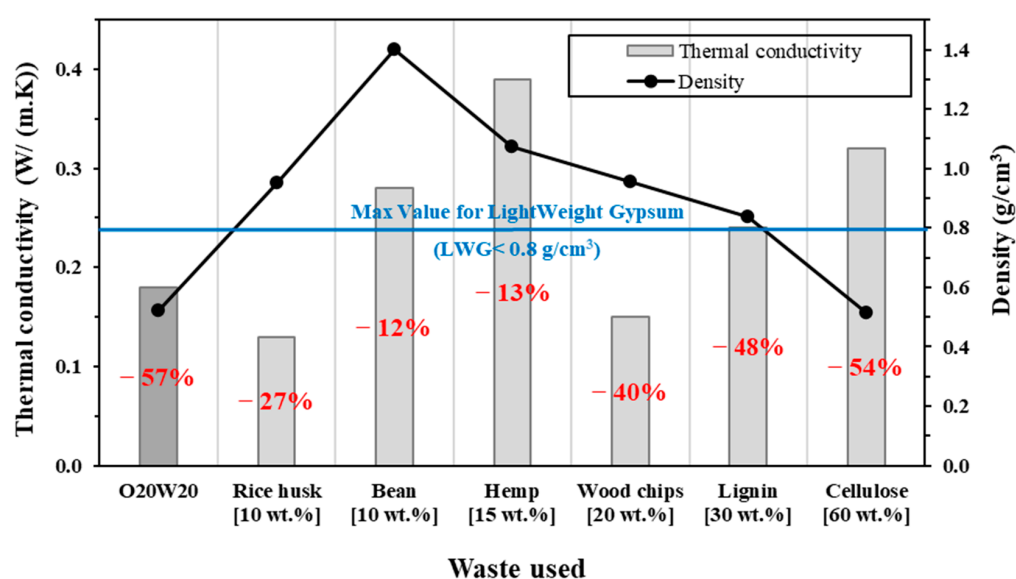

Figure 12. Comparison of O20W20 thermal conductivity with other gypsum composites incorporating waste materials from the literature: rice husk [54], beans [55], hemp [56], wood chips [9], lignin [2], and cellulose [25].

Figure 11 shows that all composites have lower thermal diffusivity values than pure gypsum does (REF = 0.42 ± 0.01 mm^2/s), with values ranging from 0.28 ± 0.01 to 0.33 ± 0.01 mm^2/s. Regardless of the amount of waste added, the thermal diffusivity is far from constant. The efficiency of heat transfer within a material is reflected in its thermal diffusivity, with higher values indicating better heat transfer qualities. However, when compared to pure gypsum, the composites exhibit lower thermal diffusivity values. This suggests that composites containing waste additions or possessing high porosity experience less efficient heat transfer. Materials with high porosity and amorphous structures tend to have lower thermal diffusivity values because heat transfer is hindered by voids and uneven features within the material. In contrast, crystalline materials generally have higher thermal diffusivity due to the facilitated heat transfer provided by their regular and organized atomic structures [57].

Finally, the evolution of the heat capacity of the different gypsum composites is shown in Figure 11. Overall, the addition of wood waste at the expense of ouate leads to a decrease in the volumetric heat capacity of the developed gypsum samples. The range of volumetric heat capacity is between 0.99 ± 0.02 MJ/m^{3}K (REF) and 0.60 ± 0.01 MJ/m^{3}K (O20W20). According to some authors [2,58], the addition of natural fibers leads to a simultaneous decrease in conductivity and heat capacity. As they unequivocally show, the addition of natural fibers not only reduces the density of the elaborated materials but also improves their thermal properties.

Thermal conductivity tests show that adding ouate and wood waste reduces the density of the materials, lowering the thermal conductivity coefficient and ultrasonic pulse velocity of the biobased gypsum. Figure 13a shows an exponential trend with an R^2 value of 1.00, indicating that density and thermal conductivity are strongly related. Several researchers [2,9] have also observed this relationship, supporting the findings of this study and confirming its validity. Figure 13b illustrates the excellent correlation (R^2 = 0.99) between the thermal conductivity and ultrasonic velocity of gypsum samples. This observation was also noted by Hacini et al. [59].

3.4. ANOVA Analysis

3.4.1. Validation of Statistical Models

As outlined in Section 2.3, ANOVA was utilized to examine various physical, mechanical, and thermal properties, including density, water absorption, ultrasonic velocity, flexural strength, compressive strength, and thermal conductivity. The purpose was to demonstrate the significant impact of ouate and wood shaving waste on the gypsum matrix. The obtained sample test results were then utilized to calculate model coefficients through the least squares method, utilizing the NemrodW$^®$ software (Version 9901). The models derived reveal the relationship between the proportions of OW (X_1) and SW (X_2), which are as follows:

$$\text{Density} = 1.0270 - 0.0262 \times X_1 - 0.0242 \times (X_1 \times X_2) \tag{3}$$

$$\text{Water absorption} = 49.0 + 2.0 \times X_1 + 3.7 \times X_2 + 3.0 \times (X_1 \times X_2) \tag{4}$$

$$\text{Ultrasound velocity} = 2400 - 179.2 \times X_1 - 299.2 \times X_2 - 4.7 \times (X_1 \times X_2) \tag{5}$$

$$\text{Flexural strength} = 2.500 - 0.146 \times X_1 - 0.034 \times X_2 - 0.097 \times (X_1 \times X_2) \tag{6}$$

$$\text{Compressive strength} = 4.280 - 0.383 \times X_1 - 0.402 \times X_2 - 0.052 \times (X_1 \times X_2) \tag{7}$$

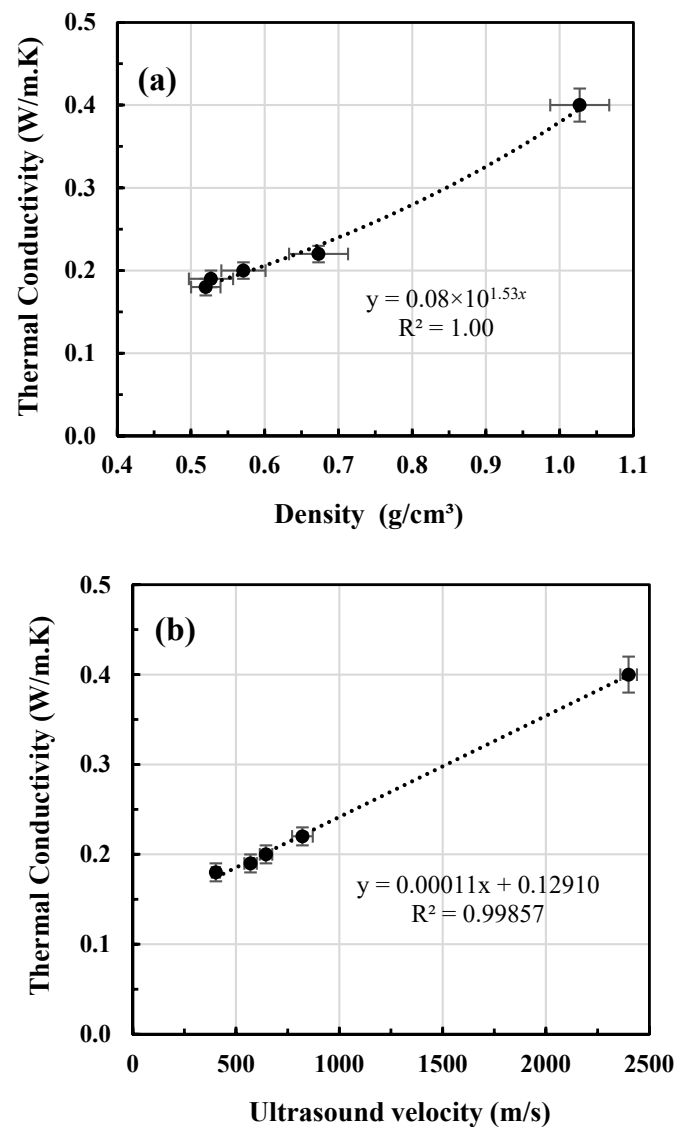$$\text{Thermal conductivity} = 0.420 - 0.023 \times X_1 - 0.029 \times X_2 \tag{8}$$

Figure 13. Thermal conductivity correlation with (**a**) density, and (**b**) ultrasound velocity.

The differences between experimental and predicted values validated the predictive model's accuracy. The coefficient of determination (R^2) and the adjusted coefficient of determination (R^2_{Adj}) are statistical parameters used to assess the reliability and performance of the proposed model. They are commonly employed as indicators of "goodness-of-fit" or measures of variability in regression models. The closer R^2 is to unity with a low standard deviation, the more accurate the predicted response. However, if the number of experiments is equal to the number of unknowns in the system, the R^2 coefficient will always be equal to 1. R^2_{Adj} was introduced to avoid this. R^2_{Adj} is defined as the difference from one of the ratios between the mean square of the deviations of the residuals and the mean square of the experimental deviations.

As shown in Tables 4 and 5, for density ($R^2 = 0.99$ and $R^2_{Adj} = 0.99$), water absorption ($R^2 = 0.99$ and $R^2_{Adj} = 0.98$), and ultrasonic velocity ($R^2 = 0.98$ and $R^2_{Adj} = 0.97$), they were close to one, indicating that the models accurately describe the physical responses. The same for the models for flexural strength ($R^2 = 0.99$ and $R^2_{Adj} = 0.99$), compressive strength ($R^2 = 0.99$ and $R^2_{Adj} = 0.98$), and thermal conductivity ($R^2 = 0.98$ and $R^2_{Adj} = 0.97$) were close to one, suggesting that the models describe the mechanical and thermal responses well.

Table 4. Results ANOVA for density, water absorption, and ultrasound velocity test results.

Source of Variation	SS	Df	MS	F-Value	Signif. (%)	η_p^2
DENSITY: $R^2 = 0.99$; $R^2(\text{Adj}) = 0.99$						
Regression	1.81×10^{-1}	3	6.03×10^{-2}	24,635.2	**	0.99
Residual	2.45×10^{-6}	1	2.45×10^{-6}			
Total	1.81×10^{-1}	4				
WATER ABSORPTION: $R^2 = 0.99$; $R^2(\text{Adj}) = 0.98$						
Regression	3.28×10^3	3	1.09×10^3	3803.8	*	0.99
Residual	2.88×10^{-1}	1	2.88×10^{-1}			
Total	3.28×10^3	4				
ULTRASOUND VELOCITY: $R^2 = 0.99$; $R^2(\text{Adj}) = 0.99$						
Regression	2.66×10^6	3	8.87×10^5	18,462.9	**	0.99
Residual	4.80×10^1	1	4.80×10^1			
Total	2.66×10^6	14				

*: 95% of significate level; **: 99% of significate level; SS: sum of squares; Df: degree of freedom; MS: mean square; η_p^2: partial eta squared.

Table 5. Results of ANOVA for flexural strength, compressive strength, and thermal conductivity test results.

Source of Variation	SS	Df	MS	F-Value	Signif. (%)	η_p^2
FLEXURAL STRENGTH: $R^2 = 1.00$; $R^2(\text{Adj}) = 0.99$						
Regression	3.75	3	1.25	866.2	*	0.99
Residual	0.0014	1	0.0014			
Total	3.75	4				
COMPRESSIVE STRENGTH: $R^2 = 0.99$; $R^2(\text{Adj}) = 0.99$						
Regression	11.52	3	3.84	505.1	*	0.99
Residual	0.0076	1	0.0076			
Total	11.53	4				
THERMAL CONDUCTIVITY: $R^2 = 0.99$; $R^2(\text{Adj}) = 0.99$						
Regression	4.02×10^{-2}	3	1.34×10^{-2}	67,061.7	**	0.99
Residual	2.00×10^{-7}	1	2.00×10^{-7}			
Total	4.02×10^{-2}	14				

*: 95% of significate level; **: 99% of significate level; SS: sum of squares; Df: degree of freedom; MS: mean square; η_p^2: partial eta squared.

The experimental data set was statistically analyzed and the fit of the density, water absorption, ultrasonic velocity, flexural strength, compressive strength, and thermal conductivity models was evaluated using ANOVA, as presented in Tables 4 and 5. The tables display the F-value and significance (*p*-value) at the 95% level of significance. ANOVA serves to assess the statistical significance of means across two or more groups. Furthermore, when the *p*-value falls below the 0.05 threshold, the observed differences are deemed statistically significant. In the context of linear regression, the statistical significance of the regression coefficient indicates the ability of predictor variables to forecast the outcome variable [60]. Our study's ANOVA results indicate that the p-values of the models are significant at 95% confidence levels for water absorption and 99% for density and ultrasonic velocity, demonstrating a strong and significant relationship between the coefficients (X_1 and X_2). The F-values of the model for density, water absorption, and ultrasonic velocity are 24,635.2, 3803.8, and 18,462.9, respectively, as shown in Table 4. For the responses of mechanical and thermal proprieties, ANOVA results indicate that the *p*-values of the models are significant at 99% confidence levels for thermal conductivity and 95% for flexu-

ral and compressive strengths. The F-values for flexural strength, compressive strength, and thermal conductivity are 866.2, 505.1, and 67,061.7, respectively, as shown in Table 5. This result underlines the importance of the models acquired and indicates that the model accurately describes the experimental phenomenon. Furthermore, as Tables 4 and 5 show, incorporating the effect size, represented by the partial square of the eta (ηp^2), into the ANOVA analyses increases the reliability of the results. Incorporating this measure provides a more accurate and illuminating perspective on the influence of the variables, thereby enhancing the reader's ability to understand the results. A partial eta squared result of 0.99 indicates that the independent variables in this analysis explain an unusually high fraction of the variance in the dependent variable. In other words, the specific variables under examination account for nearly 99% of the variability in the results.

$$\text{Partial eta}^2 = SS_{effect} / SS_{effect} + SS_{error} \tag{9}$$

3.4.2. Effects of Wastes on Gypsum Properties

Figures 14 and 15 depict the effect diagrams used for evaluating the key factors. The length of each horizontal bar is equal to the degree of influence of that factor. It also identifies the factor that has a significant positive (+) or negative (−) effect on the response results. When the effect is positive, the bars point to the right, and vice versa when the effect is negative [39].
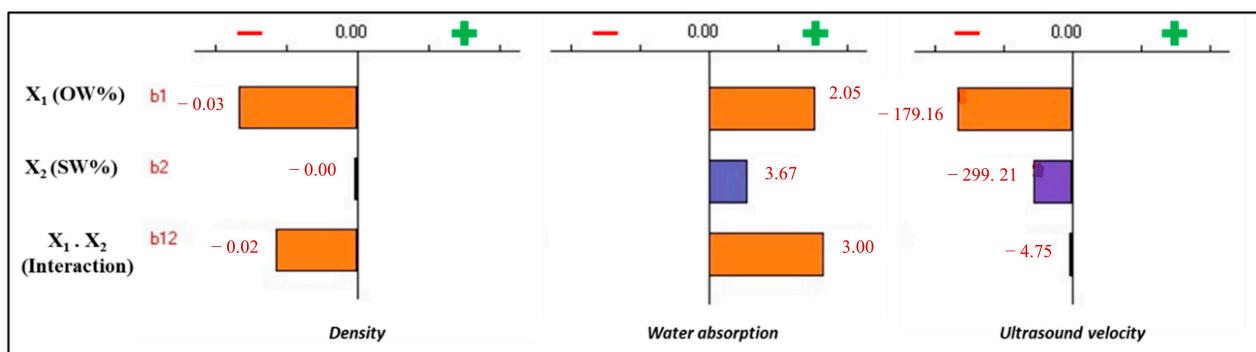

Figure 14. Chart of the factors' effects (OW%, SW%, and the interaction between the two) for density, water absorption, and ultrasound velocity results.
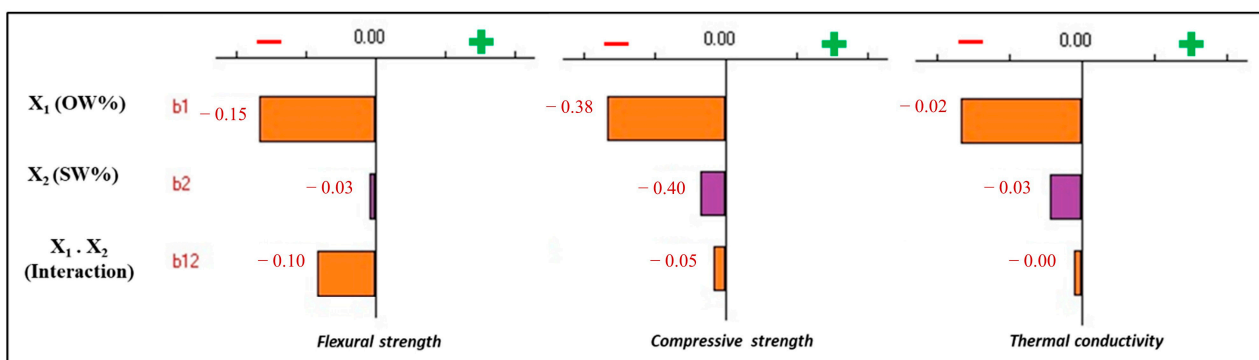

Figure 15. Chart of the factors' effects (OW%, SW%, and the interaction between the two) for flexural strength, compressive strength, and thermal conductivity.

As can be seen in Figure 14, for the density response, the most significant effect corresponds to OW% (X_1), which contributes to the large range bar, while the SW% (X_2) effect is zero, which is why we are removed from the equation model. Furthermore, the negative sign indicates that both factors, through their interaction, decreased the density results. For the water absorption response, the effect chart used identifies that the most major influence is related to OW%, which contributes to the largest height bar, and the

effect of SW% is less. Furthermore, the positive sign implies that both factors, through their interaction, improved the water absorption results. The same observation applies to the response to ultrasonic velocity, where the most considerable influence is related to OW%, which contributes to the largest height bar, and the effect of SW% is less, but the interaction is almost zero. Furthermore, the negative sign implies that both factors decreased the results of ultrasonic velocity.

In summary, the physical properties factor plot (Figure 14) shows that OW% is the key factor in decreasing density and ultrasonic velocity responses, as well as having a positive effect on increasing water absorption. This is explained by the massive presence of OW% compared to that of SW%, which results from the fact that cellulose wadding has a very low density with a hydrophilic nature that helps the material to be light and increase its absorption capacity so that the biobased gypsum becomes porous and absorbs more water, while this high porosity reduces the speed of sound through the material [31].

As can be seen from Figure 15, for the mechanical responses, OW% has the most effect, as it gives the highest bar. In addition, the negative sign indicates that these variables reduced the results of the values of resistance to flexion and compression. Moreover, with the interaction of the two factors, their effects on the resistance to flexion are greater than that on the resistance to compression. For the thermal response, the factor X_1 (OW%) has the most important influence, contributing to the significant range bar; to a lesser extent, the effect of wood appears, while for the interaction ($X_1.X_2$), it is neutral, which is why we are removed from the equation model. In addition, the negative sign indicates that each of the two factors reduced the thermal conductivity results.

In brief, Figure 15, depicting the mechanical and thermal properties' factors, shows that OW% is the most significant contributor to the decline in flexural and compressive strength, as well as thermal conductivity, while it has a positive impact on the thermal insulation property. This can be attributed to the high presence of OW% compared to that of SW%, resulting in more water absorption during gypsum hydration. Additionally, the wood waste, which is like wadding in its composition, primarily consists of cellulose. The addition of more than 5% wood shavings also has an impact on the reduction of mechanical properties as emphasized by several authors [32,48]. On the other hand, it is logical that the addition of these two components increases gypsum's insulation properties by ensuring low thermal conductivity, given that ouate has significantly lower conductivity than that of pure gypsum.

4. Conclusions

In this work, the impact of incorporating two natural waste materials, ouate and wood shavings, on the thermophysical and mechanical properties of gypsum composites was investigated to provide a complete characterization of the developed materials. The main findings of the study are as follows:

- Incorporating 20 wt. % of each waste (ouate and wood shavings) reduces the density of the composites by up to 50%. This reduction in density has a strong correlation with an increase in the water absorption rate and a decrease in ultrasonic toughness. The ultrasonic pulse velocities decrease by 83%, and the water absorption increases by 72%.
- The addition of these wastes significantly reduces the mechanical properties of the composites, both for compressive and flexural strengths. However, the materials meet the requirement for use as interior insulation materials.
- When compared to the reference sample, the thermal properties of bio-based gypsum are significantly improved. Moreover, the thermal conductivity decreases with the increased percentage of wood shavings incorporated into the gypsum matrix. Adding 20 wt. % of ouate and wood shavings to gypsum creates a composite material with improved thermophysical properties, such as a 57% reduction in thermal conductivity.
- The utilization of ANOVA in this study enables us to assess the significance of the impact of the waste used on the observed variations in sample properties, providing a

robust and reliable statistical analysis. Additionally, the proposed models accurately depict the experimental results, as evidenced by the R^2 coefficients approaching one. Consequently, we can estimate the outcomes for other waste percentages without conducting additional experimental work, which is particularly valuable for predictive purposes.

The bio-composites created from gypsum and waste materials have remarkable physical and thermal properties, rendering them well-suited for use as interior cladding on building walls. However, due to the material's elevated water absorption, we advise against employing it in areas with excessively high humidity for ceiling coverings. This recommendation stems from a consideration of the material's performance in such conditions. In short, these bio-composites offer the building sector a more sustainable and environmentally friendly alternative, contributing to a greener approach to construction practices.

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