

Article



Characterization of a Thermal Insulating Material Based on a Wheat Straw and Recycled Paper Cellulose to Be Applied in Buildings by Blowing Method

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Abstract: The thermal envelope is a key component of a building's energy efficiency. Therefore, considerable efforts have been made to develop thermal insulating materials with a better performance than the existing products. However, in the current climate change scenario, these materials must be sustainable, principally during their production stage. In this context, the use of recycled raw materials and agro-industrial waste can be the basis of a material with a low environmental impact and a good thermal performance. In this study, cellulose and wheat straw were characterized. Then, they were mixed in different proportions and densities and the best thermal behavior was selected. The materials were chemically analyzed by TAPPI 2007, thermogravimetric and infrared spectroscopy, together with the measurement of their thermal conductivity with a thermal property analyzer based on the transient line heat source method. The results show that both raw materials are chemically similar to each other. When mixed, they have a thermal conductivity ranging from 0.031 to 0.036 (W/mK), being comparable with several conventional thermal insulators. On the other hand, to achieve the commercial use of this material, an installation through a blowing process has been proposed and proves to be highly promising, achieving a proper density and efficiency in its application.

Keywords: thermal insulation materials; natural fibers; energy efficiency; building sustainability

1. Introduction

Globally, the residential park consumes about 50% of the energy produced [1,2]. Consequently, the current design of energy-efficient buildings focuses on reducing energy consumption in all phases of their lifecycle from construction to demolition [3,4].

In this context, thermal insulating materials are key, since they reduce energy consumption in the operational stage of buildings, providing greater comfort to their users [5–7]. European countries have been working for more than 30 years on the development of thermal insulating materials; however, the market is dominated by synthetic materials from petrochemical or natural sources processed with high amounts of embodied energy and high greenhouse gas emissions, producing negative environmental impacts in their production phase [8–10].

Therefore, the main challenge in recent years has been to develop insulating materials with a low environmental impact to reduce energy losses, to reduce the environmental impacts associated with the use of synthetic materials and to maintain a cost that allows them to enter and compete in the current market [11–14].

In this scenario, local wastes and residues from food industries have a high potential for the development of thermal insulation materials or for housing construction as they have good thermal insulation properties. This allows reducing environmental impacts, as



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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). it gives a second life to waste and in parallel minimizes energy consumption associated with the production of materials and the use of housing [15,16].

In Chile and other countries of the world, agricultural residues are generated in large volumes and are mostly treated by burning, thus generating air pollution problems and degrading the soil [17,18].

On the other hand, the forestry industry, which provides a large amount of material for construction, is looking with great interest at the use of renewable materials from cellulosic waste due to the growing concern about deforestation [19,20].

Many studies have shown that cellulose paper and wheat straw have good thermal insulation properties. Wheat straw has thermal conductivity values between 0.036-0.05 (W/mK), while cellulose paper has thermal conductivity values between 0.038-0.068 (W/mK); in both cases, density is a relevant factor in this variability [7,19–23].

During the last decade, research on natural thermal insulating materials has been carried out and it has been shown that they are comparable with conventional materials [24]. An example of this is the work done by Bakatovich and Gaspar, who investigated and developed low environmental impact thermal insulating materials from residual fibers in panels, achieving thermal conductivity results of 0.044–0.046 (W/mK) at a density of 156–190 (kg/m³) [25].

Other precedents indicate that developments from lignocellulosic fibers are around 0.096 (W/mK) at a density close to 440 (kg/m³), values that are directly proportional [17]. Other authors have reported thermal conductivity values between 0.03–0.1 (W/mK) and that its variability can be attributed to density, heat flow orientation, relative humidity, among others [1,16,26].

In the same line, other authors have worked on composite materials from cellulose paper, minimizing environmental problems since they do not generate secondary pollutants [27]. Other developments based on waste, but from rubber fibers from waste tires, have a variety of applications since this material has low density, high porosity and good thermal conductivity [28].

These antecedents show a great variability in the results, which depend on and have been associated with the manufacturing or production method, arrangement and conformation of the material and, above all, the application method, where the blowing technique has not been analyzed to a great extent.

This work was approached within the Chilean context, where residential energy consumption represents almost a quarter of the country's total energy consumption, 81% of which is for heating, a situation very similar to the global context [29]. This, together with the serious pollution problems in many cities in central and southern Chile caused by the emission of PM_{10} and $PM_{2.5}$ particulate matter, has led to important regulatory efforts to reduce these indicators for both economic and ecological reasons, based on three strategic lines: the regulation of the sale of firewood, the regulation of heating appliances and the regulation of the thermal efficiency of homes [30–32].

All this has kept the interest of researchers in the search, characterization and development of new thermal insulating materials that allow reducing the embedded energy of new buildings through the use of materials with a low environmental impact [11,33].

In summary, empirical evidence shows that studies for the development of construction materials from natural fibers is not a recent practice; however, for decades, natural resource materials have been replaced by industrial materials of high contained energy [34,35].

For this reason, in this work two materials were characterized, paper cellulose and wheat straw, which were mixed to generate a composite material, enhancing the properties of each of them. The material was applied by a blowing technique in different prototypes, and its physical properties were analyzed, identifying the optimal mixture that has the best performance in thermal performance.

2. Materials and Methods

2.1. Materials

The wheat straw was delivered by the company Comasa, located in Lautaro, Chile, while the paper cellulose was delivered by the Chilean company IGLU, who buys it from the company Greentop under the name Termostop.

2.2. Chemical Composition

The preparation of the raw materials to be characterized was carried out in accordance with Annex I of the TAPPI 2007 standard [36]. With these procedures, it was possible to adequately prepare the samples, determine their moisture content, determine their ash percentage, the quantity of non-volatile compounds extractable with organic solvents, the acid-insoluble lignin content, the holocellulose content (Wise, Murphy and D'Adieco method) and the α -cellulose and hemicellulose content (Rowell method). These tests were performed on wheat straw and cellulose samples with a size between 0.45 and 0.25 (mm).

2.3. Thermal Stability

Thermogravimetry (TGA) was used to analyze the thermal stability of the fibers, considering a methodology and configuration similar to other works that analyzed this property in lignocellulosic fibers [17,22]. The measurements were carried out in a TGA/DSC STA6000, Perkin Elmer, USA. The purge gas was set at 40 (mL/min) and the gas used was nitrogen (N₂) at 40 mL/min [37]. For analysis, the sample mass was 10 ± 1 (mg) and the temperature program used was as follows: a heating from 25–120 °C at a heating rate of 50 °C/min; then held 3 min at 120 °C; heating from 120 to 950 °C at 100 °C/min; cooling from 950 to 450 °C at 100 °C/min; gas shift to oxygen at a flow rate of 40 mL/min; heating from 450 °C to 800 °C at 100 °C/min; finally, an isotherm of 3 min at 800 °C.

2.4. Infrared Spectroscopy Analysis

Fourier transform infrared spectroscopy (FTIR) analysis was performed on the raw materials in their natural state. The IR spectra of the samples were recorded using the ATR FTIR equipment, model Cary 630, and will be presented in the frequency range 4000 to 500 (cm⁻¹) as reported by other authors [2,7,34].

The method used was attenuated total reflectance of diamond (ATR), which is the most common sample interface used in infrared spectroscopy, as it is easy to use and provides high quality spectra without sample preparation. ATR can be used to measure solids, liquids or gels, and the diamond ATR element is impervious to scratching and corrosion. All of these spectra were collected using only 32 co-averaged scans at 2 (cm⁻¹) [38]. This surface technique provides a short path and is particularly suitable for experiments such as sample identification, raw material verification, unknown sample identification, among others [39].

2.5. Surface Analysis

For the study of the microscopic structure of the raw materials, a morphological test was carried out. A representative sample of each material was analyzed in a superficial and transversal cut (made with a knife), where the sample was adhered to the sample holder with a double-sided carbon tape.

The morphological visualization of the fibers and the prototypes was performed by scanning electron microscopy (SEM) with a VP-SEM SU 3500 Hitachi-Japan microscope under the following conditions: magnification 40-100-200-500X (surface cut) and 30-100-200-500X (cross-section), BSE detector, 10 KeV, WD~12 (mm), 30 (Pa), according to laboratory specifications, equipment and other works that performed views of lignocellulosic fibers [7,22,32,40].

2.6. Sample Preparation

Different prototypes were developed by modifying two input variables: the weight ratio between the raw materials (paper cellulose/wheat straw) and the density of the

mixture (kg/m³). In a first stage, test specimens were made with PVC pipes of approximately 100 (mm) diameter and 100 (mm) height to measure thermal conductivity, which, by having a known volume (which was accurately measured with a vernier), could determine how much mass of material was needed to reach a defined density. In this first stage of testing, only a 50/50 raw material ratio was used, 50% of the mass of the material being cellulose and the other 50% being wheat straw (which was cut with a Thomas TH-9010V food processor with 400 watts of power).

In a second stage, the test panel to be used for the thermal conductivity measurements was a $29 \times 19 \times 9$ (cm) box made of planed pine and a 9 (mm) plywood bottom, to replicate a space similar to the interior of a wooden partition wall. In addition, in this second phase of tests, due to the results obtained in the experimental full-scale test of the application of the material using the blowing technique, another method was used to prepare the wheat straw to obtain a material that could be easily blown. For this, a biomass grinder was used, consisting of a system of rotating blades mounted inside a metal chassis, which rotates thanks to a 1500 watts electric motor. Then, the material was sieved through a N°12 sieve according to the US Standard Sieve standard, which has an opening of 1.7 mm. The material was injected into the test panels by means of the blowing technique, using an X-Floc M99-DS blowing machine (Figure 1), which pushes the insulating material with pressurized air through a hose into the test panel, reaching densities between 60 and 90 (kg/m³), depending on the strength of the air, the airlock speed and the aperture of the airlock feed gate.



Figure 1. X-Floc M99-DS insulation blowing machine.

2.7. Thermal Conductivity

The conductivity measurement was performed with the KD2 Pro instrument (Figure 2), the same as other studies of fiber-based thermal insulation materials for construction [41–43]. This portable device uses an interchangeable sensor that is inserted into the material to be measured and delivers a thermal conductivity value in a time that can vary between 2 and 10 min. The measuring principle is based on the transient heat source method, which allows rapid analysis of the properties of homogeneous fibers and materials. Thus, the results of processed or chipped fibers vary from those of traditional fibers according to length, diameter, density, moisture content, arrangement (orientation), among others. This analysis is presented in Chapter 4, in the discussion of this work. The device measures in 1 s



intervals during a heating and cooling cycle of 90 s. The KD2 Pro meets the specifications of the IEEE 442-1981 standard and ASTM D5334-0 [44,45].

Figure 2. Thermal conductivity equipment measurement.

2.8. Biological Resistance

To measure the resistance of wheat straw and cellulose to the action of fungi, a durability test was carried out on these raw materials to evaluate their weight loss, exposing them to wood-rot fungi native to Chile in concordance with other works that used fungal treatment in lignocellulosic fibers [46].

The native strains of wood-rot fungi used were Ganoderma lobatum CCCT16.03, a white-rot fungus that is characterized by degrading lignin mainly in lignocellulosic biomass (it can also degrade cellulose and hemicellulose), and Gloeophyllum trabeum CCCT16.04, a brown-rot fungus that degrades mainly hemicellulose and cellulose in lignocellulosic biomass. These fungal strains were previously identified (Hermosilla et al., 2018 [46]) and are available in the Chilean Type Culture Collection at Universidad de La Frontera, Temuco, Chile.

For strain activation, CCCT16.03 and CCCT16.04 fungi were grown on Petri dishes containing a solid culture medium (15 g agar, 3.5 g malt extract and 10 g glucose). The plates were inoculated with an agar mycelium disc (7 mm) and then incubated for 7 days at 30 °C in the dark.

The durability test was carried out in 250 mL Erlenmeyer flasks containing 5 g of chopped wheat straw or cellulose and then inoculated with 5 agar micellar discs of the activated cultures of each fungus. Each flask was plugged with hydrophobic cotton and gauze and incubated in a LABWIT ZXDP-B2120 oven at 25 °C in the dark. To evaluate the effect of humidity in a parallel test (under the same conditions mentioned above), sterile distilled water was added until 30% humidity was reached in relation to the substrate (wheat straw or cellulose). The wheat straw tests were carried out in triplicate and the cellulose tests in duplicate.

To determine the dry weight loss generated by the colonization and growth of the fungi, after 7 and 14 days the substrate was removed, dried at 105 $^{\circ}$ C for 8 h and the dry weight was determined. The percentage of weight loss was determined by the difference between the initial dry weight (calculated using the moisture percentage of the substrate) and the final weight.

2.9. Fire Behavior

To evaluate the fire performance of the mixtures, a methodology based on the UNE 23-725-90 drip test was adapted [47].

To begin with, 11 mixtures of wheat straw and paper cellulose were made, starting with a mixture made only of cellulose and increasing the proportion of wheat straw in the mixture by 10% until reaching a test tube made only with wheat straw. These mixtures were placed in a metal container of approximately 102 (cm³) volume, in which the mixture was placed at a density of between 75 and 80 (kg/m³). Each mixture was subjected to a heat source, in this case, a portable butane gas torch whose flame reaches a temperature of 1200 °C, which was applied for 3 s over the center of the mixture at a constant distance and withdrawn to observe the behavior before the heat source, its ignition and subsequent extinction of the flame. The procedure was repeated for three minutes and the mixture was removed to later record the ignition and extinction times by analyzing a video recorded during the entire test.

The variables evaluated were the average time of each ignition (flame appearance) and the total sum of all ignitions.

3. Results

3.1. Chemical Composition

3.1.1. Paper Cellulose

The results of the chemical analysis of cellulose presented in Table 1 show that it is mainly composed of α -cellulose (49.07 ± 2.84%) and lignin (22.38 ± 0.30%). The high percentage of lignin shows that the cellulose used for the analysis comes from newspaper scraps, which are characterized by having a lignin percentage close to 22%, unlike office paper, which presents lignin percentages close to 1% [48].

Component (%)	Sample			Auorago
	S1	S2	S 3	– Avelage
Moisture	8.36	9.06	9.01	$8.81 \pm 0.17\%$
Ashes	15.16	15.27	15.11	$15.18\pm0.08\%$
Removable compounds	25.51	25.71	25.49	$25.57\pm0.12\%$
Lignin	22.70	22.32	22.11	$22.38\pm0.30\%$
Holocellulose	65.73	67.79	65.83	$66.45 \pm 1.16\%$
α-Cellulose	49.28	51.80	46.14	$49.07\pm2.84\%$
Hemicellulose	16.45	15.99	19.68	$17.37 \pm 2.01\%$

Table 1. Paper cellulose chemical composition.

3.1.2. Wheat Straw

The analysis of the chemical composition of wheat straw presented in Table 2 shows that the fiber is mainly composed of α -cellulose (34.17 \pm 0.59%) and hemicellulose (35.18 \pm 0.39%) in similar percentages and lignin (18.77 \pm 0.16%) in lower percentages.

Table 2. Wheat straw chemical composition.

Component (%)	Sample			A
	S 1	S2	S 3	– Average
Moisture	9.33	9.06	9.03	$9.14\pm0.17\%$
Ashes	4.32	4.45	4.34	$4.37\pm0.07\%$
Removable compounds	11.47	11.71	11.75	$11.64\pm0.15\%$
Lignin	18.59	18.90	18.81	$18.77 \pm 0.16\%$
Holocellulose	69.57	69.04	69.43	$69.35 \pm 0.27\%$
α-Cellulose	34.84	33.71	33.96	$34.17 \pm 0.59\%$
Hemicellulose	34.73	35.33	35.47	$35.18\pm0.39\%$

3.2. *Thermal Stability*

Figure 3 shows the result of the thermogravimetric analysis of cellulose and wheat straw.



Figure 3. Thermal stability of paper cellulose and wheat straw.

The diagram shows that in the paper cellulose test up to 246.79 °C there is a mass loss close to 12%, corresponding to the moisture present in the sample, then from 246.79 °C there is a mass loss of 63.92%, which is due to the elimination of volatile compounds formed by the pyrolytic decomposition of hemicelluloses, cellulose and lignin. The gas is then exchanged for oxygen, and from 463.53 °C there is a 17.54% mass loss, associated with lignin losses. The remaining weight at the end of the test corresponds to mineral compounds present in the sample, which mainly correspond to boron salts present in the cellulose used for the blowing.

These results are consistent with the results obtained in the chemical characterization using the TAPPI standard and show that the paper cellulose is thermally stable up to approximately 262 °C; at temperatures higher than this it begins to lose mass, corresponding to the cellulose that is part of its chemical composition.

In the wheat straw case, the graph shows that at 174.36 °C the sample loses about 10% of mass, corresponding to moisture, and then has a mass loss of 72.87% from 174.36 °C, corresponding to volatile compounds in the sample. Next, the gas change is made to oxygen, and from 474.41 °C there is another mass loss of 16.61%, corresponding to lignin. The remaining weight corresponds to the ashes of the sample.

These results confirm the chemical characterization using the TAPPI standard and show that wheat straw is thermally stable approximately just to $174 \,^{\circ}$ C.

These values of thermal stability are slightly lower than other insulation composites with lignocellulosic fibers in their composition [49–51]. However, the results suggests that these materials can be used as a building material, since the stability temperature of 170–260 $^{\circ}$ C is enough for building applications at low and medium temperatures [52].

3.3. Infrared Spectroscopy Analysis

According to the results of the infrared spectroscopy analysis shown in Figure 4, both raw materials have similar infrared spectra, just with magnitude differences in some absorption bands.



Figure 4. FTIR spectra of paper cellulose and wheat straw.

At approximately 3290 (cm⁻¹), an important absorption band is observed, corresponding to O-H bonds and belonging to the hydrogen bridges between the hydroxyl groups of the glucose chains that compose the cellulose, as well as belonging to the remains of water that could have been in both samples. At 2900, 1420 and 1370 (cm⁻¹), vibration bands of C-H bonds associated with aliphatic organic compounds are observed. Around 2080 (cm⁻¹), C=O stretches of carbonyl groups present in hemicellulose are observed, and approximately between 1640 (cm⁻¹) and 1760 (cm⁻¹), vibrations of C-C groups present in lignin are present; however, in the wheat straw sample these groups are much more noticeable than in the FTIR analysis of cellulose. Finally, the last group identified corresponds to C-O identified around 1030 (cm⁻¹), related to the stretching of the acetyl functional group, which is much stronger in wheat straw compared with the FTIR analysis of cellulose. These results are similar to other FTIR analyses of lignocellulosic fibers for building applications reported in the literature [2,32,49,53], that show the presence of similar functional groups, indicating that the raw material has no toxic components.

3.4. Surface Analysis

3.4.1. Paper Cellulose

Figure 5 shows four microscopic images of the paper cellulose. The fibrous structure of the cellulose can be observed in image (a), accompanied by agglomerations of material. These elongated fibers are the ones that probably give cellulose its thermal insulating capacity. In images (b) and (c), in addition to observing in greater detail the elongated fibers of the cellulose, small white clusters begin to be observed, which are particles of boron salts that are present in the analyzed cellulose and that give it the ability to retard the action of fire and to repel insects, among others. In image (d), the particles of boron salts are clearly observed, which are homogeneously mixed throughout the material. This fibrous macrostructure is similar to other natural fibers used for insulation applications reported in other works, such as pine fiber [51], palm surface fiber [49] or pisum sativum [2].



Figure 5. Microscopic morphology of paper cellulose. (a) View at \times 30 magnification, (b,c) views at \times 100 magnification, and (d) view at \times 500 magnification.

3.4.2. Wheat Straw

Figure 6 shows four microscopic images of wheat straw. Image (a) shows the surface of the wheat straw fiber and it can be seen that it has a rigid and well-defined structure, which can also be seen in image (b), where the tubular structure of the fiber can also be seen on the outside. Images (b), (c) and (d) show the porous structure of the wheat straw fiber inside. This porous structure helps to enhance the thermal insulation of wheat straw, giving it good properties for insulation material applications [54], because, in general, materials with closed pores, such as can be seen in wheat straw, have a lower thermal conductivity than those with large and open pores [52].



Figure 6. Microscopic morphology of wheat straw. (a) Longitudinal view at \times 500 magnification, (b) cross-sectional view at \times 30 magnification, (c) cross-sectional view at \times 100 magnification and (d) cross-sectional view at \times 200 magnification.

3.5. Thermal Conductivity

The thermal conductivity measurement initially corresponds to a mixture composed of 50% paper cellulose and 50% wheat straw.

Figure 7 shows the material blown into a full-scale panel, where a homogeneous mixture covering the entire empty space of the panel is observed. In these samples, thermal conductivity was also measured, which shows negligible variations with respect to the material in the test cylinders.



Figure 7. Mix of blown material in a test panel.

Figure 8 shows the results of the measurement at different density levels, where it can be observed that the mixture has a thermal conductivity lower than 0.041 (W/mK), which demonstrates its potential as a thermal insulating material, since this value is comparable with conventional insulating materials in the current market [1,11,16,20,26]. Furthermore, it is observed that the thermal conductivity of the mixture increases as the density increases, similar to other lignocellulosic fiber materials but different to other conventional materials that decrease their thermal conductivity as they have a higher density.



Figure 8. Thermal conductivity of wheat straw and paper cellulose mixture in proportion 50/50.

According to these results, and considering that cellulose is usually blown into vertical elements at densities between 50–90 (kg/m³) [48], it was decided to restrict the density variable between 60–90 (kg/m³) and to work with mechanically processed wheat straw for the second stage of tests, thus obtaining the thermal conductivities shown in Figure 9.

These results show that, when mixing wheat straw (processed) with cellulose, the thermal conductivity achieves at most 0.036 (W/mK), regardless of the density of the mixture, a value that is well below the 0.065 (W/mK) maximum recommended value for thermal insulation [55]. Furthermore, it is observed that as in the first tests, in all

compositions the thermal conductivity increases as the density of the mixture increases, which is a typical behavior observed in the thermal insulating materials composed of natural fibers, where the lower the density the lower the thermal conductivity and, therefore, the better the thermal behavior [55].



Figure 9. Thermal conductivity of wheat straw and cellulose mixtures.

3.6. Biological Resistance

In the wheat straw and cellulose durability test without moisture modification, no fungal colonization was observed after 14 days of incubation. It was also not possible to measure changes in the dry weight of the substrates during this period. On the other hand, a rapid desiccation of the inoculant was observed. This is due to the fact that fungi need high humidity to develop and most of the wheat straw degradation work is carried out at humidity levels close to 50%.

To observe the behavior of the fungi under more favorable conditions, the percentage of humidity was increased to 30% in relation to the weight of the substrate, considering the moisture content of each substrate. In the wheat straw durability test at 30% humidity, colonization by both fungi was observed at 4–5 days. At 7 days, a dry weight loss of about 13% was observed for both fungi. In cellulose, less colonization was observed than in wheat straw, which was also reflected in a weight loss of 3.8% for CCCT16.03 and 4.3% for CCCT16.04 (Figure 10).



Figure 10. Dry weight loss produced by G. lobatum CCCT16.03 and G. trabeum CCCT16.04 on cellulose and wheat straw at 30% moisture content after 7 days of incubation.

3.7. Fire Behavior

The results of the fire performance test (Figure 11) show that, when wheat straw is added to the paper cellulose, the fire resistance is diminished; however, this resistance is drastically affected with proportions of straw equal to or greater than 70% of the total mixture. This result shows that just cellulose fiber adds fire resistance to the mixture, which can be explained by the boron salts being added to the paper cellulose for a better fire behavior [48], unlike the wheat straw that has no additions for its fire behavior and has a thermal stability much lower than paper cellulose, according to the TGA results. The fire performance mixture shows that is important for the fire behavior of the material to maintain a percentage of paper cellulose in the mixture over 30% to keep the flammability of the material within acceptable levels.



Figure 11. Fire performance of cellulose and wheat straw mixtures.

4. Discussion

The chemical composition of local wheat straw and paper cellulose indicates a very similar composition to materials from other local works with lignocellulosic materials and also from other countries [2,7,22,48]. In this context, the high presence of cellulose and hemicellulose in cellulose paper is striking, but this is due to the fact that it comes from paper recycling with the addition of borax salts in order to generate a material with fireproof properties, which is also favorable for the composite material.

The TGA results show a high thermal stability in both fibers, especially the paper cellulose. However, although the thermal insulation materials used for building insulation generally are not exposed to high temperatures or direct sun exposure, it is still an important factor for the comparison with traditional insulation materials and the consideration of the use of some processing techniques that require heat application.

The FTIR analysis shows the presence of similar functional groups in both raw materials, which suggests that paper cellulose and wheat straw can be mixed without expecting some chemical reaction. In addition, the FTIR analysis does not find functional groups that can suggest the presence of toxic components, something especially important in a material that is looking to become a more sustainable option than traditional insulation materials.

The microscopic images of the raw materials analyzed show that the paper cellulose has a structure similar to some natural wools [53] and biomass waste [49] used for insulation applications. On the other hand, wheat straw has a cell-type internal structure and a thick stem wall, which has a positive impact on its thermal properties, such as polyurethane foam, an insulation material highly used in construction [56]. Both microscopic structures

had the capacity to generate little air spaces, which improved the thermal capacities of these raw materials, allowing their applications for building insulation.

The findings of this work show average thermal conductivity results of 0.036 (W/mK) at a density of 80 (kg/m³) for a mixture of wheat straw and cellulose paper at a ratio of 50–50%. These results are favorable with respect to the properties possessed by conventional materials and the results reported by other authors in similar lines of research. For example, Bakatovich and Gaspar report results of 0.045 (W/mK) and Si Zou et al., report 0.096 (W/mk) [17,25].

However, other authors report a variability of the thermal conductivity of materials; for example, humidity is an essential factor, and its increase is directly proportional to the thermal conductivity. Yapin Zhou et al., reported results between 0.03–0.1 (W/mK), while other authors have reported results for lignocellulosic materials of 0.033 and 0.065 and 0.07 and 0.08 (W/mK) at different densities, achieving in some cases values over 400 (kg/m³) [1,16,20,21,26,57]. It is important to note that the research mentioned above generates materials in a rigid or panel format, unlike the proposal, which uses a much less commonly used method: the blowing method.

This indicates that moisture, density, fiber arrangement and application are major factors for lignocellulosic thermal insulators. This is why the good results presented in this work are associated with the quality of both raw materials: the porosity of the wheat straw, which can be seen in Figure 6, and the fibrous structure of the cellulose paper, which can be seen in Figure 5. This favors the thermal conductivity property and, in addition, thanks to its application, since the blowing technique makes it possible to add the material in a partition and to generate air capsules, thanks to the structure of the aforementioned materials.

The durability tests of wheat straw and cellulose paper provide very interesting information regarding the proliferation of pathogens, which do not generate any colonization, and mass losses only occurred when humidity increased to 30%.

This analysis was carried out with the main fungi that proliferate in wood in southern Chile; therefore, it is suggested that for future research this aspect could be expanded and deepened. On the other hand, the researchers agree with other research, where it is suggested to integrate a methodology to analyze the durability of bio-based materials to ensure their good behavior in buildings, considering that the developments, for example, of corn cob are very advanced and its application as a thermal insulating material is evident [58,59].

The fire behavior of the material indicates that, in order to obtain self-ignition of the flame, at least 30% of paper cellulose must be added to the mixture, because wheat straw has no fire treatment, while paper cellulose does. These results, under a standardized methodology, allow the material to be compared with other research, where it is important to highlight that the fire resistance of thermal insulation materials is not key, as, in general, it is the interior linings that provide the capacity to resist fire in the event of an incident. It is important for such materials to be self-extinguishing and not to generate toxic gases, which is why conventional materials mostly generate problems when combusting [56,60].

Research by Elżbieta Janowska-Renkas et al., analyzing a material based on wheat straw, indicates that, due to a high density of fiber application, over $100 (kg/m^3)$ allows the material to be non-combustible; however, the increase in density generates the thermal conductivity that also increases, reaching almost 0.07 (W/mK), which makes it less competitive with respect to other developments [16].

5. Conclusions

The mixture with the best thermal performance, according to the results, is that of 40% paper cellulose and 60% wheat straw. In this context, after a thorough review of the behavior of lignocellulosic materials, it is pointed out that the application density of the material, the moisture content and the application method are fundamental. For this reason, the conductivity result of around 0.036 (W/mK) is very favorable and is attributed to the

porosity of the wheat straw plus the fibrous composition of the cellulose paper, which can be confirmed by the morphology analysis carried out in this research.

It is important to note that the blowing method allows the injection of a material without the need to remove the interior or exterior cladding of a building element and generates air chambers (variable depending on the type of fiber) that provide benefits in terms of thermal performance.

In addition, the mechanical defibration method used demonstrates that it can be carried out on a large scale, since there are only simple processes that do not require complex machinery, such as chopping and screening the biomass. Moreover, there is no need to add processes involving the use of chemicals or additives or processes that increase the environmental impact. In this last aspect, future research will be to analyze through a lifecycle analysis the impacts associated with the development and the application of this material.

Thermal stability provided interesting results from the point of view of the application of the thermal insulation material, indicating that wheat straw fiber is stable up to 170 °C (approximately) and cellulose paper above 250 °C. The results are favorable, as these types of materials are not directly exposed to the indoor and outdoor environment. However, the authors point out the importance of further analysis for the material mixture in the ratio 60–40%, wheat straw plus cellulose paper.

Regarding the fire behavior and the biological resistance of the material, the results were favorable. This is attributed to the fact that the recycled cellulose paper has incorporated retardant additives that provide resistance to fire and microorganisms; thus, it is very interesting to be able to analyze these parameters with cellulose paper without this treatment.

Finally, based on these promising results, an economic analysis of the material will be carried out in the next steps, with the purpose of advancing the commercialization of these types of materials under the concept of a circular economy and sustainable construction.

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