Potential of the Residual Fibers of Pisum Sativum (PS), for use in a Development of a Thermal Insulator Material

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Abstract. In the context of climate change, a special interest has been aroused by thermal insulator materials from natural fibers, which can promote sustainable construction in the sector. The objective of this research was to evaluate the potential of the residual fibers of Pisum Sativum (PS), for their future use in the development of a thermal insulator material with low environmental impact. The physical, chemical and thermal properties of the fiber were determined. The results show that the residual fiber has a great potential with an extraction of raw material of 11.6%. It shows a good proportion in the length of its fibers and roughness and significant number of grooves, which is verified using a scanning electron microscope and after thermal conductivity tests that indicate a best value of 0.033 W/mK. It is therefore concluded that from the residual fibers of PS, an opportunity is generated for the food residual fibers within the sustainable construction industry, given the proposed innovation and its interesting results, which could contribute with its low environmental impact throughout the life cycle of buildings.

1. Introduction

The buildings need high quantities of energy in its whole life cycle: construction, operation and demolition [1]. Several analysis about energy consumption in the life cycle of buildings indicates that between the 10 and 20% of the energy is destined to the fabrication of all the materials used in the construction, while in the operational phase its used around 80-90% of the total energy [2], [3]. The construction industry have a direct and significate influence into de consumption of natural resources and emissions of greenhouse gases [4]. At world scale, it is estimated that buildings consume between a 30 and 40% of primary energy and they are responsible between 40 and 50% of the emissions of greenhouse gases [2]. In Chile, the commercial, public and residential sectors are responsible of the 22% of total energy consumption of the country. For this reason, during the 2013, the Ministerio de Vivienda y Urbanismo (MINVU) develop the first version of “Sustainable Construction National Strategy” with the purpose of guide and impulse the integration of the sustainable standards in the construction area in Chile. Actually its evident that the thermal insulation materials are essential in the buildings because they allow to reduce the energy losses by transmission [5]. However, the thermal insulation of buildings its constructed mainly with synthetic materials coming from petrochemicals products or from natural sources processed with high energy consumption [6].
In this way, we have begun to study alternatives for the development of natural thermal insulation materials that allow to reduce the energy losses and reduce environmental impacts associated to the fabrication of synthetic materials, reaching a more sustainable buildings development, what has drawn attention of a lot of entities and companies [7]–[10].

In the last years, various researches about natural materials have been carried out and it has been managed to verify that they are comparable with conventional materials in terms of insulation capacity (Korjenic et al., 2011). Only one example among many others is the work made by Aliaksandr Bakatovich y Florindo Gaspar [11], who researched and developed thermal insulation materials with low environmental impact from residual fibers reaching results of thermal conductivity of 0.044 – 0.046 [W/(mK)] in a density of 156 – 190 [kg/m3]. In this context, the residual and waste from alimentary industry have a high potential for the development of thermal insulation materials because they have good thermal insulation properties and they are also generated in large volumes [12], [13]. In specific, in Chile the crop of Pisum Sativum (PS) was of 1281 ha in 2015/2016 with a tendency to increase every year [14]. In this context, the target of this work was study the natural fibers from the alimentary sector of PS to review the potential use of the fiber like a thermal insulation material, considering the extraction process of the fiber and the physic and chemical characterization.

2. Materials and methods

2.1. Materials
The residual fiber characterized in this study was obtained from the pods of pisum sativum, commonly called pea, acquired at the Feria Pinto located in Temuco, Region of the Araucania in southern Chile. The PS pods was cleaned with potable water and the fibers in disrepair were discarded. The PS fiber was introduced in a juice extractor Omega 8226 to separate and crush the fiber. The pods were dried for 24 hours at 60°C. These pods were on average of 3.2 ± 9.9 cm in length and 1.0 ± 2.7 in width.

![Sample type 1 and Sample type 2](image_url)

**Figure 1.** a) Sample type 1. b) Sample type 2.

2.2. Chemical composition
The chemical analysis of the fiber was performed according to TAPPI procedures. The sample preparation for further analysis was done following the TAPPI 257 cm-85 procedure; water content determined by using TAPPI T264 cm-97; ashes content following TAPPI T211 om-93 and lignin by TAPPI method T222 om-98. The holocellulose content of the samples was determined briefly with 2.5 ± 0.1 g of samples extractive and moisture free samples, were added 80 mL of hot water at 70-80°C and heated in a water bath at 70°C, shaking periodically. After 60 min, for a period of 6-8 h, 0.5 mL of acetic acid and 2.6 mL of sodium chlorite (25%) were added. After the additions, the sample was maintained for 12 further hours without any addition. Finally, the residue was filtered and dried at 105°C and weighed. Cellulose and hemicellulose were determined following the Rowell metod.
2.0 ± 0.1 g of sample obtained from the holocellulose determination was mixed with 10 mL of NaOH (17.5%) by shaking until dispersed particles appeared. Afterward, there were three more additions of 5 mL of NaOH (17.5%) every 5 minutes; the sample was maintained for 1 h and finally filtrated. The final sample was dried for 24 hours at 105 °C. Cellulose was calculated as a percentage of sample before treatment with NaOH and hemicellulose as the difference between holocellulose and cellulose.

2.3. Density
To determine the average density from the PS samples, five samples were made with a mold with measurements of 20 x 5 x 1 cm and the weight of each sample was registered with a digital balance. The fiber was in wet state when they were introduced and compacted manually in the mold and then dried at 60°C in oven. Then, the length, width and height of each sample was measured. Finally, the density was calculated using the equation “density = mass / volume”, obtaining the value of the average of five samples.

2.4. Thermal stability
The thermal stability of each sample was determined by measuring the global mass loss at different temperatures with a thermogravimetric analyzer (TGA) model STA 6000 from Perkin Elmer Co. Measurements were performed under nitrogen atmosphere. Temperature changes were controlled in the range between 20 and 600°C at a heating rate of 10°C/min for 60 min. The degradation temperature and maximum degradation rate were also calculated.

Figure 2. TGA and DTGA curve of PS

2.5. Surface analysis
A variable pressure scanning electron microscope (VP-SEM) Hitachi SU 3500, was used to examine the microstructure of the PS in different sections. A backscattered electron detector (BSE) in compositional mode was used to analyze the surface of each sample.

2.6. Infrared spectroscopy analysis
Fourier transform infrared spectroscopy (FTIR) analysis of each sample was performed. For this analysis, samples were loaded into the sample holder of a Cary 630 FTIR spectrometer. Resolution
of the spectrophotometer was set at 4 cm⁻¹ and the analysis was made in a range between 4000 – 6000 cm⁻¹ spectral region.

2.7. Thermal conductivity

The thermal conductivity of PS samples was determined by using the transient line heat source method by ASTM D5334-14. The equipment used was the KD2Pro device manufactured by Decagon Devices, Inc. For this test were measured two types of samples as shown in fig.1. In the sample type 1 the sample weight was measured and introduced in to a PVC container, the sample was compacted manually and then measured. Due to heterogeneity of the sample, it was measured in several ways with the KS-1 sensor. Three sample was fabricated and each one was measured three times. The sample type 2 was developed in to a wood mold, where was introduced the wet sample, to adoptate the mold form and then were dried for 24 hours at 60°C. The mold had measurements of 20 x 5 x 2 cm. Five sample was fabricated and each one was measured three times with the KS-1 sensor. A 10 min gap between analyses was used to ensure the stabilization of the sensor.

3. Results

3.1. Chemical composition

The chemical composition of the PS fiber is presented in table 1. Chemical analysis showed that PS is constituted by similar amounts of α-cellulose (28.77 ± 1.24%) and hemicellulose (28.17 ± 0.35%), followed by lignin (17.33 ± 0.21%), removable compounds (9.21 ± 0.45%) and finally ashes (3.12 ± 0.16%). The equilibrium humidity was 10.13 ± 0.54%.

| Components (%)       | M₁   | M₂   | M₃   | Average        |
|----------------------|------|------|------|----------------|
| Humidity             | 10.69| 9.67 | 10.02| 10.13 ± 0.54   |
| Ashes                | 3.29 | 3.28 | 2.8  | 3.12 ± 0.16    |
| Removable compounds  | 8.88 | 9.75 | 8.99 | 9.21 ± 0.45    |
| Lignin               | 17.17| 17.12| 17.7 | 17.33 ± 0.21   |
| Holocellulose        | 57.61| 56.66| 56.56| 56.94 ± 0.67   |
| α-Cellulose          | 29.59| 28.02| 28.71| 28.77 ± 1.24   |
| Hemicelulose         | 28.02| 28.64| 27.85| 28.17 ± 0.35   |

3.2. Density

Density results of PS was calculated based on five prototypes. The density was determined recording the mass and volume data in the five prototypes. Table 2 shows the measured data. The density results show values in the range of the lignocellulosic materials. While this density is higher than typical insulating materials, it may be an opportunity in terms of thermal inertia.

| Nº samples | Volume (cm³) | Mass (g) | Density (g/cm³) | Density (Kg/m³) |
|------------|--------------|----------|-----------------|-----------------|
| 1          | 100          | 9.97     | 0.10            | 99.70           |
| 2          | 101.5        | 10.32    | 0.10            | 101.37          |
| 3          | 101          | 10.89    | 0.10            | 107.82          |
3.3. Thermal stability
The results of thermogravimetric analysis of PS samples are presented in figure 2, which graphically illustrates the mass loss of the PS. The test temperature fluctuates between 25°C and 100°C, at a heating rate of 10°C/min. It is observed that in the range from 25°C to 125°C takes place the first mass loss of 9.31% associated to the evaporation of the water present in the sample. The start of fiber degradation takes place after the 200°C. The curve shows the big mass loss of approximately a 70% between 210°C and 450°C, in this band take place a series of thermal decompositions which starts with the hemicellulose and continues with degradation of the α-cellulose of the fiber. Another significative loss of a 17.38% generates in a approximate temperature of 519°C. The results obtained shows that the PS pod in natural state have a low thermal stability over the 200°C.

The proximal analysis results plotted in the figure 3, shows the reduction of equilibrium humidity in the sample, this is attributed to the mass loss in the first section between the 25°C and 120°C. The volatile material content is shows between the 200°C and 500°C, corresponding to a 70.98%. After the ambient change to oxygen, it shows losses of fixed carbon of 17.38% and the ashes content was a 2.03%.

![Figure 3. Proximal analysis curve of PS](image)

In table 3 there’s a summary of the proximal analysis results realized to the PD pods in natural state, dried for 25 hours at 60°C.
Table 3. Composition based on proximal analysis results of PS pods

| Humidity (%) | Volatile material (%) | Fixed carbón (%) | Ashes (%) |
|--------------|-----------------------|------------------|-----------|
| 9.31         | 70.98                 | 17.38            | 2.03      |

3.4. Surface analysis
The analysis realized to the PS pods in natural state detected the presence of microorganisms associated to an early decomposition of the fiber (figure 4 c). The images corresponding to the external surface of the fiber, the figure 4 shows a huge amount of intertwined fiber. In the figure 4 c it can be seen cellular structures with little holes of honeycombs in a side of the fiber. In the figure 4 d it can be seen the difference between length and thickness of the PS.

Figure 4. Longitudinal outside SEM images of PS

3.5. Infrared spectroscopy
The infrared spectrum of the PS sample plotted in the figure 5, shows a wide and high band between the 3400 and 3200 cm\(^{-1}\), this reveals a O-H link, related to the presence of water. The peak 2917 cm\(^{-1}\) is related to the stretching vibration of the C-H link. The vibration of the group in 2115 cm\(^{-1}\) is associated to a C=C link, an acetylene compound. In the absorption band at 1700 cm\(^{-1}\) there is a reference to a stretching of a C=O double link, attributable to carboxyl groups presents in hemicellulose, like esters and acids. The flexion vibration band at 1239 cm\(^{-1}\) reveals a possible O-H link, aromatic ethers. The band 1027 cm\(^{-1}\) corresponds to associated vibrations characteristic of C-OH and C-OR, of alcohols and ethers.
3.6. Thermal conductivity

The tables 4 and 5 present the results of thermal conductivity of both PS samples types. According to the thermal conductivities measured, it is feasible the use of PS residual fibers like a material for the fabrication of a thermal insulation material in a granular or block format. The difference between the results is related to the type of test tube that was used, with the purpose of representing the heterogeneity of the material and different options of future use.

| Sample | TC 1 (W/mK) | TC 2 (W/mK) | TC 3 (W/mK) | Average (W/mK) |
|--------|-------------|-------------|-------------|----------------|
| PS-1   | 0.041       | 0.042       | 0.040       | 0.041          |
| PS-2   | 0.039       | 0.041       | 0.037       | 0.039          |
| PS-3   | 0.039       | 0.038       | 0.040       | 0.039          |
| Average (W/mK) |           |             |             | 0.040          |

| Sample | TC 1 (W/mK) | TC 2 (W/mK) | TC 3 (W/mK) | Average (W/mK) |
|--------|-------------|-------------|-------------|----------------|
| PS-1   | 0.033       | 0.030       | 0.037       | 0.033          |
| PS-2   | 0.036       | 0.037       | 0.036       | 0.036          |
| PS-3   | 0.031       | 0.030       | 0.034       | 0.032          |
| PS-4   | 0.035       | 0.033       | 0.031       | 0.033          |
4. Conclusions
The PS fibers have a low thermal conductivity, around 0.040 - 0.033 W/mK, this value represents an excellent thermal conductivity for a natural insulation material. If this value is compared with the expanded polystyrene value, is a 9% lower. This result can be explained by the microstructure of the fibers, which presents small cells, which generates tight air spaces; and have a good length/thickness proportion that generates a slower heat transfer trough the fiber. The thermal analysis of PS pods confirms its stability up to the 200°C, that is a positive result for future applications in construction area. The future researches will focus on its biological stability and its fire behavior.

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