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Valorización de residuos textiles para producir estructuras porosas sostenibles con potencial como aislantes térmicos

Beatriz Magalhães[(*)], Ashly Rocha[(**)], Ligia Rodrigues[(***)] y Carla Silva[(****)]

Abstract: Every European generates about 15 kg of textile waste per year, with around 65% of this waste still ending up in landfills, despite the mandatory selective collection of textile waste from January 1st, 2025. Currently, mechanical recycling is the most common method of textile recycling. This recycling process has several shortcomings (for example, low productivity, difficulty in separating by type of fibre, difficulty in recycling clothing due to the need to remove accessories) and usually generates products of low commercial value (downcycling). In this work, a porous textile structure of aerogel-type was developed using mechanically recycled cotton (CO) from textile waste, tailored to obtain a material with insulating properties. During this process, recycled cotton fibre was used as a supporting matrix for the porous structure, that was obtained by combining of TEOS as a precursor, TMES as co-precursor, and oxalic acid as a catalyst, affording a structure with a porosity of 92.7 %, density of 0.068 g/cm^3 and apparent thermal conductivity of 0.0008 W/m °C. The final textile structure also showed high flame resistance, as well as mechanical properties and flexibility suitable for use in textile products where thermal insulation is a requirement, such as personal protective clothing and sports or technical textiles for the automotive sector. The results obtained in this work show the importance of developing upcycling processes for textile waste, as a strategy for the sustainable and circular development of the textile industry.

Keywords: upcycling - textile residues - textile porous structures - thermal insulation - flame resistance

[Resúmenes en inglés y portugués en la página 98]

(*) Máster en Ingeniería Química y Biológica (UM 2022). Es investigadora del Departamento de Química y Biotecnología del CITEVE. Su trabajo se basa en biomateriales procedentes del reciclaje textil y funcionalización de sustratos textiles.

(**) Licenciada en Ciencias de la Vida (2009, UofT), Máster en Química Médica (2011, UM) y doctoranda en Química Aplicada a las Ciencias. Es investigadora del Departamento de Química y Biotecnología del CITEVE. Su trabajo se basa en el reciclaje textil, la funcionalización textil mediante modificaciones superficiales químicas y el desarrollo de materiales innovadores.

(***) Máster en Ingeniería en Biotecnología y Bioprocesos (UM 2001) y Doctor en Ingeniería Química y Biológica (UM 2005). En la Universidad de Minho, es Profesora Asociada desde 2018, Directora del Programa de Doctorado en Bioingeniería desde 2020 y Directora Ejecutiva del Laboratorio de Bioprocesos y Bioproductos Sostenibles.

(****) Grado en Ingeniería Biológica (UM 1996), Máster en Biotecnología Industrial (USP-Brazil 1999) y Doctorado en Química Textil (UM 2005). En CITEVE es directora del Departamento de Química y Biotecnología, y tiene más de 20 años de experiencia en investigación aplicada a la industria, con más de 50 artículos y 10 patentes publicadas.

Introduction

The textile and clothing industry is one of the most intricate industrial sectors due to the intensive use of resources, increased greenhouse gas emissions, and generation of large volumes of wastewaters and textile residues. The largest source of textile waste is consumer discarded clothing and household textiles, representing approximately 85 percent of total waste (Terra & Refashion, 2022). According to a study by the Fashion for Good platform, only 2% of end-of-life clothing is used for fibre recycling, with the vast majority going into landfill or incineration (*Circular Fashion in Europe: Turning Waste into Value | McKinsey*, n.d.).

Researchers are actively exploring new alternatives for recycling textile waste to reduce impacts on the environment. To increase the economic incentive for recycling textile waste, we have successfully developed a highly porous aerogel-like material, with thermal insulation properties, using cotton (CO) fibers derived from post-consumer textiles.

Aerogels are very light, ultra-porous materials (> 90%) having a high a superficial area (>500 g/m^2), low density (< 0.2 g/cm^3) and a low thermal conductivity (< 0.02 W/m °C) (Rezaei & Moghaddas, 2016), (Zhang et al., 2020). The production of aerogels commonly occurs in three steps: (i) formation of *sol*-gel, the sol is prepared by a precursor, typically a silica based aqueous solution and gelation occurs with the addition of a catalyst (ii) ageing, which involves solvent-exchange that strengthens the gel by reinforcing the cross-linking and prevents excessive shrinkage during the drying step and (iii) drying, a critical step as the properties possessed by the final product are highly influenced by the temperature and pressure applied (Akhter et al., 2021).

Tailoring important parameters such as, molar ratio, *sol* pH, gelation time, aging, washing, solvent exchange, and precursors used can alter the physico-chemical characteristics of aerogels, offering distinct properties depending upon the specific applications.

Our approach is established by using ambient pressure drying of the silica-based gel which is reinforced with recycled CO fibers and suitable for thermal insulation applications.

Ultimately, the aerogel-like material was incorporated into two textile substrates through a lamination process and its thermal conductivity was studied along with other pertinent properties. Herein, the thorough synthetic approach and characterisation of the aerogel-like materials are described.

Materials

The materials used to obtain the porous structures consisted of the following: recycled CO (Citeve), textile substrate 1, textile substrate 2, TEOS (TCI, 97 %), Oxalic acid (Thermo Scientific, 0.1 N), MTMS (Thermo Scientific, 97 %), Absolute Ethanol (Carlo Erba), TMES (Acros Organics, 95 %), Sodium silicate (Sigma-Aldrich), Urea (QuimiTécnica), Acetic Acid (Fisher, 99.7 %), Sodium Hydroxide (NaOH) (Fisher), Glyoxal (Sigma-Aldrich, 40 % aqueous), Ammonium Hydroxide (Carlo Erba, 30 % aquoso), PVA (Sigma-Aldrich, 87 – 90 %), Glycerol (Himedia), ECH (Fluka Analytical, 99 %) e MBA (Sigma-Aldrich, 99.5 %).

Methods

In an initial phase, three methods for obtaining aerogels (with some modifications) were used as reference according to some procedures found in literature (Rezaei & Moghaddas, 2016), (Zhang et al., 2020), (Qi et al., 2011). The drying phase was common to all three methods used and consisted of placing the petri dish in an oven between 6 and 18h at 55 to 70 °C.

Method 1: According to Rezaei & Moghaddas, the selected silica precursor was mixed with water and a catalyst to form the sol solution. The latter solution was then poured over a petri dish containing the recycled CO fibres.

Method 2: Based on the procedure of Zhang *et al.*, the selected silica precursor was mixed in an aqueous ethanolic solution, in the presence of a catalyst. The latter solution was placed in a heating plate at 35 °C for 30 minutes. Next, an equivolume solution of diluted ammonium water and ethanol was prepared and added to the previous solution and placed stirring at room temperature for another ten minutes. Finally, the solution was poured into the petri dish that contained the recycled CO fibres.

Method 3: Following the approach used by Qi *et al.*, a dissolution of the cotton fibres before gel formation was carried out. Initially, a mixture of NaOH and urea was prepared and placed in the freezer for two hours. The latter solution was added to a beaker containing the recycled CO fibres and mixed for 5 minutes using an overhead stirrer. In some trials, different cross- linkers were used to reinforce the structure.

- Study of Procedural Conditions

This study was carried out to evaluate the effect of different parameters, namely, drying temperature, the mass fraction of CO, and concentration of reagents used. To identify the ideal drying temperature, the drying phases were carried out at 40, 60, and 80 °C. The mass fraction of CO was studied using 3, 5.5 and 8% of CO. Moreover, to determine optimal proportions of the amount of silica precursor and catalyst to be used these parameters were varied.

- Production Optimisation

A new method was developed based on method 1 and 2. In this method, the catalyst and the solvent volumes were maintained to study different reagents, namely, TEOS, TMES, MTMS. Different cross-linking agents were also studied such as, ECH and PVA. The drying phase was carried out at 60 °C, for 16 hours.

- Scale-up and Uniformity of the Material Surface

The best method was applied to develop the scale-up sample using three times the volume of the abovementioned solutions. To smoothen the surface of the material, two samples were prepared and overlapped for 1 hour before the drying stage.

- Lamination Process

The incorporation of the porous structure produced within the textile substrate was performed using a thermal press. To obtain the composite material, a thermal adhesive (ABE 001 20 150) was placed between the two materials for 10s at 130 °C and pressed at 3 bar.

Characterisations

Porosity and density

The porosity (ϕ) was calculated using Equation 1 (Jiang et al., 2020):

$$\phi = \left(1 - \frac{\rho_{aerogel}}{\rho_{skeleton}}\right) \times 100 \qquad \text{Equation 1}$$

where $\rho_{aerogel}$ is the density of the aerogel (in g/cm³) and was calculated using Equation 2 and the $\rho_{skeleton}$ is the density of the structure of the reagents (in g/cm³) that originated the aerogel, calculated by Equation 3.

$$\rho_{aerogel} = \frac{m_{aerogel}}{v_{aerogel}} \qquad \text{Equation 2}$$

where $m_{aerogel}$ is the mass (in g) of the aerogel weighed on a digital balance (Mettler Toledo) and $v_{aerogel}$ is the volume (in cm³) of the aerogel measured with a pachymeter (VITO Digital Caliper).

$$\rho_{skeleton} = \frac{1}{\sum \frac{x_{reagent}}{\rho_{reagent}}}$$ Equation 3

where $x_{reagent}$ is the mass fraction of each reagent in the respective initial solution and $\rho_{reagent}$ is the density (in g/cm^3) of each reagent.

Thermal Conductivity

To analyse the behaviour of the samples to temperature, a textile substrate was placed on top of a heating plate (CAT) and each sample was placed on this textile substrate. With a thermal camera (Flir) the temperature was measured every 30 s, over a span of five minutes.
The thermal conductivity (in W/m °C), *k*, was calculated using Equation 4 (Liu et al., 2019).

$$k = \frac{Q \times \Delta x}{\Delta t \times \Delta T \times A_{superficial}}$$ Equation 4

where Q is the transferred heat (in kJ), is the thickness of the aerogel (in m), $A_{superficial}$ (in m^2) is the surface area, ΔT is the temperature difference between the surfaces of the materials (in °C) and Δt is the time during which the heat exchange occurred (in s).
The transferred heat, Q, was calculated using Equation 5.

$$Q = m \times c \times \Delta T$$ Equation 5

where m is the mass (in kg), c is the specific heat capacity of the cotton (in kJ/kg °C), ΔT is the temperature increase that occurs in the material during the test (in °C). The thermal conductivities presented are an estimate based on the theoretical thermal conductivity of the cotton fabric. The experimental conductivity of a cotton fabric was calculated as described above for the samples, and the results were subsequently extrapolated for comparison purposes.

Contact Angle

To assess the hydrophobic nature of the aerogel-like materials, the contact angle was determined using the optical tensiometer (Attension Theta).

Washing Resistance

The resistance of the aerogel-like material to washing was carried out using a domestic washing machine (Miele WS 5425). Using the washing machine, the aerogels were subjected to a washing cycle at 30 °C for 45 min using non-phosphate detergent (4% aqueous).

Flame resistance

The fire resistance was evaluated by placing each sample, aerogel, textile substrate, and functionalized textile approximately 1 cm from the flame during 10 s.

Results

Method Selection

Figure 1 shows a sample of the porous structure obtained using method 1. One can observe that it does not have a regular surface, the initial fibre colour was maintained and shrinkage did not occur during the drying phase.

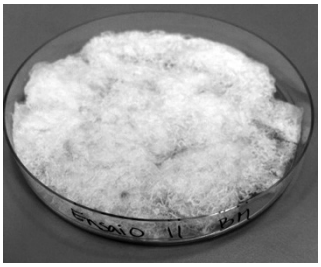

Figure 1. An example of the sample obtained by using method 1.

The graphics in Figure 2 present the density (a) and (b) porosity values for the samples obtained from method 1.

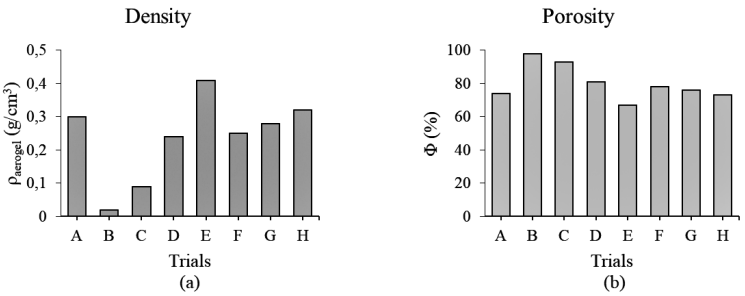

Figure 2. Values of density (a) and porosity (b) calculated for method 1 samples.

Regarding thermal conductivity, a test was conducted to better understand the behavior of the samples under temperature increase. Figure 3 shows the temperature gradient during the test.

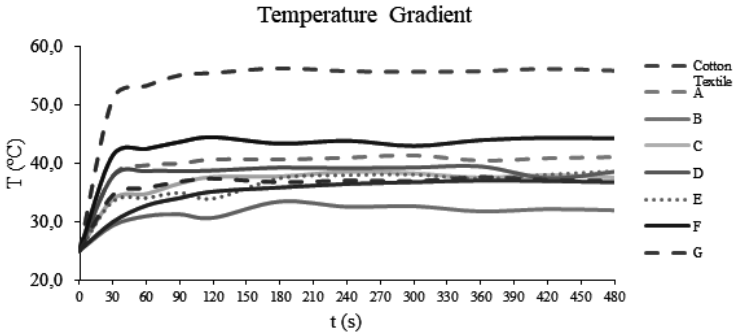

Figure 3. Temperature gradient obtained during thermal conductivity tests.

Generally, all the samples achieve their highest temperature at 90 s. It is evident that the textile substrate reached significantly higher temperatures, about 10 °C higher in relation to the porous structure samples. Figure 4 shows a sample obtained using method 2. The structures formed kept the initial colour of the CO fibres, shrinkage did not occur during the drying phase, but revealed uneven surfaces.

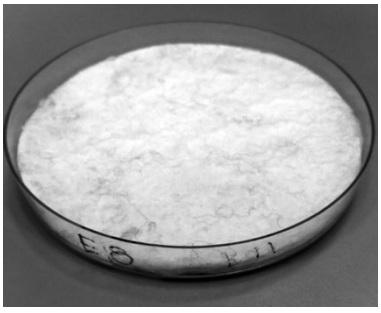

Figure 4. An example of the sample obtained by using method 2.

The values of the calculated density (a) and porosity (b) for samples obtained using method 2 are shown in Figure 5.

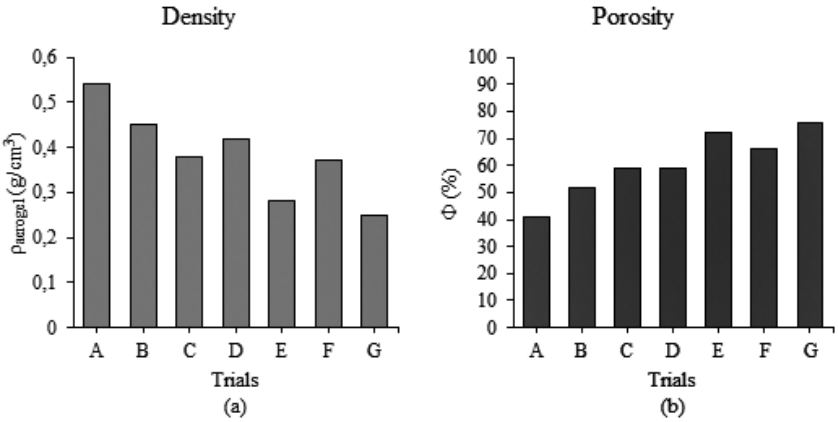

Figure 5. Values of density (a) and porosity (b) calculated for method 2 samples.

The calculated density values ranged between 0.28 and 0.54 g/cm^3 whereas the porosity values varied between 41 and 76%.
The apparent thermal conductivity values for samples obtained using method 2 are shown in Table 1.

Trial	Apparent Thermal Conductivity (W/m)
A	0.31
B	0.24
C	0.09
D	0.15
E	0.09
F	0.40
G	0.28

Table 1. Calculated apparent thermal conductivity for method 2 samples

The apparent thermal conductivity values ranged between 0.09 and 0.40 W/m .
An example of the structures obtained using method 3 are shown in Figure 6. Shrinking was not observed for these samples and colour change of the initial CO fibres did not occur. In contrast to the other two methods, the surface of the structures using this approach were more even.

Figure 6. An example of the sample obtained by using method 2.

In Figure 7, the calculated values of density (a) and porosity (b) for samples obtained using method 3 are shown.

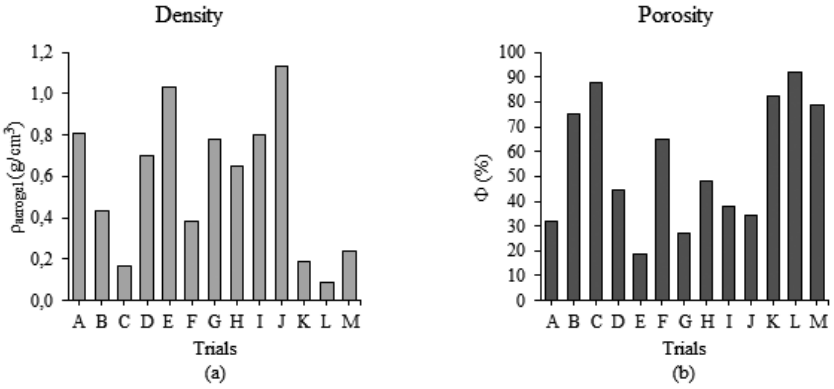

Figure 7. Values of density (a) and porosity (b) calculated for method 3 samples.

This method contained the largest difference of the porosity values ranging between 19 and 92%. The highest density value attained was 1.13 g/cm^3 and the lowest 0.09 g/cm^3. The apparent thermal conductivity values for the samples obtained using method 3 are listed in Table 2. The porous structure with the best apparent thermal conductivity was achieved for trial C, 0.09 W/m .

Trial	Apparent Thermal Conductivity (W/m)
A	0.87
B	0.33
C	0.09
D	0.74
E	0.63
F	0.41
G	0.3
H	0.35
I	0.11
J	0.67
K	0.4
L	0.12
M	0.57

Table 2. Calculated apparent thermal conductivity for method 3 samples

Procedural Conditions Study

In Figure 8, the aerogel-type structures achieved during this phase are presented.

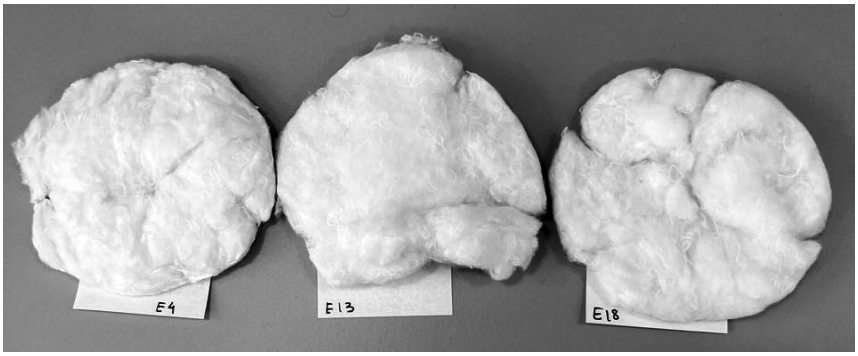

Figure 8. Examples of the samples developed during procedural conditions study.

The materials obtained lacked cohesiveness, showing loose fibres which is indicative that the reaction between the CO fibres and the solution was insufficient. Hence, a three-dimensional network was not achieved. These structures have density values between 0.03

and 0.08 g/cm^3. The porosity values oscillated between 93 and 97 %, not presenting considerable differences for this parameter. Based on the properties studied, the mass fraction of CO (6%) and the temperature (60) of the drying phase were established.

Optimisation Process

During the optimisation process, the aerogel-type structures were improved showing a well-defined three-dimensional structure, as shown in Figure 9. These samples did not reduce in size drying the drying stage. Moreover, the side in contact with the petri dish presented a smooth surface.

Figure 9. Examples of the samples developed during optimisation.

The flexibility was qualitatively accessed by cautiously folding the samples to understand their behavior, as shown in Figure 10.

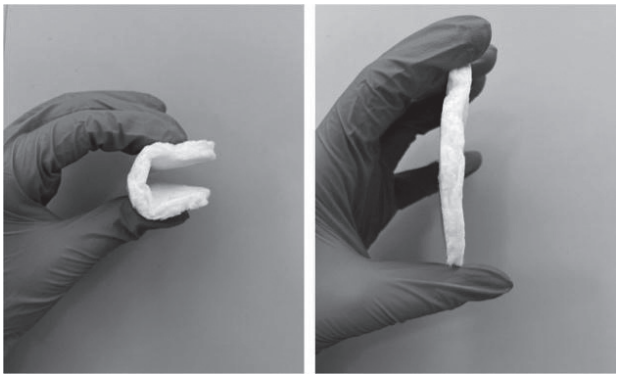

Figure 10. Flexibility demonstration of the optimised samples.

The samples presented a good degree of flexibility, as they were folded and returned to the original position without damage.

The parameters evaluated are shown in Figure 11. In general, all samples demonstrate low density (Figure 11 a), high porosity (Figure 11 b), and low apparent thermal conductivity (Figure 11 c). The best results were obtained for sample T. This experimental condition was selected for use in future experiments.

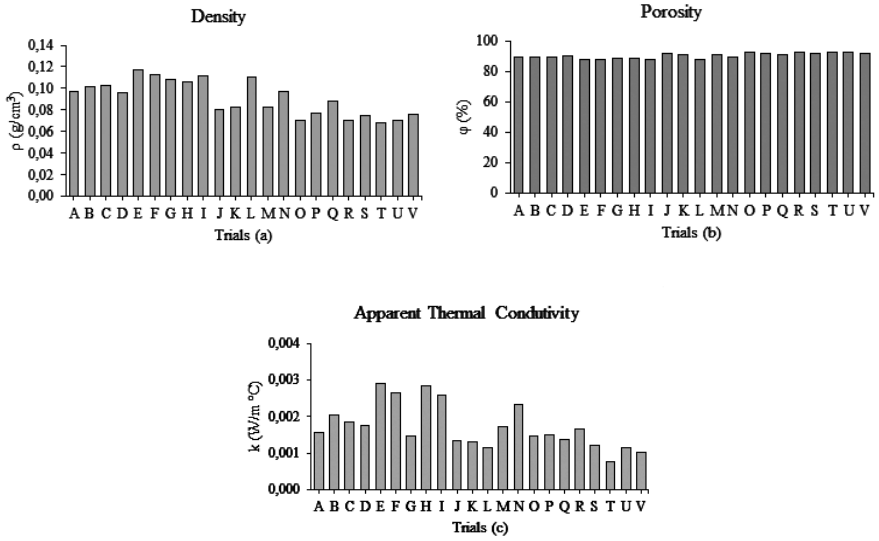

Figure 11. Values of density (a) porosity (b) and apparent thermal conductivity (c) calculated for the optimised samples.

The contact angle was only measured for the samples with the best results in terms of porosity and density obtained during the optimization phase. The results are shown in Figure 12. Samples with contact angles of at least 130° are considered hydrophobic materials. The results obtained show that all structures have hydrophobicity.

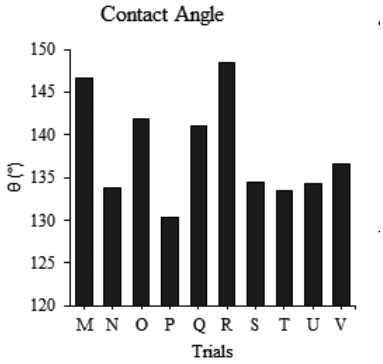

Figure 12. Contact angle measured for the optimised samples.

The hydrophobic behavior of the samples is demonstrated in Figure 13, where a drop of water is repelled by the hydrophobic surface of one sample.

Figure 13. Hydrophobic behavior of the samples.

Scale-Up and Uniformity Process

A scale-up was performed using the same conditions as sample T to access the potential applications of the aerogel-type structure developed. In parallel, another trial was conducted to smoothen both sides of the porous structure surfaces. The scale-up sample is shown in Figure 14 (a) and the uniformisation trial is shown in Figure 14 (b).

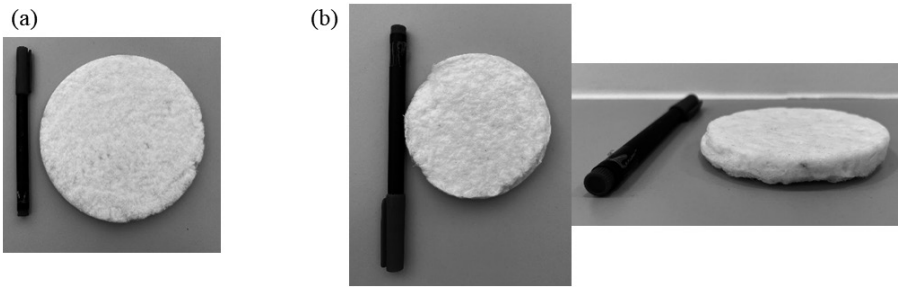

Figure 14. Scale-up sample (a) and uniform sample (b).

These trials were characterized regarding their porosity, density, and apparent thermal conductivity (Table 3).

	Porosity (%)	Density (g/cm^3)	Apparent Thermal Conductivity (W/m °C)
Trial T	93	0.068	0.001
Scale-up	92	0.076	0.002
Uniformisation	91	0.082	0.001

Table 3. Comparison of properties studied for each sample

Both trials were successfully carried out and in comparison, to trial T, both maintained similar characteristics.

Lamination

The scaled-up and uniformised samples were each incorporated with one of the two different textile structures using a thermal press. Figure 15 shows the laminated scaled-up composites.

After the lamination process both composites were submitted to a washing resistance test, using a washing cycle at 30 °C for 45 minutes. Overall, both materials kept their structural integrity.

The thermal conductivity was calculated for both textile substrates, 1 and 2, which presented an estimated thermal conductivity of 0.007 W/m °C and 0.003 W/m °C, respectively. After the incorporation of the scale up trial in textile substrate 1, it presented an apparent thermal conductivity of 0.001 W/m °C and after the incorporation of the uni-

formisation trial in textile substrate 2, it presented an estimated thermal conductivity of 0.0006 W/m °C. Therefore, the lamination was successful and resulted in an improvement of the individual characteristics of the fabric materials.

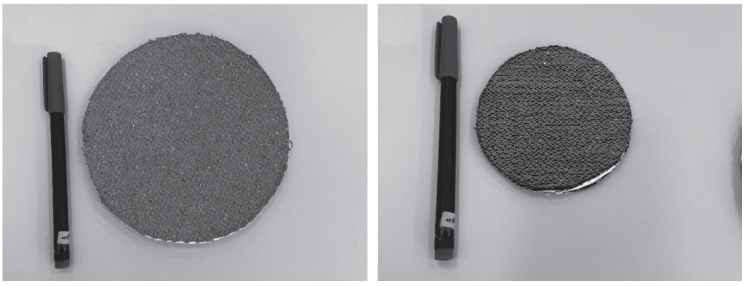

Figure 15. Laminated scale-up (a) and uniform (b) samples.

In addition, the laminated porous structure were submitted to a flame resistance test, as demonstrated in Figure 16.

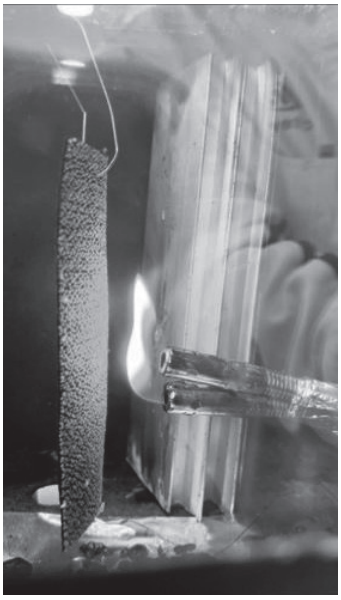

Figure 16. Flame resistance test.

The materials maintained their initial properties and demonstrated flame retardant capacity as depicted in Figure 17, where the samples show no signs of contact with fire.

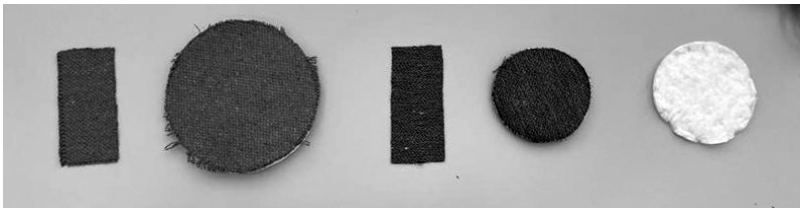

Figure 17. Samples after the flame resistance test.

Discussion

When trying to identify the best method to produce the aerogel-type structures, method 1 produced the materials with the best properties and it presented a homogeneous behaviour regarding the apparent thermal conductivities. However, in comparison to the other two methods, this method achieved higher porosity and lower density values. Hence, method 1 was selected to study the processing conditions.

Different conditions were carried out using method 1 to identify the best conditions to generate the porous structures. Even though these conditions produced porous structures lacking cohesiveness it was possible to establish some conclusions. Regarding the drying temperature, 60 °C was identified as the most adequate drying temperature since it produced the most convenient structures in relation to the other drying temperatures studied, 40 and 80°C. Moreover, the ideal mass fraction of cotton was identified (6% m/v). When using a lower CO mass fraction such as 3% (m/v), structures with void spaces were obtained. Whereas, when using a higher CO mass fraction, such as 8% (m/v), the volume of *sol* used was insufficient to incorporate all the CO fibres used.

Ultimately, the combination of the experimental conditions of method 1 and 2 were used to optimise the production process. All the samples produced during this phase had low densities, high porosities, and low apparent thermal conductivities. The samples were predominately qualitatively evaluated, based mostly on the flexibility of the porous structures. Based on the best structures developed, the most adequate reagents were selected, namely TEOS, TMES as precursors, and ECH as cross-linker. The best porous structure developed was Trial T which showed a porosity of 93%, density of 0.06 g/cm^3 and an apparent thermal conductivity of 0.001 W/m °C. Hence, the experimental conditions used to develop Trial T were used for further studies, namely, scale-up and uniformisation trials.

The scale-up and uniformisation trials were also evaluated in terms of porosity, density, and apparent thermal conductivities, not showing considerable changes in comparison

to Trial T (Table 3). The porous structures obtained from the latter trials were combined with different textile substrates to produce composites. The lamination resulted in an improvement of the individual characteristics of the materials (fabric and spacer), reducing apparent thermal conductivity hence, proving to be suitable as thermal insulating materials. These composites were then tested for their washing resistance, showing no considerable modifications of the structure. Additionally, a flame resistance test was conducted in which the composites show flame retardant properties.

Conclusions

We successfully prepared an aerogel-type composite made from recycled CO. The composite is a promising candidate and suitable for use as thermal insulation in textiles as it has an apparent low thermal conductivity of 0.0006 W/m °C. In addition, these materials exhibit good hydrophobic, compressive and fire-resistant properties. Obtaining fibers from textile waste to produce these aerogel-like composites further enhances the sustainability and circularity of the textile and clothing industry.

Acknowledgments

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Resumen: Cada europeo genera alrededor de 15 kg de residuos textiles al año, y alrededor del 65% de estos residuos todavía terminan en los vertederos, a pesar de la recogida selectiva obligatoria de residuos textiles a partir del 1 de enero de 2025. Actualmente, el reciclaje mecánico es el método más común de residuos textiles. reciclaje. Este proceso de reciclaje tiene varias carencias (por ejemplo, baja productividad, dificultad para separar por tipo de fibra, dificultad para reciclar ropa por la necesidad de quitar accesorios) y suele generar productos de bajo valor comercial (downcycling). En este trabajo se desarrolló una estructura textil porosa de tipo aerogel utilizando algodón (CO) reciclado mecánicamente a partir de residuos textiles, a medida para obtener un material con propiedades aislantes. Durante este proceso, se utilizó fibra de algodón reciclado como matriz de soporte para la estructura porosa, que se obtuvo mediante la combinación de TEOS como precursor, TMES como co-precursor y ácido oxálico como catalizador, proporcionando una estructura con una porosidad de 92,7 %, densidad de 0,068 g/cm3 y conductividad térmica aparente de 0,0008 W/m °C. La estructura textil final también mostró una alta resistencia a la llama, así como propiedades mecánicas y flexibilidad adecuadas para su uso en productos textiles donde el aislamiento térmico es un requisito, como ropa de protección personal y textiles deportivos o técnicos para el sector de la automoción. Los resultados obtenidos en este trabajo muestran la importancia de desarrollar procesos de upcycling de residuos textiles, como estrategia para el desarrollo sostenible y circular de la industria textil.

Palabras clave: upcycling - residuos textiles - estructuras textiles porosas - aislamiento térmico - resistencia a la llama

Resumo: Cada europeu gera cerca de 15 kg de resíduos têxteis por ano, sendo que cerca de 65% destes resíduos ainda vão parar em aterros, apesar da obrigatoriedade da recolha seletiva de resíduos têxteis a partir de 1 de janeiro de 2025. Atualmente, a reciclagem mecânica é o método mais comum de reciclagem de têxteis reciclando. Este processo de reciclagem apresenta várias deficiências (por exemplo, baixa produtividade, dificuldade

de separação por tipo de fibra, dificuldade de reciclagem de roupas devido à necessidade de retirar acessórios) e costuma gerar produtos de baixo valor comercial (downcycling). Neste trabalho, desenvolveu-se uma estrutura têxtil porosa do tipo aerogel utilizando algodão (CO) reciclado mecanicamente a partir de resíduos têxteis, sob medida para a obtenção de um material com propriedades isolantes. Nesse processo, foi utilizada fibra de algodão reciclada como matriz suporte para a estrutura porosa, que foi obtida pela combinação de TEOS como precursor, TMES como coprecursor e ácido oxálico como catalisador, resultando em uma estrutura com porosidade de 92,7 %, densidade de 0,068 g/cm3 e condutividade térmica aparente de 0,0008 W/m °C. A estrutura têxtil final também apresentou alta resistência à chama, bem como propriedades mecânicas e flexibilidade adequadas para uso em produtos têxteis onde o isolamento térmico é um requisito, como roupas de proteção individual e esportivas ou têxteis técnicos para o setor automotivo. Os resultados obtidos neste trabalho mostram a importância de desenvolver processos de upcycling para resíduos têxteis, como estratégia para o desenvolvimento sustentável e circular da indústria têxtil.

Palavras-chave: upcycling - resíduos têxteis - estruturas porosas têxteis - isolamento térmico - resistência à chama

[Las traducciones de los abstracts fueron supervisadas por su autor]