Simple preparation of cellulosic lightweight materials from eucalyptus pulp

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ABSTRACT: Cellulosic foams and aerogels are tridimensional materials prepared from cellulose fibers and nanostructures that display interesting properties, such as extremely low density, high fluid permeability, sound and heat insulation. Currently, the most common techniques to obtain such porous matrices are gel or foam forming, followed by freeze-drying or critical point drying, which are energy and time-consuming processes for solvent removal. In this work, we present a new methodology to produce cellulosic lightweight materials from eucalyptus pulp, using cellulose fibers partially hydrolyzed with sulfuric acid. This method is based on a drying step easily performed at mild temperatures around 60°C in a convection oven and eliminates the need of more sophisticated drying techniques. In addition, the procedure does not require the use of surfactants or special foam forming equipment. Micro-CT and FESEM analysis showed the formation of a porous and lightweight material (density as low as 0.15 g/cm^3), where the fibers are randomly assembled in a 3D-network with a few contact points. Mechanical testing reveled that foams of hydrolyzed fibers have great performance under compressive strain, with high mechanical energy absorption (*ca.* 360 kJ/m^3). This purely cellulosic material is suitable for the incorporation of particles or functional groups aiming a wide range of final applications.

INTRODUCTION

Lightweight materials of cellulose are highly porous 3D-structures composed of fibers, nanocrystals (CNC) and/or nanofibers (CNF), generally referred in the literature as foams[1] or aerogels.[2] These naturally-derived materials have been investigated covering various applications due to their long range of interesting properties such as lightness (ultralow density),[3,4] high surface area,[5,6] high fluid permeability,[7] sound absorption,[8] mechanical properties, such as relatively high strenght[7,9] and energy absorption capability,[6] along with the intrinsic characteristics of cellulose, such as renewable source, tunable surface chemistry, biodegradability and low toxicity.[2]

Cellulose foams can be prepared using micrometric fibers and also nanoparticles, mainly CNF.[10,11] A dispersion is firstly obtained by stirring the particles in water for air incorporation in the presence of surfactants, followed by foam draining and drying.[12,13] During draining, the water excess is usually removed by gravity, balancing opposing forces, such as capillary pressure, gravity, and mechanical pressure. The residual humidity that remains after liquid flow can be dried under heating using ovens, air impingement, microwave or infrared radiation.[13] These methods are scalable and can be continuous, so that cellulose foams emerge as interesting green substitutes for polymeric foams derived from fossil fuels. However, dealing with wet foams is not a simple task, as these systems are thermodynamically unstable and many factors must be carefully controlled to slow down processes such as draining, coalescence and Ostwald ripening,[14] in order to keep the system metastable until liquid removal. Moreover, it is important to notice that, since the preparation requires an interface active agent, the final material will not be a pure cellulose matrix.

Differently, cellulose aerogels are usually obtained by dispersing CNC and/or CNF in a solvent, forming a hydrogel or organogel, which subsequently undergo freeze-drying or critical point drying, so that the structure of the colloidal dispersion remains in the final solid.[2] Cellulose foams can also be freeze-dried, though the heating techniques previously cited are more common.[10,11] The freeze-drying technique requires an initial freezing step and long periods of time for solvent removal under vacuum. In its turn, critical point drying requires special equipment, high temperatures and pressures, and many steps for solvent exchange[5,15] prior drying with supercritical CO_2. Solvent changes must be carried out avoiding mechanical disturbance to the structure and can take several days to be concluded. Besides the demand of energy and time, both techniques restrict the final size of the dried pieces due to the equipment size. Thus, though very efficient in a laboratory scale, these drying techniques are not adequate for low-cost applications.[9,16]

A less expensive and simpler alternative procedure to prepare nanocellulose lightweight materials was presented by Wågberg and collaborators,[17] in which foams were obtained by mechanical agitation of a dispersion of CNF and octylamine in water. The formed foam was filtered to remove the water excess and then dried in oven, leading to a highly porous cellulosic material (87 to 98%), with low density (0.03 to 0.20 g/cm^3).[17] In a second publication, they improved the drying step to produce more homogeneous pores, keeping a high porosity (99.6%), and introducing a chemical cross-linking to make the foam wet-resilient.[18] Another ambient-drying method applicable to thin samples was developed by Ikkala and colleagues,[9] using a wet gel-cake that was prepared from an aqueous CNF dispersion by vacuum filtration. This gel was solvent exchanged 3 times with 2-propanol (soaking for 5 min in each change) and, then 3 times with octane, after which the gel was allowed to dry at ambient conditions.[9] In a different methodology, foams of cellulose fibers were prepared under vigorous agitation, in the presence of sodium dodecyl sulphate (SDS) and dried at 70°C on a moving draining plate. These solid foams obtained in the form of sheets presented low density (0.005 g/cm^3) and high softness, and were proposed for applications as support for polymer-impregnated composites and absorbents.[12]

Here, a new and simpler methodology to produce cellulosic lightweight materials is proposed, using cellulose micrometric fibers from eucalyptus pulp partially hydrolyzed with sulfuric acid. Hydrolyzed fibers are manually dispersed in water with a glass rod and highly porous matrices can be easily obtained after oven drying at 60°C. This procedure does not require the use of surfactants, neither an air incorporation step, but we consider the final materials as foams, because of the similarity with cellulose materials prepared by foam-forming methods.[7,12] These solid foams present a superior mechanical energy absorbing capability as compared to the typical results reported for cellulosic lightweight materials.[5,18]

EXPERIMENTAL

2.1. Eucalyptus pulp hydrolysis

Eucalyptus pulp was kindly donated by *Suzano Papel e Celulose* (São Paulo, Brazil). A dispersion was prepared with 15 g of eucalyptus pulp and 150 mL of distillated water and homogenized at 200 rpm during 30 min, using a mechanical stirrer (713D, Fisatom). The fiber dispersion was added to 90 mL of a 60% (w/w) H_2SO_4 (purity ≥98%, Synth) aqueous solution (both were previously heated to 45°C) to achieve a final proportion of 1 g of fibers : 40 mL of acid solution and a final concentration of 48% (w/w) H_2SO_4. This is an acid concentration typically lower than the ones commonly used in cellulose fiber hydrolysis[19] and should result in partial hydrolysis. The mixture was kept at 45°C under magnetic stirring (752A, Fisatom) for 1 h, after which the hydrolysis was interrupted by the addition of 500 mL of water and ice. The mixture was centrifuged at 3500 rpm (RC-3B, Sorvall) during 10 min, then the supernatant was removed, leaving a pellet of sedimented fibers. This pellet was rinsed with 700 mL of deionized water and centrifuged 5 times, until the supernatant was clear and at neutral pH.

2.2. Cellulose foam preparation

Aqueous dispersions of hydrolyzed fibers were diluted to different final fiber concentrations (25, 30, 40, 50 and 63 g/L) and homogenized with a glass stirring rod. 33 g of each aqueous dispersion were transferred to polyethylene cylindrical molds (30 mm diameter and 48 mm height) with a permeable bottom of Teflon filter (70 mesh, Spectrum) and dried at 60°C in convection oven (TE-394/3, Tecnal), until constant weight (*ca.* 12h). At least 3 foams were prepared at each fiber concentration to estimate mean variations. For comparison, cellulose foams were also prepared with non-hydrolyzed eucalyptus pulp at 63 g/L, following the same procedure.

2.3. Field Emission Scanning Electron Microscopy (FESEM)

Scanning electron micrographs were obtained on the surface of transversal sections of the dried foams, using a FESEM FEI Quanta 650 microscope, operating at a 5 kV accelerating voltage, with a 3.0 spot size and using a secondary electron detector. Prior to FESEM analysis, samples were fixed with copper tape to a stub and sputtered with gold using a Bal-Tec SCD 005 coater equipped with a planetary drive stage to obtain a uniform gold coating.

2.4. Apparent density and porosity determination

The apparent density of the foams (ρ_{app}) was calculated by the ratio between the dried foam weight and the volume, with foam dimensions determined using a caliper. Foam porosity (P_{app}) was determined using ρ_{app} and cellulose density (ρ_{cel} = 1.6 g/cm^3),[20] according to Equation 1.[21]

$$P_{app} = \left(1 - \frac{\rho_{app}}{\rho_{cel}}\right) x\ 100 \qquad (1)$$

2.5. X-Ray Computed Microtomography (micro-CT)

Cellulose foams were analyzed using a Skyscan 1272 microtomographer (Bruker), operating at 20 kV, 175 µA, with a 0.4 ° rotation step and 4 frames per position. Scannings were carried out with resolutions of 21.6 µm (1224 x 820). NRecon software (v. 1.6.9.8, SkyScan) was used to reconstruct 3D images from cross sections images, using the Feldkamp algorithm. For the reconstruction, ring artifact correction number 20 was applied and smoothing correction level 1 was used for noise reduction. Contrast limits of electronic density were set at the same range for each sample, allowing comparisons between them. The CTVOx software (v. 2.2.3.0, SkyScan) was used for 3D visualization and image acquisition.

2.6. Mechanical Testing

Foams of hydrolyzed fibers (63 and 40 g/L) and foams prepared with non-hydrolyzed fibers (63 g/L) were cut in a 0.5 inch (1.27 cm) cubic shape and compressed at 1 mm/min using a Universal Testing Machine (Emic 23-20, Instron), according to ASTM D695 standard. Prior to mechanical testing, samples were conditioned at (23 ± 2)°C and (50 ± 5)% humidity for 48 h. Young modulus values were obtained from the angular coefficient in linear region of compressive curves (<5% strain). Energy absorption values were determined as the area under the curves from 0 to 70% strain.

RESULTS AND DISCUSSION

3.1. Physical properties of cellulose foams

Cellulosic materials obtained from partially hydrolyzed eucalyptus pulp fibers dispersed at different concentrations are presented in Figure 1. They have the visual aspect of solid foams and a cylindrical shape due to the vials used as molds. After hydrolysis, the fibers were dispersed in water at different concentrations, so that the existence of a minimum threshold concentration for foam formation by drying at 60°C could be investigated. But cellulose foams were formed from hydrolyzed fibers at all the concentrations tested, as well as from the non-hydrolyzed eucalyptus pulp at 63 g/L (Figure 2f), indicating no limit of concentrations for foam formation in this range. Foam heights are different for each concentration because the amount of water is higher in less concentrated foams, although the weight and the total volume of dispersion (33 mL) were kept constant in all the cases. In spite of the high water content in the dispersions, the final foam diameter after drying conserves between 76% and 92% of the mold diameter, showing a minor horizontal shrinkage in this step, which is desirable to obtain high porosity.[22] Moreover, the macroscopic shape of the cellulose foam made of non-hydrolyzed fibers (Figure 2f) is similar to the cellulose foam of hydrolyzed fibers prepared at the same concentration (Figure 2a).

The apparent density values of cellulose foams (Figure 2a) lie within 0.15 and 0.18 g/cm^3, thus indicating a high surface area. The foams prepared from non-hydrolyzed fibers have density of (0.161 ± 0.002) g/cm^3, which is below the density of cellulose foams of hydrolyzed fibers prepared in the same concentration (0.178 ± 0.007) g/cm^3. For hydrolyzed fibers, foam density tends to decrease with the concentration used, reaching a minimum value of (0.151 ± 0.004) g/cm^3 at 40 g/L. Below this concentration, density values tend to increase again and, at the minimum concentration tested (25 g/L), the density is (0.179 ± 0.008) g/cm^3. All the density values are very low for a material composed by micrometric fibers, since they are comparable to the density values of nanocellulose aerogels and foams reported in literature, which can be found in the 0.001 to 0.2 g/cm^3 range.[1] Porosity values are inversely proportional to apparent density (Figure 2b) and, consequently, the cellulose foam prepared using a 40 g/L dispersion is the most porous material (90.6% of porosity).

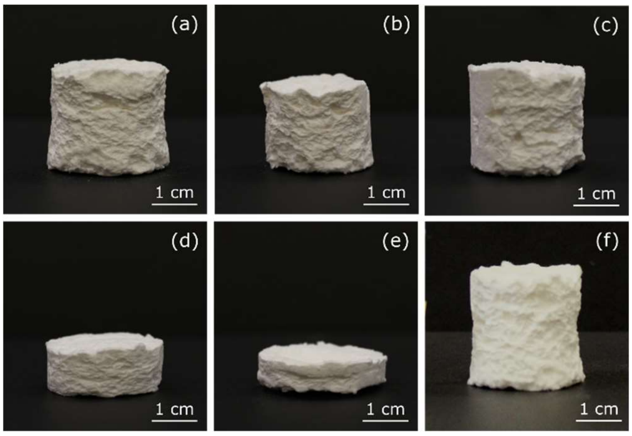

Figure 2. Cellulose foams prepared from aqueous dispersions of hydrolyzed eucalyptus fibers at different concentrations: (a) 63, (b) 50, (c) 40, (d) 30, (e) 25 g/L; and (f) cellulose foam prepared from aqueous dispersion of non-hydrolyzed cellulose pulp at 63 g/L.

3.2. Morphology and structure of cellulose foams

Although apparent density indicates similarity among the foams, they may have different morphological characteristics, such as internal pore sizes and fiber distribution. For this reason, foam morphologies were investigated using micro-CT and FESEM techniques. Reconstructed images of micro-CT reveal the 3D-morphology of cellulose foams at millimetric scale (Figure 3). In these images, lighter colors represent areas with higher electronic density, which in the case of these single-component samples are the regions where cellulose fibers are accumulated. Cellulose foams of hydrolyzed fibers have a homogeneous and random distribution of fibers throughout the material,

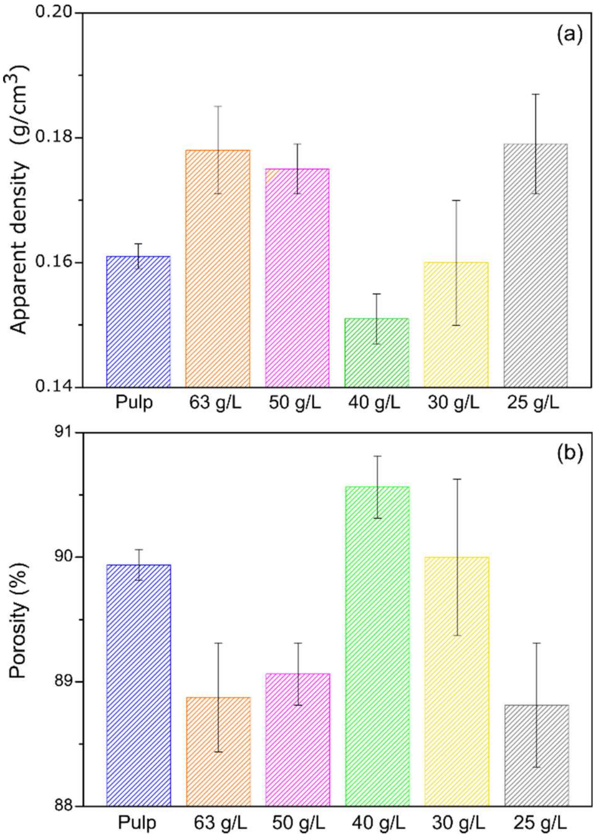

Figure 1. Physical properties of cellulose foams determined by measuring their mass and dimensions: (a) apparent density and (b) porosity calculated from the apparent density.

with pores that are more uniform in size and well distributed (Figures 3a-e). On the other hand, in the foam prepared with the non-hydrolyzed pulp (Figure 3f) an inhomogeneous distribution of fibers prevails, characterized by lighter regions and larger pores. Timonen and collaborators also observed that, although density values of cellulose foams were similar, their internal network structures could be very different.[23]

In addition, the morphology observed in the foams of hydrolyzed fibers is similar to those of fiber materials obtained by foam forming method in the presence of surfactants,[7] which also may have a layered orientation perpendicular to the draining direction.[24] The random orientation of fibers obtained here usually does not occur in foams of CNF, because their preparation uses surfactants, which induces CNF accumulation at the water/air interface, producing a bubbly morphology that can appear in ambient-dried[17,18,25] and freeze-dried foams.[26] A more random assemble of CNF can be acquired using a method free of surfactants, but then CNF tend to align perpendicularly to the draining direction.[9]

Microscopy images show open assemblies of fibers with a few contact points, evidencing that the foams prepared from hydrolyzed fibers present high surface area (Figure 4a). This is a structure quite different from the one presented in paper, where fibers have a compact 2D-plane orientation.[12] Throughout the material, it is possible to find points where fibers are intertwined (detail in Figure 4b) and tensioned (detail in Figure 4c). These connected regions with tensioned fibers are important to prevent shrinkage during drying, because it would be necessary to deform these tensioned points to contract the material. Figure 4d reveals a detail in the contact of two fibers,

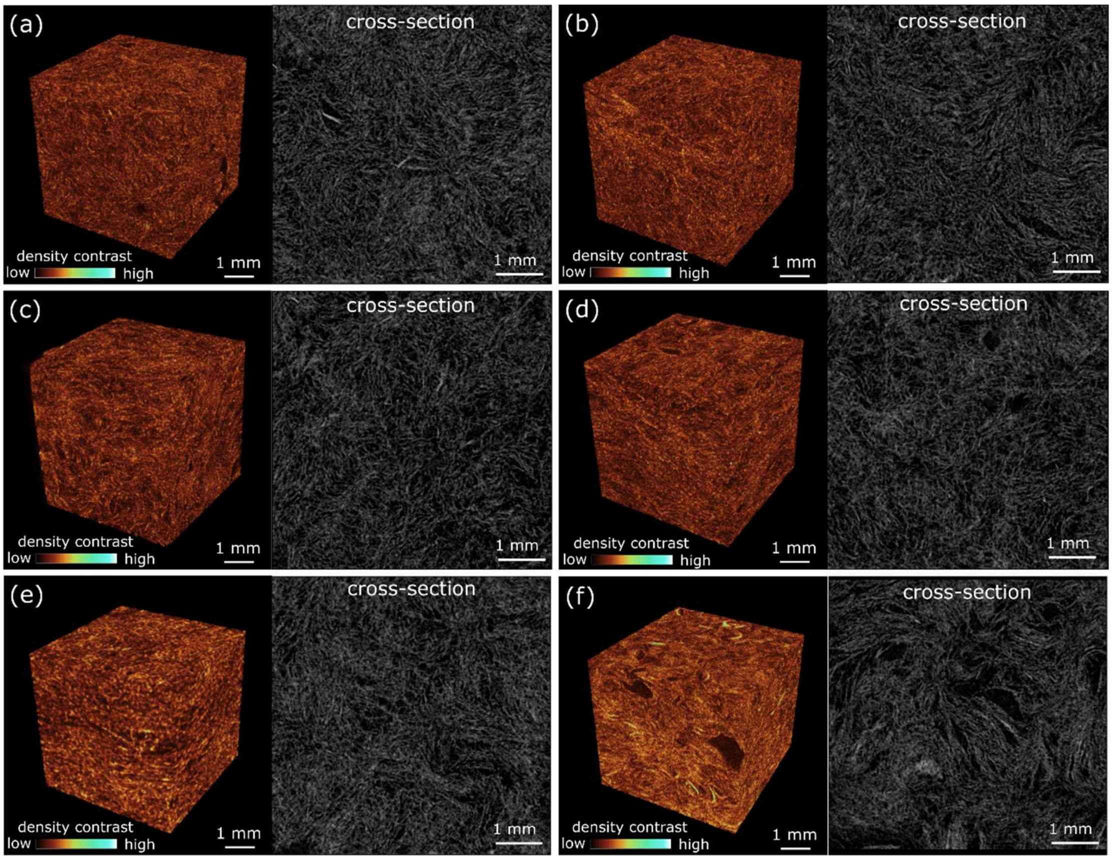

Figure 3. Micro-CT reconstructed images of cellulose foams prepared with aqueous dispersions of hydrolized fibers at concentrations: (a) 63, (b) 50, (c) 40, (d) 30, (e) 25 g/L and (f) foam with non-hydrolyzed fibers at 63 g/L.

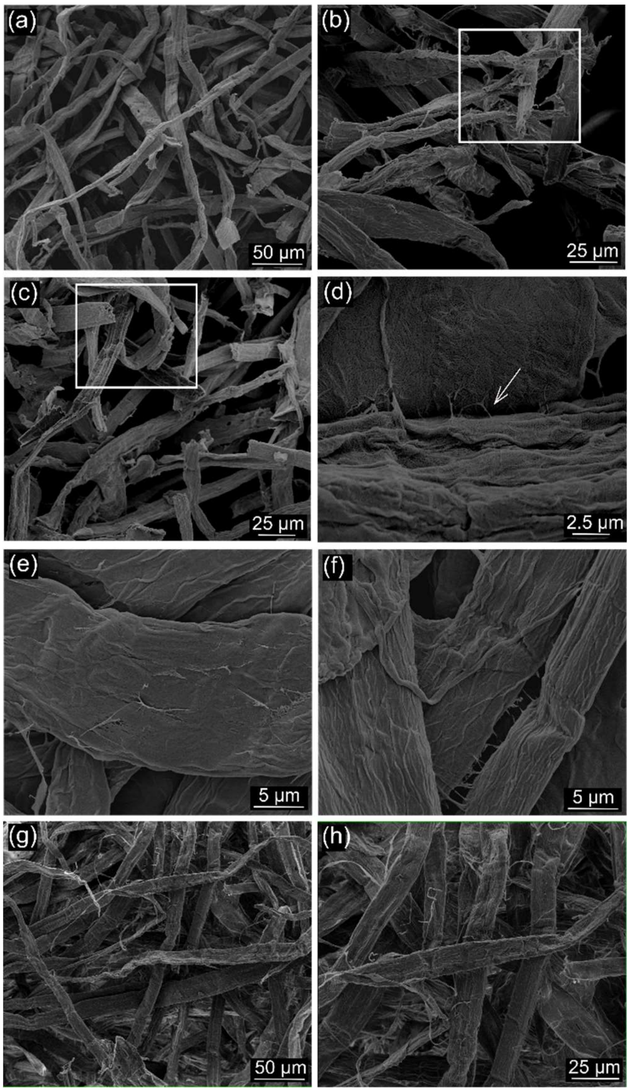

Figure 4. FESEM micrographs of cellulose foams prepared with an aqueous dispersion (63 g/L) of cellulose hydrolyzed fibers: (a) overview of the hydrolyzed foam, (b) detail in the structure showing fiber interlocking, (c) tensioned fibers, (d) detail of the foam structure showing nanofibrils in the joint of two fibers; images of eucalyptus fibers: (e) non-hydrolyzed and (f) hydrolyzed; (g-h) overview of cellulose foam prepared with non-hydrolyzed fibers.

showing fibrils interconnected. Moreover, acid hydrolysis exposed thin fibrils on the surface, providing conditions for interlocking at nanometric scale, as well as greater surface area for intermolecular interactions (Figures 4e-f).

Pulp fibers can have entwined during stirring of the dispersion for foam preparation or during drying, when capillary forces approximate fibers, making their surfaces come into contact and adhere. In the wet state, the contact area between fibers is improved, because the fiber outer layer is soft and covered by a hydrogel of thin fibrils partially attached to the fiber structure, which allows the fibrils to migrate and interdiffuse, favoring the adhesion.[27]

Foams prepared from non-hydrolyzed pulp (Figures 4g-h) exhibits some fibrils on the fiber surface and a less porous structure as compared to the foam of hydrolyzed fibers. Although the apparent density of the foam composed of pulp fibers is lower, its surface area can be smaller compared with those with hydrolyzed fibers. The different arrangements of the two types of foams, observed in Micro-CT and FESEM, indicate that hydrolyzed fibers may have distinct characteristics in aqueous media that prevent the structure to collapse during drying.

Prior partial acid hydrolysis is required to achieve a more opened structure, because this step disjoints the fibers, disaggregating their constituent fibrils. But when an aqueous dispersion of fibers is dried, fibers approximate, leading to the formation of aggregates and closing the structure. So, an opposite repulsive interaction must be considered to explain the maintenance of the random assembly and that may be related to the presence of sulfate groups on fiber surface. It is well known in literature that cellulose hydrolysis with sulfuric acid causes addition of sulfate esters in cellulose molecule.[19,28,29] At the mild conditions of acid hydrolysis used here, some sulphate ester groups can also be formed on the surface, as it was evidenced by X-ray fluorescence (Supplementary Information).

The role of negative charges in maintaining an open structure can be described as follows: as the dispersion is dried, fibers became near in contact due to liquid draining and capillary pressure. However, fibers do not get entirely in contact due to the negative charges repulsion of sulfate esters (Figure 5). The balance between attractive forces (capillary adhesion and intermolecular forces) and repulsive forces (electrostatic repulsion of sulfate groups) keeps fibers at an equilibrium distance, which is favored by the mechanical interlocking that restricts fiber displacement at long distances. Another evidence of this stabilization mechanism is the fact that using fibers partially hydrolyzed with hydrochloric acid (HCl) at equivalent conditions, the foams prepared collapse during drying, probably because there is no electrostatic contribution to prevent fiber approximation (for more details, see Supplementary Information).

3.3. Mechanical properties of cellulose foams

Compressive testing of cellulose foams (Figure 6) exhibit typical compression curves for foams, with three distinct regimes:[30] (I) small linear region at low compression (elastic regime); (II) plateau region, where the material absorbs energy but maintains stress almost constant by plastic deformation and cell collapse; (III) densification region, where opposite cell-walls are compressed against each other and stress increases abruptly with compression. As porosity values of the tested foams are very similar (89 to 90%), plateau (II) and densification (III) regimes are situated in the same compression regions for all samples. During the testing, cellulose foams did not fail, which indicates that they absorb mechanical energy by pore collapse and plastic deformation mechanisms.

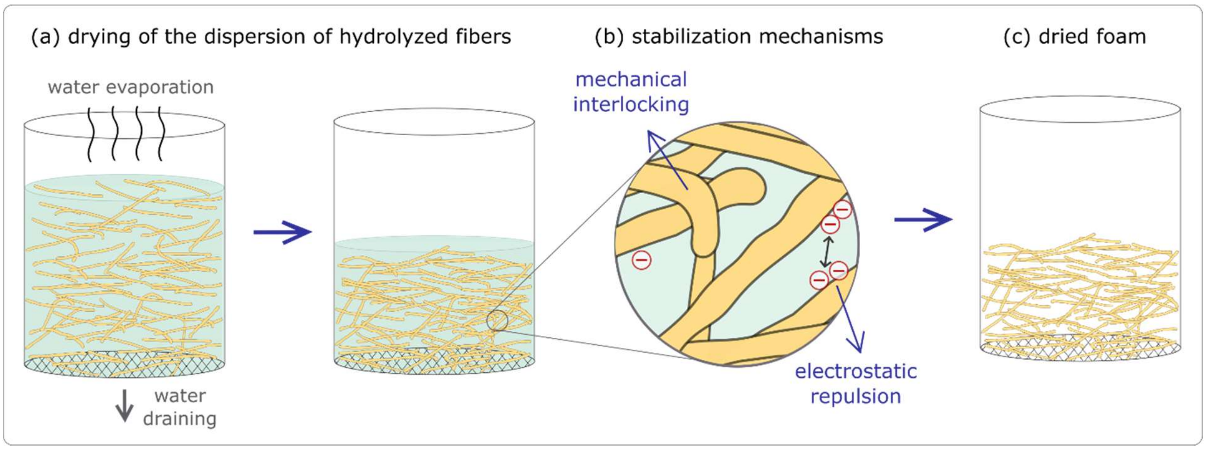

Figure 5. Schematic representation of (a) hydrolyzed fiber dispersion being dried, (b) stabilization mechanisms that maintain the network structure and (c) dried foam.

Foams of hydrolyzed fibers prepared at 63 and 40 g/L concentrations have distinct stress values at low compression (less than 10%), resulting in distinct Young modulus. As compression increases, stress reach equivalent values of mean maximum stress: (3.8 ± 0.2) MPa and (3.9 ± 0.2) MPa, for 63 and 40 g/L foams, respectively. On the other hand, the foam of pulp fibers presents a different mechanical profile, achieving lower value of maximum compressive stress (2.4 MPa).

Berglund and collaborators proposed a relationship between density and mechanical properties of lightweight materials, verifying that Young modulus increases with the aerogel density.[6] This correlation was also previously reported for polyurethane (PU) foams[31] and predicted for polymeric foams in general.[32] Therefore, for comparisons between different materials, it is more pertinent to use values of Young modulus normalized by density (specific Young modulus).[33] Cellulose foams with hydrolyzed fibers present specific Young modulus of 24 and 12 MPa cm^3g^{-1} (Table 1), which are higher than the value reported for CNF freeze-dried aerogel,[33] and are in the same order of magnitude of specific Young modulus for CNF aerogels obtained by critical point drying[5] and freeze-drying.[6] Specific modulus can be greatly increased by clay addition to this materials, forming a composite CNF aerogel (10270 MPa cm^3g^{-1}).[34] Moreover, although PU foams are relatively rigid, specific Young modulus of cellulose foams can be higher than in some PU foams,[35] which indicates that cellulose lightweight materials may be good candidates to replace those synthetic materials derived from fossil fuels.

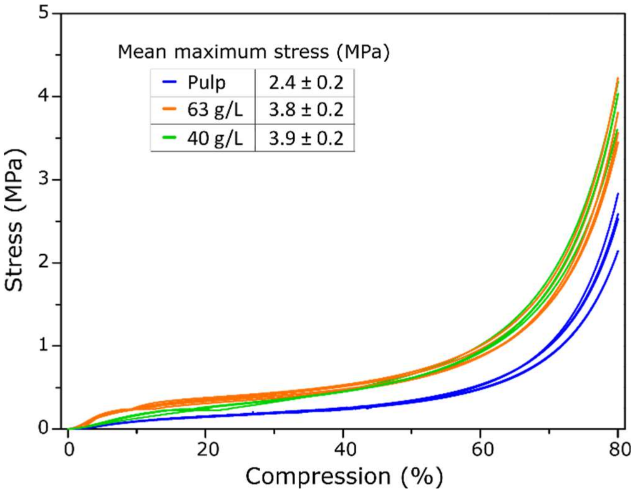

Figure 6. Stress-strain diagrams for compression of cellulose foams prepared with non-hydrolyzed fibers at 63 g/L concentration (blue) and foams of hydrolyzed fibers at concentrations 63 g/L (orange) and 40 g/L (green).

Young modulus is not usually reported for foams of cellulose fibers, and the parameters used to describe compressive mechanical properties in literature are bending strength (stress required to cause 50% of deformation)[8] and pressure at an specific deformation.[12] Mean bending strength values of cellulose foams of non-hydrolyzed fibers and hydrolyzed fibers at 63 g/L and 40 g/L are: (331 ± 9), (634 ± 32) and (625 ± 16) kPa, respectively. Isomoisio and collaborators reported a stress of 75 kPa to compress to 50% a foam of eucalyptus fibers. Although this material has an additive for mechanical improvement (anionic starch),[8] the stress required to deform it is one order of magnitude lower than the values presented herewith. At 80% of compression, maximum stress achieved was (3.9 ± 0.2) MPa, for foams prepared with hydrolyzed fibers at 40 g/L. Under the same condition (80% of compression), a stress of 8 kPa was registered for a foam prepared with eucalyptus fibers and SDS.[12]

Table 1. Mechanical properties of lightweight materials evaluated by compressive testing.

Material	Density (g/cm^3)	Young modulus (MPa)	Specific Young modulus ($MPa\ cm^3 g^{-1}$)	Energy absorption (kJ/m^3)
pulp fiber foam	0.161 (0.002)	1.2 (0.1)	7.1 (0.1)	194 (6)[a]
hydrolyzed fiber foam 63 g/L	0.178 (0.007)	3.9 (0.3)	24 (2)	377 (20)[a]
hydrolyzed fiber foam 40 g/L	0.151 (0.004)	1.9 (0.5)	12 (3)	354 (5)[a]
CNF, freeze-dried[33]	0.0081	0.05	6.7	-
CNF, tert-butanol freeze-dried[6]	0.105	2.8 (0.1)	27	720 (20)[a]
CNF, critical point dried[5]	0.03	0.9	29	75[a]
Crosslinked CNF/octylamine, oven dried[18]	0.02	1.1 (0.3)	55	70 (8)[a]
CNF/clay, freeze-dried[34]	0.00037	3.8 (0.2)	10270	46 (3)[b]
PU foam (high molecular weight)[35]	0.077	0.01463	~1.9	-
PU foam (low molecular weight)[35]	0.068	13.056	192	-

Values in parentheses denote standard deviations. Energy absorption values obtained for compressive strain between: 0 and 70%,[a] and 0 and 60%.[b]

Mechanical energy absorption is important for applications such as protective packaging and automotive components,[36] where shock absorption is required to prevent failure or injury.[36] At 70% of compression (Table 1), foams of non-hydrolyzed fibers presented an energy absorption of (194 ± 6) kJ/m^3, while foams of hydrolyzed fibers at 63 and 40 g/L concentrations presented an energy absorption of (377 ± 20) and (354 ± 5) kJ/m^3, respectively. These values are much higher than typical energy absorption reported for CNF aerogels (*ca.* 70 kJ/m^3).[5,18] Superior energy absorption capability can be achieved (720 kJ/m^3), but in aerogels prepared with CNF, followed by solvent exchange to tert-butanol and freeze-drying.[6]

3.4. Outlook and perspectives

Cellulose foams and aerogels have been pointed out in the literature as promising materials for several applications, such as effluent treatment, separations, extractions, catalysis, energy storage, packinging, and sound insulation. However, the techniques that are currently available to produce those porous matrices present high energy and time consumption and may also demand additives. In this work, a new method to prepare cellulose foams is proposed using simple and scalable steps, without the need of additives.

Prior cellulose preparation was carried out using hydrolysis under milder conditions, as compared to methods for CNC isolation. Besides, an additional advantage of this hydrolysis is that the process yield is relatively high (86.0 ± 0.9)%. At typical reaction conditions to obtain CNC (H_2SO_4 64% (w/w), 45°C, 1 h), hydrolysis yields are 43.5% and 39% for cotton and eucalyptus pulp, respectively.[37] Though both CNF and CNC can be found commercially available in some countries, it is relevant to find ways to achieve a more efficient use of the cellulose sources.

Also, regarding the experimental difficulties above-cited related to the production of aerogels and foams of cellulose nanoparticles, it is important to take into account if breaking the structure of cellulose fibers to obtain nanoparticles before preparing foams is really necessary for some applications. In some cases, the cellulose matrix well-organized structure, formed by crystalline and amorphous regions, could be suitable as it is, eliminating the necessity of steps such as TEMPO oxidation, mechanical shearing, long-drying steps, solvent exchange, *etc.* The simple method presented in this work exemplified a route to obtain lightweight foams, with very high shock-absorbing properties. Although these foams do not present densities as low as it can be obtained in the lightest CNC and CNF aerogels, they have an excellent balance between lightness and mechanical properties.

CONCLUSIONS

Lightweight materials of cellulose with apparent density as low as 0.15 g/cm^3 can be easily prepared from aqueous dispersions of fibers partially hydrolyzed by sulfuric acid. This procedure does not require the use of additives or special stirring equipment and the drying step is carried out in a conventional oven. Micro-CT and FESEM morphological analysis showed a random distribution of fibers throughout the matrix with a homogeneous distribution of porous. Fiber hydrolysis is important to obtain this random and porous structure, since it acts disaggregating the original fiber bundles present in eucalyptus pulp and also imparting electrostatic charges to the fiber surface that will contribute to maintain an open structure during drying. The mechanical performance under compression in the foams prepared with hydrolyzed fibers at 63 and 40 g/L showed great energy absorption (377 ± 20 and 354 ± 5 kJ/m^3, respectively), indicating the possible applicability of these renewable materials to replace polymeric foams derived from fossil fuels in packaging and other applications requiring mechanical resistance.

ASSOCIATED CONTENT

X-ray fluorescence analysis of fibers and details of HCl hydrolysis of fibers. This material is available free of charge via the Internet at http://pubs.acs.org.

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Author Contributions

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

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