

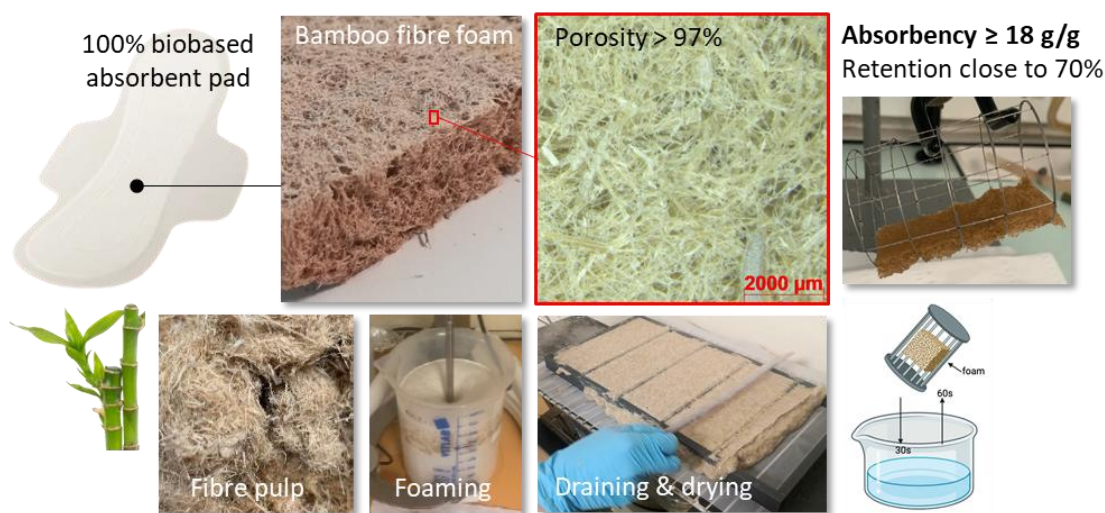
Liquid Absorbent Bamboo Fiber Foams: Towards 100% Ligno-cellulosic Menstrual Absorbent Pads

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GRAPHICAL ABSTRACT



Liquid Absorbent Bamboo Fiber Foams: Towards 100% Ligno-cellulosic Menstrual Absorbent Pads

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Traditional menstrual absorbent pads typically combine cellulose fiber fluff pulp with non-biodegradable, petroleum-based superabsorbent polymers (SAPs). To eliminate the need for SAPs, this study explores foaming as a method to create a highly porous lignocellulosic fiber network capable of storing large amounts of fluid. Bamboo fibers were chosen due to their high lignin content, which is expected to help maintain the structural integrity of the porous network during liquid absorption and preventing collapse compared to 100% cellulose fibers. The bamboo fiber foams demonstrated remarkable porosity and superior absorbency compared to commercial pads, but they exhibited lower water retention when subjected to compression. Refining the fibers and incorporating microfibrillated cellulose offer promising opportunities to enhance water retention.

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Keywords: Absorbency capacity; Lignocellulosic fibers; Foaming process; Highly porous fiber network; Menstrual napkins

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INTRODUCTION

There is a growing concern about the environmental impact of disposable menstrual hygiene products, as they are made from non-biodegradable materials, constituting 50% of the product's weight. Such items contribute to plastic pollution when discarded. This poses significant threats to ecosystems. This issue is closely linked to socio-economic problems, particularly in low- and middle-income countries, where limited access to affordable and safe menstrual hygiene products can have adverse health and social consequences for women and girls (Panjwani *et al.* 2023). The scope of this research aims to develop entirely bio-based highly absorbent materials using straightforward processes, with the objective of achieving sustainable and locally-produced menstrual pads that match the performance of commercial counterparts in terms of liquid absorbency and retention.

Commercial menstrual pads consist of multiple layers designed for absorption and comfort. The absorbent core, responsible for the absorption of menstrual fluid, is typically composed of a fabric-like material consisting of fluff pulp combined with superabsorbent polymers (SAPs), such as sodium polyacrylate and its derivatives. Fluff pulp is created by disassembling dense wood or cotton fiber pulp mats into individual fibers through gentle processes like defibration and air-induced separation (Askling *et al.* 1998).

In the pursuit of replacing non-biodegradable petroleum-based SAPs, researchers are exploring natural polysaccharides such as cellulose, starch, and chitosan as eco-friendly alternatives (Hubbe *et al.* 2013; Luo *et al.* 2019; Reshma *et al.* 2020; Darwesh *et al.* 2023). Nevertheless, their sorption capacity remains lower than that of conventional SAPs, and

these developments continue to be too expensive to meet the criterion of creating affordable products.

One potential solution is to enhance the capacity of the fluff pulp fabric. Fluff pulp, owing to its high porosity, typically ranging from 80% to 90%, can absorb up to 4 to 6 times its own weight of liquid. To achieve greater performance, one could optimize the fluffing process and engineer an ultra-porous structure, with porosity exceeding 95%, akin to recent advancements demonstrated with sisal fibers (Molina *et al.* 2023). Another viable and straightforward method to create such an ultra-porous network entails processing the fiber pulp into a fiber slurry, subsequently subjecting it to a foaming process. This process incorporates air into the slurry through mechanical agitation and the introduction of foaming agents. The foamed pulp is shaped into the desired form, such as sheets or blocks, and is subsequently consolidated by drying (Hjelt *et al.* 2022).

The objective of this study was to evaluate the potential of foaming as a method for creating a highly porous lignocellulosic fiber network capable of storing fluid within its porous structure, with the goal of matching the absorbency performance of commercial absorbent pads without the use of SAPs. To achieve this, bamboo fibers were chosen due to their elevated lignin content (Sillard *et al.* 2023). This choice offered distinct advantages, since these fibers maintain some of their stiffness when exposed to water. This inherent property was expected to maintain the structural integrity of the highly porous fiber network during liquid absorption, preventing the network's collapse and allowing for maximum utilization of the foam-generated porosity for liquid storage.

Furthermore, the inherent antimicrobial properties of lignin could provide an additional benefit, especially in ensuring hygiene and health security in applications such as sanitary napkins (Zahed *et al.* 2018). For instance, diapers made from pure bamboo exhibit significantly enhanced antibacterial effectiveness compared to cotton fabrics (Shanmugasundaram *et al.* 2011).

However, lignin content might affect fiber-fiber bonding during consolidation and potentially limit liquid retention. To address this, refining the bamboo pulp and adding microfibrillated cellulose (MFC) were considered. The refining process, involving compression and shear forces, induces changes in fibers: removal of primary wall, delamination and swelling of the secondary wall (internal fibrillation) that improve fiber flexibility, and peeling off the fibrils from the fiber surface (external fibrillation). Such peeling improves the potential bonding area and produce fines (fragments with size less than 76 μm) that may act as bonding bridges at inter-fibre contacts (Gharehkhani *et al.* 2015). Micro-fibrillated cellulose (MFC) addition was found to act in the same way in low density fibre media (Morais *et al.* 2021).

Four pulp types were examined: raw bamboo pulp, obtained through a thermomechanical process to meet criteria of lignin content and suitability for small-scale production; bamboo pulp refined using single disc refiner pilot system; unrefined bamboo pulp with 2.5% MFC; and linter cotton pulp for comparison with 100% cellulose fibers. The subsequent sections provide details of the processes involved, including foaming and characterization methods. Morphological properties of each fiber type are then presented. Liquid foam properties, such as air content and drying shrinkage, are detailed and compared with those of fully cellulosic foams. Finally, structural properties of solid foams, along with liquid absorbency capacity and retention, are characterized and compared with commercial absorbing pads.

EXPERIMENTAL

Materials

Bamboo strips

The central internodal segment of a mature bamboo specimen (*Dendrocalamus elegans*) aged over 3 years, sourced from Mok Far Mont Ngo Resort in Chiang Mai, Thailand, served as the raw material. The bamboo underwent an initial cutting process to produce small strips measuring 1 mm × 5 mm × 60 mm. Cut strips were air-dried under ambient conditions for a period of 7 days and subsequently stored in plastic bags.

Cotton pulp and commercial absorbing pad

A commercial cotton linter fiber pulp (Celsur, CS 21 DHS) was used for comparative purposes in foam production and characterization. Additionally, an absorbent pad extracted from a commercial product (Always Discrete, Procter & Gamble), was employed as a commercial reference in conducting tests for liquid absorbency capacity.

Micro-fibrillated cellulose

A 1 wt% CNFs suspension from the University of Maine (Orono, ME, USA) was used. They were made using bleached softwood kraft pulp, after enzyme pretreatment (US Patent, US 20170073893A1).

Methods

Thermo-mechanical process for individual bamboo fibers

Bamboo strips underwent a thermo-mechanical process (TMP) to produce fiber pulp. They were steamed 10 min at 2 bars, then subjected to mechanical treatment using a disk refiner under 3 bars of pressure with reference plates 12SA001 at 3000 rpm. This apparatus generates mechanical forces, including grinding and compression, to disintegrate the wood chips into individual fibers, as well as fiber fragments or bundles of fibers that may remain. The heat generated in this process served to soften the lignin, facilitating the separation of fibers.

Mechanical refining

A portion of fibers underwent refining using a single disc refiner pilot system described in (Lecourt *et al.* 2011) until a fibrillation degree of 60 °SR, as measured with a Shopper Riegler (°SR) tester (Paper Testing Association, France) following the ISO 5267-1 standard.

Preparation of fiber-based foams

A fiber suspension at 6% consistency underwent mixing using a Turbotest® mixer (VMI, France) equipped with a deflocculating blade. The suspension was agitated for 3 minutes at a rotational speed of 2000 rotations per minute (rpm) to attain a homogeneous state. A non-ionic polyglucoside surfactant, GlucoPON UP 215 (BASF, Germany), in concentration 1 g/L, around twice more than the micellar critical concentration, was then added to the fiber suspension. The mixture was agitated at a constant rotation speed of 2000 rpm for 10 minutes. The air content, referring to the air introduced into the fiber suspension through stirring and stabilized by the surfactant, was assessed by measuring the initial volume of the suspension ($V_{\text{initial}}=1.5\text{ l}$) and the final volume (V_{final}) of the liquid foam in the beaker, following Eq. 1:

$$\text{Air content (\%)} = 1 - \frac{V_{\text{initial}}}{V_{\text{final}}} * 100 \quad (1)$$

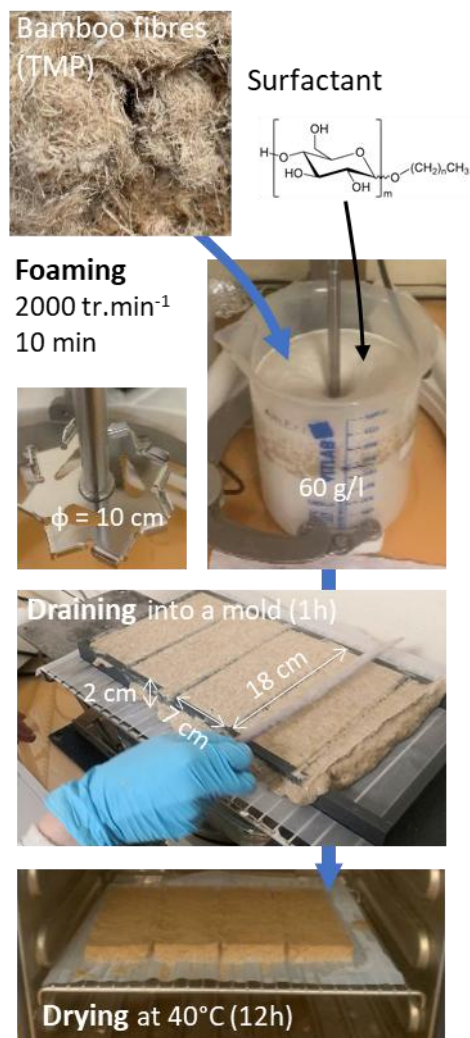


Fig. 1. Various stages of preparation of fiber-based foam

In this study, it was decided to maintain fixed processing conditions and not reach a specific air content level to enable a comparison of fibers under identical processing conditions. The resulting liquid foams were transferred to rectangular molds. They were placed on a 100-micron filtering screen for one hour to drain off the water. Subsequently, the mold was removed, and the foams were transferred to an oven for 12 hours at a temperature of 40 °C for drying. The dimensions of the foams were systematically measured after drainage and after drying. The shrinkage in volume was calculated after each step considering the mold dimensions at the initial volume of the liquid foam. It should be noted that the uncertainty in the thickness measurement was significant, as it was determined using a caliper that only touches the foam without applying any pressure. The various steps are illustrated in Fig. 1.

Morphological analyses

A MorFi LB-01 fiber analyzer (Techpap, France) was used to assess the morphological properties of the fibrous elements. The fiber / fine limit was set to 200 µm

in length. The resolution of the MorFi is estimated to be around 5 μm . Two analyses were done on each sample, and average fiber length (μm), fiber width (μm) and fine content (% in area) were determined.

Optical microscopy

The foam structures were observed by using an optical microscope (Discovery V20, Zeiss, Germany) under reflected light at a magnification of 32x.

Calculation of porosity

The dry foam density was assessed by measuring the mass and thickness of the produced sample, and porosity ε was calculated following the Eq. 2. This calculation considered both the foam density ρ_{foam} and the density of the fiber wall. The latter calculation considered the proportions of cellulose, lignin, and hemicellulose in the fiber wall denoted $\rho_{\text{cell+lign+hemi}}$.

$$\varepsilon = 1 - \frac{\rho_{\text{foam}}}{\rho_{\text{cell+lign+hemi}}} \quad (2)$$

Measurement of liquid absorbency capacity (LAC)

The absorbency tests were conducted by drawing inspiration from ISO Standard 12625-8, which is designed for measuring the absorbency capacity of cellulosic fiber networks, such as tissue papers. The method consisted in putting a foam sample into a standardized immersion basket (see Fig. 2). The sample was weighed together with the basket. The basket was dropped into a water basin, which was filled with distilled water. There, the sample was soaked for 30 seconds. Then the sample was lifted above the water for 1 min at a defined angle of 30° to allow the water to drain. After this, the sample and the basket were weighted again. The authors defined the liquid absorbency capacity (LAC) as the ratio of the wet mass (mass_{wet}) and the dry mass (mass_{dry}) of the sample.

$$\text{LAC (g/g)} = \frac{\text{mass}_{\text{wet}}}{\text{mass}_{\text{dry}}} \quad (3)$$

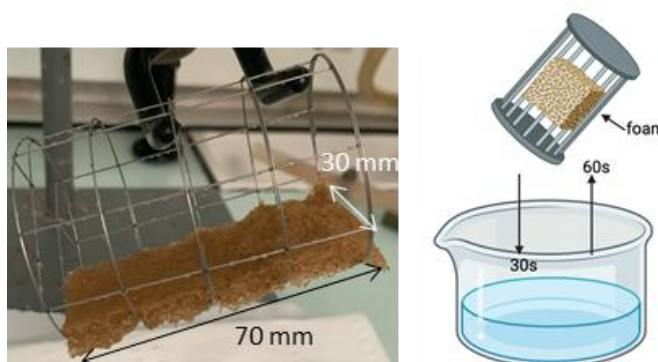


Fig. 2. Absorbency test adapting the basket method (ISO 12625-8) for determining the LAC of solid foams

To assess the sample's retention ability, the wet sample was subjected to a 5 kPa load for 30 seconds and then weighed. Retention was determined by calculating the ratio of the sample's weight after applying the pressure to its weight before applying the pressure.

$$\text{Retention (\%)} = \frac{\text{mass}_{\text{wet after 5 kPa loading}}}{\text{mass}_{\text{wet}}} \quad (4)$$

RESULTS AND DISCUSSION

Morphological properties of fibers

Table 1 presents the chemical composition and mean morphological properties of the various fibers used in this study. Bamboo fibers exhibit a composition of 50% of cellulose and over 30% of lignin, while cotton fibers are primarily composed of cellulose. The TMP process produced short fibers through mechanical action, comparable in mean length and width to cotton fibers. However, analysis of the distributions depicted in Fig. 3 reveals the presence of certain large elements, such as fiber bundles, in the bamboo pulp, which exert an influence on the mean values of length and width. Indeed, well-individualized bamboo fibers tend to be thinner and slightly shorter than cotton fibers. Refining the bamboo fibers resulted in a reduction in fiber length, primarily through fiber cutting. As expected, the fines content was high for bamboo, at 26.9%, and notably lower for cotton (4.9%). This difference was attributed to the thermomechanical pulping (TMP) process, which caused the detachment of fiber wall fragments, transforming them into fine elements in the fiber pulp. Subsequent refining further increased the fines content due to fiber cutting and the peeling off of the fiber wall surface.

Table 1. Chemical Compositions and Morphological Properties of Different Types of Fiber Pulp. Properties of the Liquid Foams Obtained from the Fiber Pulps. Structural Properties and Absorbency Performance of the Solid Foams.

Pulp Properties	Ref	Cotton	Bamboo	Refined	+2.5% MFC
Lignins content (%)		<1	32		
Hemicelluloses content (%)		<2	19		
Fiber mean length (μm)		689±5	744±6	450±1	
Fiber mean width (μm)		20.3±0.1	21.8±0.2	20.4±0.1	
Fines content (in area, %)		4.9±1.4	26.9±1.9	44.8±2.9	
Liquid Foam Properties					
Air content (%)		≈35	≈75	≈45	≈60
Shrinkage in volume (%)		26	14	20	22
Solid Foam Properties					
Density (kg/m ³)		73.5±2.9	28.5±1.1	47.0±1.0	35.8±1.2
Porosity (%)		95.0	98.1	96.9	97.6
LAC (g/g)	16.9±0.3	12.4±0.3	22.5 ±1.1	17.7 ±0.7	18.8 ±0.5
Retention after load (%)	100	69	50	73	68

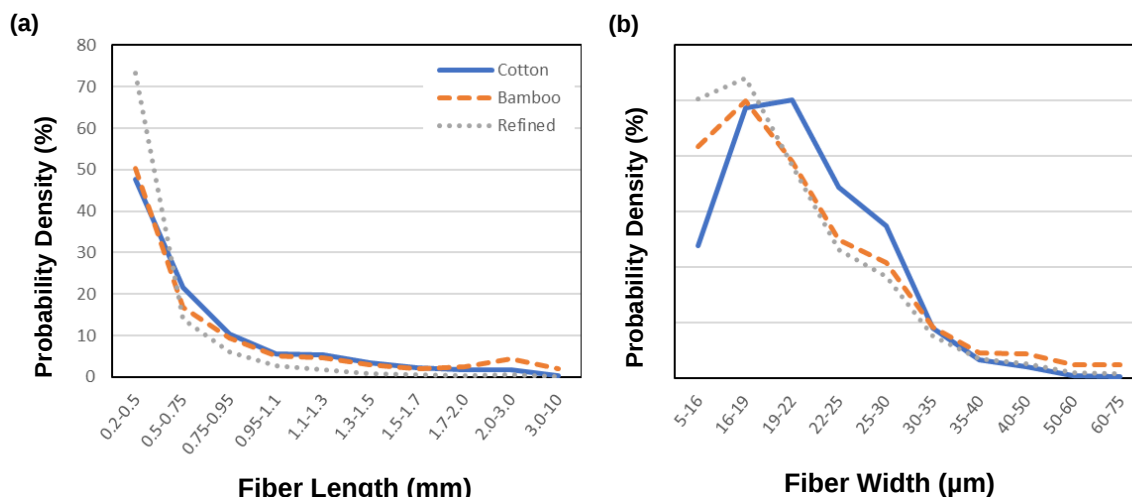


Fig. 3. Distributions in (a) length and (b) width of cotton, bamboo and refined bamboo fibers

Liquid Foam Properties

The created liquid foams were compared in terms of their air content and shrinkage during draining and drying. Both factors are expected to influence the porosity of the solid foam: higher air content should lead to increased porosity, while shrinkage is likely to reduce it. Since absorbency capacity is expected to improve with greater porosity, achieving high absorbency requires maximizing air content and minimizing shrinkage to produce a highly porous foam.

Air content

The air content in liquid foam was found to vary among different types of fibers under identical foaming conditions, being lowest for cotton fibers and highest for bamboo fibers, as shown in Table 1. Notably, the air content was significantly reduced following the refining of bamboo fiber pulp. Additionally, the inclusion of MFC led to a decrease, albeit to a lesser extent. While drawing definitive conclusions from these trends is challenging, hypotheses can be formulated. The presence of lignin and fines in bamboo fiber pulp plays a key role in the stabilization of bubbles. In the liquid foam of bamboo fibers, bubbles may adhere to fibers and fines since bubbles exhibit an affinity for lignin-rich surfaces (Hjelt *et al.* 2022). This adhesion may block air diffusion, thereby limiting bubble coalescence and stabilizing the foam (Ketola *et al.* 2022). Conversely, with 100% cellulose fibers such as cotton, coalescence may occur more readily, resulting in lower stabilization (Al-Qararah *et al.* 2015). This observation offers a plausible explanation for the lower air content in cotton. MFCs, being 100% cellulosic, might stabilize bubbles through the Pickering effect due to their small shape, but their chemical composition may limit adhesion to bubbles, reducing stabilization. Refining generates fines and is known to degrade the fiber wall, freeing microfibrils that are richer in cellulose. This process might also limit adhesion and stabilization.

Shrinkage when draining and drying

The consolidation of fiber walls between bubbles is believed to occur through mechanisms similar to those identified in paper when water is removed by draining and drying. Capillary forces, generated by water bridges, serve as the primary driving forces that bring fiber surfaces into close proximity, promoting fiber bonding through various low-energy interaction mechanisms (Hirn and Schennach 2015; Wohler *et al.* 2021).

Lignin-rich fibers, known for being less flexible in the wet state compared to 100% cellulose fibers, result in lower bonding due to a smaller contact area at the molecular scale. Table 1 presents the volume shrinkage observed after the draining and drying phases for each type of foam. Cotton showed the highest shrinkage, resulting in a volume reduction of 26%. In contrast, bamboo fiber foams exhibited significantly lower shrinkage, measured at 14%. Refined bamboo fiber foams showed higher shrinkage, reaching 20%. When adding MFC to bamboo pulp, the shrinkage tended to be higher (22%). The differences in shrinkage are primarily assumed to result from the transfer of drying shrinkage deformation among fibers. This transfer depends on the degree of bonding between fibers. The greater shrinkage observed during the drying phase of cotton may be attributed to the high bonding resulting from numerous contacts between fibers, due to low air content, and a 100% cellulose surface. Furthermore, refined bamboo pulp and the addition of MFC are assumed to enhance bonding, facilitating the transfer of shrinkage between fibers. The refining process improves the wet flexibility of fibers, generating fines and detached fibrils that increase the contact area between fibers and facilitate interactions. MFC acts similarly, forming bonding bridges between fibers.

Solid Foams Properties

Table 1 depicts the density and porosity of solid foams with the different fiber pulps. The cotton fiber foam exhibited the highest density and the lowest porosity at 95%, as expected due to its lower air content and higher shrinkage during the draining and drying process compared to the other foams. In contrast, the foams containing bamboo fibers exhibited a very high porosity of 98.1%. The porosity was reduced to 97.6% with the introduction of 2.5% MFC and further decreased to 96.9% with refining. These observations align with the observations on air content and liquid foam shrinkage.

Observations of the fiber networks in Fig. 4 show that the bamboo fiber foam exhibited an open structure and a wide fiber size distribution from individual fibers to bundles of fibers, as suggested by morphological measurements (Fig. 3). In contrast, the refined fiber foam displayed a closer structure with finer elements, likely fibrils ripped off from the fiber wall and detached fines.

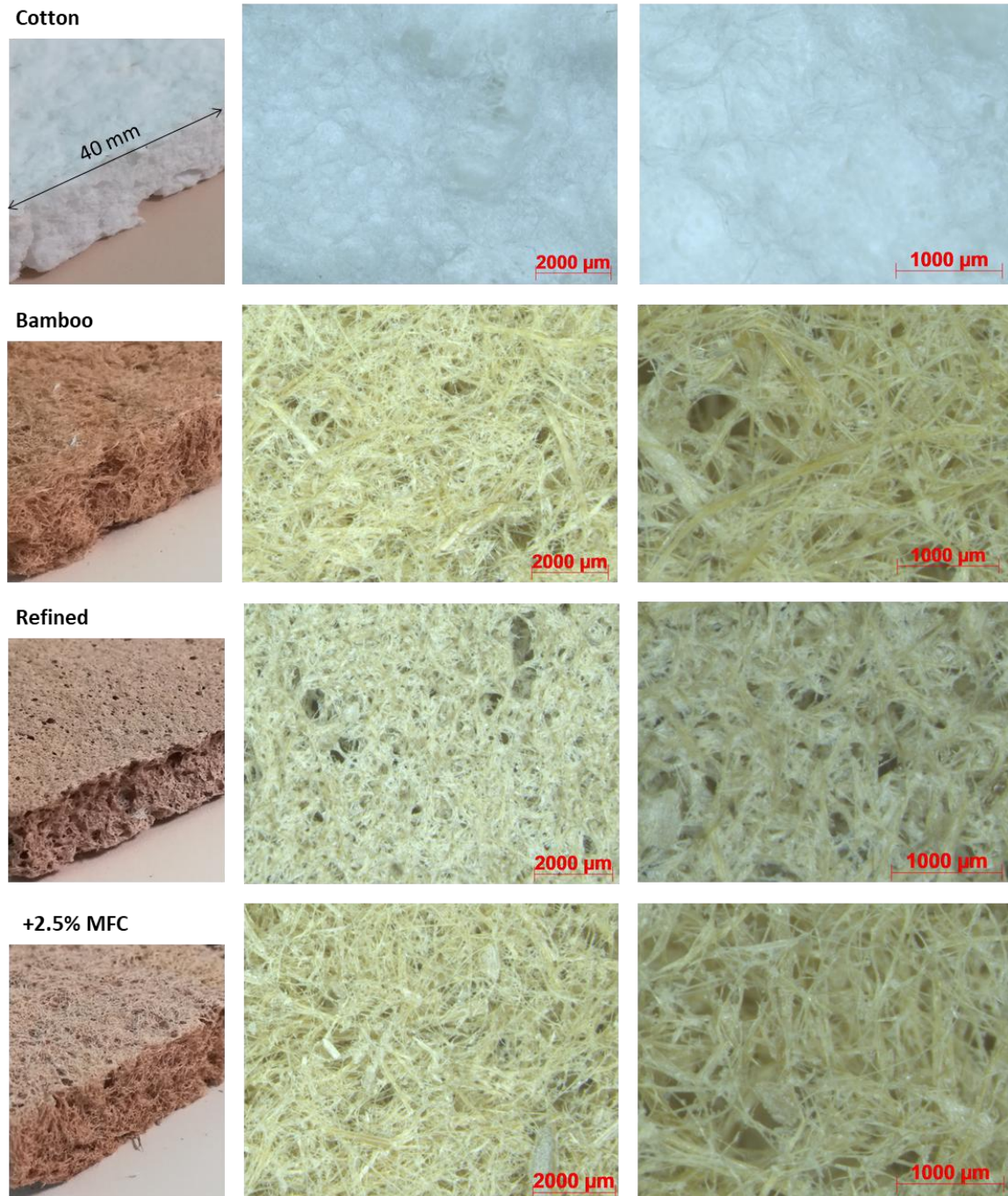


Fig. 4. Pictures of solid foams and surface structures imaged by optical microscopy

These elements form bridges between fibers contributing to increasing shrinkage and densifying the fiber network. It was assumed that the impact of MFC on bonding is less observable as it acted at a smaller scale, developing bonding bridges between the fiber walls at fiber-fiber contacts (Hobisch *et al.* 2021). For cotton fibers, while the network was evidently denser, the lack of contrasts prevents relevant observations regarding bonding and pore size.

These differences in density and bonding resulted in variations in the stiffness of the solid foams. Qualitatively, bamboo foam appeared more flexible than cotton foam.

However, refined bamboo samples seemed stiffer than standard bamboo samples. Excessive stiffness could pose a challenge in terms of overall comfort and performance in the developed napkins. This should be quantified in a future study using samples with weight and dimensions equivalent to commercial pads.

Liquid Absorbency Capacity of Solid Foams

The liquid absorbency capacity (*LAC*) of the various foam types are presented in Table 1. Among them, the bamboo fiber foam demonstrated the highest *LAC* when tested with water, reaching 22.5 g/g, surpassing the capacity of the commercial reference absorbing pad limited to 16.6 g/g. Both refined fiber foam and fiber with MFC foam exhibited slightly higher values compared to the commercial reference pad. In contrast, the cotton foam displayed the lowest *LAC* value of 12.4 g/g.

LAC values appeared to correlate with porosity: higher porosity corresponded to greater *LAC* (Fig. 5). To further explore this relationship, the theoretical liquid storage capacity of the porous structure (*LSC*) was calculated under the assumption that all pores were completely filled with water, and fibers remained insensitive to water (*i.e.*, they did not swell and remained stiff). This calculation was performed using Eq. 4, where ε represents porosity, and ρ_{water} and $\rho_{fibre\ wall}$ are the water and fiber wall densities respectively (Viguié *et al.* 2022).

$$LSC = \frac{\rho_{water}}{\rho_{fibre\ wall}} \left(\frac{\varepsilon}{1-\varepsilon} \right) \quad (5)$$

The theoretical curve is shown in Fig. 5. For cotton and refined bamboo fibers, the *LAC* values were closely aligned with the *LSC* curve. However, for unrefined bamboo foam, the *LAC* deviated. This discrepancy suggests that, at high levels of porosity, the structure tended to collapse in the wet state, thereby limiting absorbency capacity.

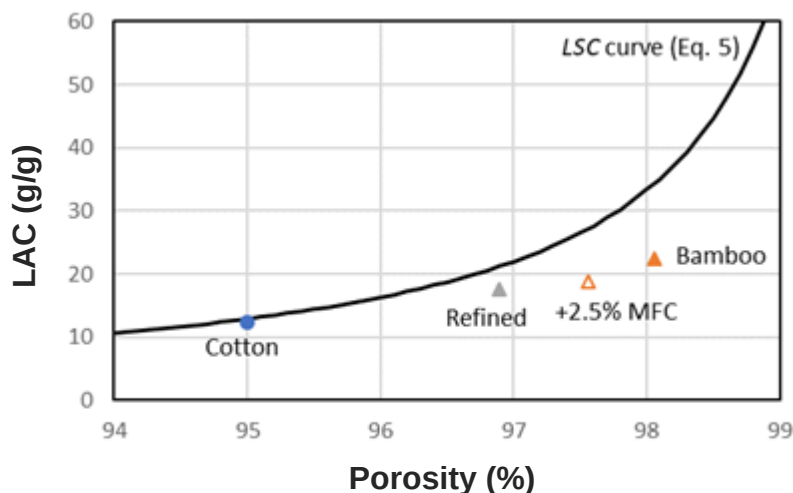


Fig. 5. Liquid absorbency capacity (*LAC*) as a function of porosity for all types of foam and evolution of theoretical liquid storage capacity (*LSC*) of the porous structure according to Eq. 5

It is also worth noting that the surfactant may negatively affect absorbency performance by reducing water's surface tension and, consequently, the capillary driving force (Ko *et al.* 2016). However, it is difficult to determine the exact impact of this factor on the final absorbency capacity. This influence could also depend on the adsorption of the

surfactant onto the fibers, which is affected by their specific surface area, especially when modified by refining or the presence of MFC.

Retention values for water in the pad are presented in Table 1. Bamboo fibers exhibited the lowest retention level (50%). In contrast, retention was better for refined fibers (73%) and fibers with MFC (68%). The improved retention could result from better mechanical resistance to compression in the wet state, expected from the more bonded fiber networks of refined bamboo or with 2.5% MFC. The improved retention of the refined pulp could be also attributed to the smaller pore sizes, allowing for stronger water entrapment in the pores due to capillary forces. In the case of MFC, their high hydrophilicity and ability to form a gel even at very low concentration in water (1 to 3%), could also explain the enhanced retention behavior. This presents promising prospects for the development of bio-based and biodegradable liquid-absorbing pads, knowing that there is also potential for enhanced water retention by incorporating bio-based retention agents such as alginate or carboxymethyl cellulose (CMC), or through chemical modifications of fibers (Hubbe *et al.* 2013).

CONCLUSIONS

The potential of foaming bamboo fibers was explored as a method to create highly liquid-absorbent materials, aiming for a sustainable and affordable alternative to conventional commercial menstrual pads.

1. Under identical foaming conditions, bamboo fiber solid foams exhibited remarkable porosity ($\approx 98\%$), surpassing fully cellulose foams made with cotton linter fibers (95%). This result was attributed to the high amount of lignin in thermomechanically processed bamboo fibers, leading to higher air content in the liquid foam and lower shrinkage at draining and drying compared to cotton fibers.
2. Finally, bamboo fiber foams exhibited a liquid absorbency capacity (LAC) of 22.5 g/g when tested with water, surpassing a commercial absorbing pad limited to 16.6 g/g. In contrast, cotton foams registered the lowest value, highlighting a direct correlation between porosity and LAC. Despite this, bamboo fibers demonstrated the lowest water retention (50%) after 5 kPa compression. However, refined bamboo fibers and fibers with MFC exhibited a more favorable retention (73% and 68%, respectively).
3. The present findings indicate promising prospects for developing 100% bio-based liquid-absorbing pads. However, further work is needed to achieve this goal. The absorbency of 0.9% saline solution and synthetic blood should be examined. Bio-based veils that can be integrated with the absorbent pad to create a fully bio-based napkin product need to be developed. Additionally, the stiffness of the final product must be assessed and compared with commercial products of similar weight. Finally, the economic benefits of this approach, once implemented on a large scale, should be evaluated.

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