



## A unique microstructure of the fiber networks deposited from foam–fiber suspensions



Ahmad M. Al-Qararah<sup>a</sup>, Axel Ekman<sup>b</sup>, Tuomo Hjelt<sup>a</sup>, Jukka A. Ketoja<sup>a,\*</sup>, Harri Kiiskinen<sup>a</sup>, Antti Koponen<sup>a</sup>, Jussi Timonen<sup>b</sup>

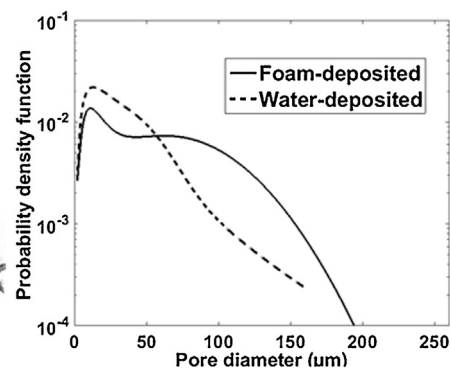
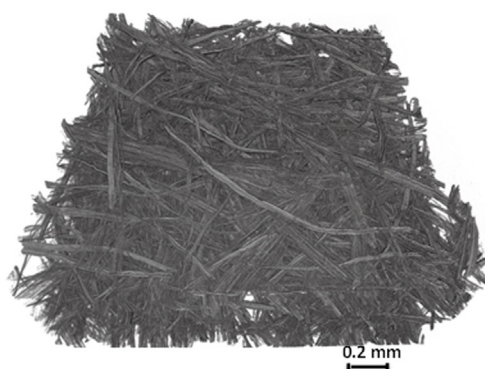
<sup>a</sup> VTT Technical Research Centre of Finland Ltd., P.O. Box 1000, FI-02044, VTT, Finland

<sup>b</sup> University of Jyväskylä, Department of Physics and Nanoscience Center, P. O. Box 35, FI-40014, University of Jyväskylä, Finland

### HIGHLIGHTS

- Foam and water deposited porous fiber structures differ at equal sheet density.
- Bubble-size distribution of the foam affects the mean pore size.
- The effect of bubble size on the porous structure is strongest for stiff fibers.
- Strength and other macroscopic sheet properties are affected by foam properties.

### GRAPHICAL ABSTRACT



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### ABSTRACT

Fiber networks can be formed using aqueous foam as the suspending medium. The mean bubble size of the foam affects the resulting pore-size distribution of the fiber network. The foam–fiber interactions cause in particular an increase in the proportion of large micropores of the network, in comparison with the fiber networks that result from traditional water forming at a similar material density. Experiments were carried out for two different types of cellulose fiber, and characterization of the resulting pore structure was based on X-ray microtomography of the resulting fiber networks. The unique pore structure obtained with foam forming was reflected in various macroscopic properties of the networks, which provides an intriguing opportunity to control the material properties of fiber networks via the selection of their forming.

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### 1. Introduction

Aqueous foam [1,2] consists of a close packing of gas bubbles in a continuous liquid phase. With added fibers, such a system provides an interesting suspending medium, which forms when removing the liquid phase, i.e., by a deposition process, a fiber network sim-

\* Corresponding author.

E-mail address: [jukka.ketoja@vtt.fi](mailto:jukka.ketoja@vtt.fi) (J.A. Ketoja).

ilar to those found in paper making. This provides excellent sheet homogeneity and enables the increase of solids content of the fibre furnish during the forming operation [3–6]. In addition to that, fibre materials with low density can be obtained [5–6]. The main drawback of this technology is a decrease in strength. To address the loss of strength in foam-formed sheets, it has been demonstrated that the strength can be regained by using micro-fibrillated cellulose (MFC) without losing the obtained bulky structure [7]. Foam-formed fibre structures have received an increasing interest recently as sustainable solutions for a wide range of material applications [5].

There is a long-lasting tradition in paper physics to investigate (when water is the liquid phase) the factors that affect the microstructure of the network [8,9]. These factors have ranged from properties of raw materials (e.g., fiber dimensions, fiber flexibility, their external fibrillation, and the fine-particle content of the furnish) to various process factors (e.g., concentration and rheology of the fiber suspension, fiber orientation, drainage, wet pressing, drying). In water forming, a great deal of effort has been put to creation of a turbulent suspension, before its deposition, which helps achieve a homogeneous fiber network in small (about a fiber length or a couple of millimetres) scale. Otherwise, the characteristics of the suspending medium as such has not been considered much in the paper-making literature.

This paper goes beyond the current paradigm (water forming) in paper making and analyzes the role of the suspending medium in the structure of the resulting fiber network. To this end, porous structures obtained by foam forming are compared with those obtained using a water suspension. It has been already suggested that the porous structure obtained with foam forming could differ significantly from that obtained with a traditional water forming [10,11]. However, the two forming methods have been previously compared only as separate technologies, i.e., without any attempt to avoid the contribution of the density of the final (or targeted) structure. In general, density of the resulting fiber network will depend on several factors, like the type of fiber, the vacuum level in drainage, and the load used in wet pressing [8]. In this work, the structures resulting from the two forming methods were compared for a similar final density of the network. This required in fact that no vacuum was applied in the water forming, whereas a normal 0.5 bar vacuum level was applied in the foam forming. Moreover, no wet pressing was applied in either process so as to leave a maximal trace of the foam-fiber interactions in the resulting network structure. Here the word “interaction” means any mutual influence between the two phases (foam and fibers) [12–16]. This influence may also be of geometric origin so that the bubble structure limits the orientation and movement of the fibers and vice versa.

Characterization of the structure of the fiber network has earlier been mainly based on (two-dimensional) visible-light microscopy or various indirect measurements. In recent years, however, modern three-dimensional (3D) imaging methods have opened a new avenue for the structural characterization of the network [17,18] together with a possibility to simulate material properties of the real network structure [19]. Here, characterization of the network structure was based on X-ray microtomography of the networks.

In addition to determining the pore-size distributions of these networks, we measured several of their macroscopic properties, and various properties (e.g., bubble size) of the suspending medium. Because of absence of wet pressing and vacuum dewatering (drainage) in the case of water forming, the densities of the samples remained in the range 100–300 kg/m<sup>3</sup>.

In Chapter 2 we explain the experimental forming procedures and characterization methods. Chapter 3 reports the differences in the pore-size distributions for the foam-formed and water-formed samples. The effects of the microscopic structural parameters on macroscopic physical properties are discussed in Chapter 4. In Chapter 5, some general conclusions are drawn based on the results.

## 2. Experimental

### 2.1. Fiber materials

Pre-refined bleached kraft pulp (Scots pine, coarseness 142 μg/m, freeness 607 ml) and chemi-thermo-mechanical pulp (CTMP; Norway spruce, coarseness 220 μg/m, freeness 570 ml) were obtained from Finnish paper mills. The coarseness gives the dry fibre mass per unit length, whereas the freeness of a pulp is an indication of its dewaterability. The average (length-weighted) length of the kraft fibers, as measured with a common fiber-analysis device (an optical measurement), was 2.2 mm [4]. The average length of the CTMP fibers was 1.6 mm [4]. For other fiber properties, we refer to typical values found in the literature for similar fiber materials [8]. The cross-sectional shape of the pre-refined wet kraft fibers was quite circular with an average diameter in the range 35–40 μm. For the CTMP fibers, with similar shape, the diameter was about 30 μm. Thus, the overall dimensions of the fibers in the two furnishes were quite similar. The main difference between the qualities of these two types of fiber was the bigger conformability (a higher level of their shear deformation seen as more bending) of the kraft fibers, which gave rise to a higher density of the resulting fiber network [8].

### 2.2. Preparation and characterization of the foam-fiber suspension

Pulp furnish (kraft pulp or CTMP) was diluted with distilled water to 0.33% consistency. Here, the term consistency denotes the mass fraction (or its percentage) of the solid and filterable materials in the suspension. The furnish was made such that three liters of a diluted furnish (10 g solids) was mixed with 0.60 g (kraft) or 0.90 g (CTMP) anionic surfactant (Sodium Dodecyl Sulphate (SDS), C<sub>12</sub>H<sub>25</sub>SO<sub>4</sub>Na). Thus, the ratio of added SDS was 0.20 g/l (kraft) or 0.30 g/l (CTMP). SDS was obtained from Sigma-Aldrich and its purity was 90%. A different amount of the foaming agent was needed in order to achieve a high enough air content of the respective foams. At 0.20 g/l, the air content of the CTMP foam remained at a low level (c.a. 60%).

A foam-fiber suspension was generated by axially agitated mixing in a cylindrical vessel (diameter 200 mm) with three impellers

**Table 1**

Mean bubble size,  $r_{32}$ , and air content,  $\varphi$ , for various rotation speeds of a pure foam (SDS concentration 0.3 g/l) and the foam-fiber suspensions studied. Air content was determined from the final foam volume  $V_f$  and the initial water amount  $V_w$ ,  $\varphi = (V_f - V_w)/V_f$ . The error in  $V_w$  was very small as the initial water amount was measured by weighing. Thus, the absolute error in the measurement of the air content remained on the low level  $\pm 0.004$  in all cases.

Rotation speed (RPM)	Pure foam		CTMP		Kraft	
	$r_{32}$ (μm)	$\varphi$	$r_{32}$ (μm)	$\varphi$	$r_{32}$ (μm)	$\varphi$
2000	95.6 ± 2.1	0.643	93.8 ± 3.5	0.655	99.3 ± 2.6	0.655
3500	72.5 ± 2.1	0.681	70.5 ± 2.0	0.688	56.6 ± 1.4	0.684
5000	61.2 ± 1.3	0.737	48.7 ± 1.0	0.709	48.8 ± 1.4	0.714
6900	39.2 ± 0.9	0.758	34.4 ± 0.8	0.639	39.6 ± 1.1	0.647

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