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RHEOLOGY OF MECHANO-ENZYMATICALLY FIBRILLATED CELLULOSE IN FIBER SUSPENSIONS AND FOAMS

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ABSTRACT

Cellulosic materials are increasingly explored as renewable building blocks for applications such as packaging, insulation, and construction. Conventional production of microfibrillated cellulose (MFC) is energy intensive and results in suspensions with strong gel-like behavior even at low solids contents.[1-3] High-consistency mechano-enzymatic fibrillation of cellulose (HC MFC, previously referred as HefCel) has emerged as a lower-energy alternative, enabling the production of MFC directly at high solid contents.[2, 4]

In this work, the rheological behavior of HC MFC is investigated over a range of concentrations in both fiber suspensions and fiber foams. Two grades of HC MFC, coarse and fine, are studied to elucidate the effects of fiber content and fibrillation degree on viscoelastic and flow properties. Commercial MFC is used as a reference material to benchmark the behavior of HC MFC against established systems.

The results show that HC MFC suspensions and foams exhibit a predominantly elastic response at small deformations over the entire range of fiber consistencies studied (2.5-15 wt% for suspensions, 2.5-7.5 wt% for foams). Rheological properties depend systematically on fiber content and fibrillation degree, reflecting differences in fiber network strength. Compared with commercial MFC, HC MFC shows much lower stiffness and therefore improved processability at high fiber consistencies. These findings support the potential of HC MFC in the production of sustainable, low-density fiber-based materials.

INTRODUCTION

Cellulosic materials have attracted significant attention as renewable and sustainable building blocks for a wide range of applications, including packaging, insulation, and construction. Among these, microfibrillated cellulose (MFC) has emerged as a versatile material due to its high surface area, tunable network structure, and ability to form strong, lightweight, and porous assemblies.[5-7] However, conventional routes for producing MFC rely primarily on intensive mechanical fibrillation processes, often preceded by limited chemical or enzymatic pretreatments. These methods are typically energy demanding and yield dilute suspensions that exhibit pronounced gel-like behavior even at low solids contents, which complicates handling, transport, and downstream processing.[3]

To address these limitations, alternative production strategies have been developed that improve both process efficiency and material functionality. In particular, high-consistency (HC) processing has gained interest to enhance fibrillation efficiency while reducing water usage.[8] In this context, consistency refers to the mass fraction of dry fibers in the system, typically expressed as a percentage relative to the total mass of the suspension or foam. At elevated solid contents, increased fiber–fiber friction during mechanical treatment promotes structural disruption of the fiber walls. When combined with enzymatic treatment, this effect is further amplified: the reduced water content enables enzymes to more effectively locate, recognize, and adsorb onto cellulose surfaces, thereby enhancing their catalytic activity.[8] As a result, high-consistency mechano-enzymatic pretreatment can facilitate more efficient fiber breakdown compared to purely mechanical or enzymatic approaches alone, enabling extensive fibrillation that has not been observed in conventional processes.

A key advantage of HC mechano-enzymatic fibrillation is the ability to produce MFC directly at high solids contents, typically in the range of 20–25%.[4, 8] This represents a significant departure from conventional dilute systems and introduces practical benefits such as reduced water handling, improved process economics, and enhanced feasibility for off-site production and long-distance transportation. Moreover, the resulting material exhibits a paste-like, non-gel character, making it easier to handle, store, and process in industrial applications. The process is also adaptable, allowing the production of different grades of HC MFC—such as coarse and fine fibrillated fiber materials—by tuning processing conditions and the degree of fibrillation. Additionally, the approach is compatible with a wide range of lignocellulosic feedstocks, including wood and non-wood pulps, recycled fibers, textile waste, and agricultural residues, further supporting its relevance within circular material systems.[2, 4, 8]

Beyond production, understanding the rheological behavior of fibrillated cellulose systems is essential for their practical utilization, as rheology governs processability, structural stability, and end-use performance in applications ranging from coating and forming to foam structuring and additive manufacturing.[9-11]

While the rheological properties of conventional MFC suspensions have been studied extensively, comparatively little is known about the behavior of HC MFC systems, particularly at high solids contents and in structured forms such as fiber foams. Fiber foams are of particular interest due to their potential to reduce drying energy requirements, as their lower water content and high porosity facilitate more energy-efficient processing compared to conventional suspension processing.[12, 13]

In this context, the present study investigates the rheological behavior of HC MFC across a range of concentrations in both fiber suspensions and fiber foams. Two grades of HC MFC — coarse and fine — are examined to elucidate the influence of fibrillation degree and fiber content on viscoelastic and flow properties. To provide a meaningful benchmark, a commercial MFC product is included as a reference material, enabling direct comparison between HC MFC and established systems at equivalent solid and air contents. Through this approach, the work aims to advance the fundamental understanding of HC MFC rheology and to support its further development as a sustainable platform for low-density, fiber-based material applications.

MATERIALS AND METHODS

High-consistency mechano-enzymatically fibrillated cellulose (HC MFC, previously referred to as HefCel) was produced from bleached softwood kraft pulp (BSKP) using a 7 L Winkworth sigma mixer (MZ7).[4]

For the production of the standard (coarse) HC MFC grade, the pulp was subjected to simultaneous mechanical mixing and enzymatic treatment at a solids content of 25%. An enzyme dosage of 4.5 mg per gram of dry pulp was applied. The treatment was carried out at pH 5 and temperatures of 50°C and 70°C under gentle mixing for a total duration of 5.5 h.

To obtain the fine HC MFC grade, the coarse material was further processed by high-pressure homogenization using a microfluidizer. This treatment was performed at a solids content of 10%, and the suspension was passed once through the microfluidizer at a pressure of 1800 bar.

The resulting HC MFC materials were used as produced for subsequent suspension preparation, foam fabrication, and rheological characterization.

The as-received consistencies of the materials were 17.5% for the coarse grade and approximately 10% for the fine grade. A commercial MFC material was used as a reference.

Fibre morphology was characterized using a Valmet FS5 fibre image analyzer to quantify key parameters such as fibre length, width, and fines content, as shown in **Table 1**. The measurements were conducted according to the standard operating procedure of the instrument to ensure reproducibility and comparability between samples.

TABLE 1: Fiber properties of the two high-consistency (HC) MFC types, coarse and fine grade.

	Coarse grade	Fine grade
Fiber length, mm (Tappi)*	0.205	0.200
Fiber width, µm	29.58	22.98
F1(I) 0.00-0.20 mm, %**	94.0	99.4
Fines, %	99.08	99.95

*Length-weighted average fiber length, Tappi T271 0.1-7 mm.
**represents the percentage of fibres, determined by image analysis, with lengths between 0.00 and 0.20 mm, corresponding to the fines fraction of the sample.

In addition, optical microscopy was employed to qualitatively assess fibre structure and degree of fibrillation (**Fig. 1**). To enhance image contrast and improve the visibility of fibrillar features, Congo red dye was applied as a staining agent prior to imaging.[4]

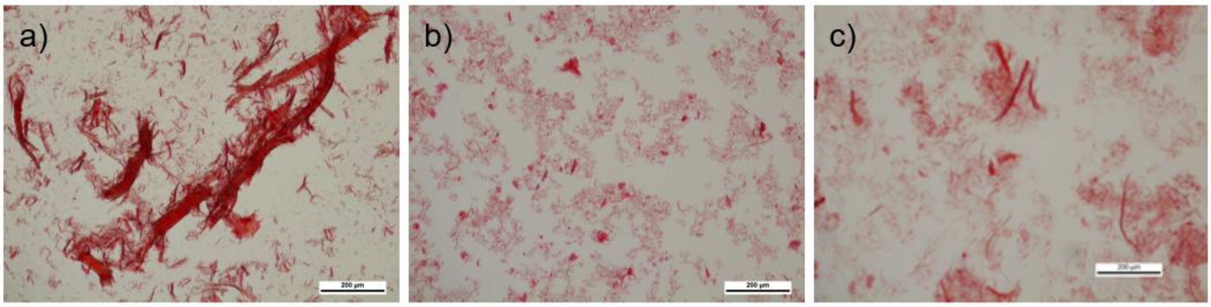

FIGURE 1: Microscope images of the two HC MFC grades, a) standard (coarse) HC MFC and b) microfluidized (fine) HC MFC, as well as c) commercial MFC reference at 10x magnifications.

Before the rheological measurements, the MFC suspensions were diluted to the given fiber consistency with deionized water and homogenized with different mixing procedures depending on the fiber consistency. The suspensions prepared from the two grades of HC MFC, namely a coarse standard grade and a fine fluidized grade, were homogenized using either syringe-to-syringe method or a Silverson mixer and commercially available MFC were homogenized with a Netzsch Shearmaster. The mixing vessel had a volume of approximately 600 mL and an inner diameter of 67 mm. Mixing was carried out at 800–1000 rpm for 15–20 minutes.

Fiber foams were prepared from both grades of HC MFC and from the commercial grade MFC. Prior to foaming, the materials were diluted to the desired working consistencies with deionised water. The coarse HC MFC suspensions were prepared at three different consistencies: 2.5, 5.0 and 7.5%, whereas the fine HC MFC and commercially available MFC reference suspensions were adjusted to 2.5%. Carboxymethyl cellulose (CMC; FinnFix 4000G) was incorporated at 3.5 m.% (based on dry fiber content). The diluted samples containing the CMC were activated using Silverson mixer at 4000 rpm for 5 minutes.

Sodium dodecyl sulfate (SDS) was added to all samples at a concentration of 0.4 g L^{-1}. For the commercially available MFC reference a higher SDS dosage of 0.8 g L^{-1} was required to reach the air content of around 70 ± 5%. Foaming was performed by mechanical mixing (Netzsch Shearmaster) using a high shear sawtooth impeller. Duration of mixing was 10-15 minutes at 2850-4000 rpm with a starting sample volume of 100 ml. The process parameters (mixing speed and time) were adjusted depending on the sample grade to obtain a target air content of 70 ± 5%. The resulting fiber foams were used directly for subsequent characterization.

It is worth noting that the mixing/homogenization procedure is likely to affect the structure and dispersion state of fiber suspensions, as well as the bubble size distribution in fiber foams. Therefore, the rheological properties of the investigated samples may vary depending on the mixing/homogenization method.

Rheological measurements were performed using a stress-controlled Discovery HR-2 rheometer (TA Instruments) with a wide-gap vane-in-cup geometry at 22°C, controlled by a Peltier system connected to a Julabo AWC 100 bath. A 3D-printed serrated cup was used to minimize wall slip at the wall of the cup.[14] The vane had a diameter of 15 mm and the cup diameter was 30 mm. This configuration minimizes wall slip and confinement effects[15], allowing samples with macroscopic fibers to be measured reliably. In the case of fiber foams, the vane geometry can be inserted into the sample without compressing it and therefore without altering its air content or bubble size distribution. An open-ended syringe was used to carefully transfer foam samples from the foaming container to the rheometer cup, ensuring that the foam samples were neither compressed nor there were air pockets created inside the sample.

Frequency sweep measurements for fiber suspension samples were performed at angular frequencies ranging from 0.1 to 100 rad/s at a strain amplitude of 0.1 %. In the amplitude sweep measurements, the strain amplitude was varied from 0.01 to 1000 % in the case of fiber suspension samples and from 0.01 to 100 % in the case of fiber foam samples, while the angular frequency was 3 or 10 rad/s. Shear rate sweep measurements were performed for fiber suspension samples first at increasing shear rate (from 0.001 to 100 s^{-1}), and then at decreasing shear rate (from 100 to 0.001 s^{-1}). Only data measured at decreasing shear rate are reported herein. All rheological data were obtained from the TRIOS software (TA Instruments) that employs the so-called "Couette analogy" in the calculation of rheological properties when a wide-gap vane-in-cup geometry is used, as described in Vadillo et al.[16]

RESULTS

MFC fiber suspensions

Figures 2a and **2b** present the results of frequency sweep and amplitude sweep measurements for the coarse HC MFC grade. The rheological behavior of HC MFC suspensions exhibit pronounced viscoelastic, gel-like characteristics that are strongly dependent on fiber consistency. Frequency sweep measurements show that HC MFC suspensions are highly elastic ($G' > G''$), while both storage and loss modulus showed little dependence on frequency within the studied range as shown in **Fig. 2a**. In the amplitude sweep measurements, all suspensions show a well-defined linear viscoelastic region (LVER), within which both the storage modulus (*G′*) and loss modulus (*G″*) remain relatively constant, indicating an intact fibrillar network. The limit for the LVER is observed to be around 0.1-0.5 % depending on the fiber consistency, and the crossover between *G'* and *G''* (commonly known as the flow point) is observed at strain amplitudes of ~35-50 %. Above this point, the material behavior becomes predominantly liquid-like. Both *G'* and *G''* decrease monotonously with increasing strain amplitude, demonstrating Type I LAOS behavior as classified by Hyun *et al.*[17]

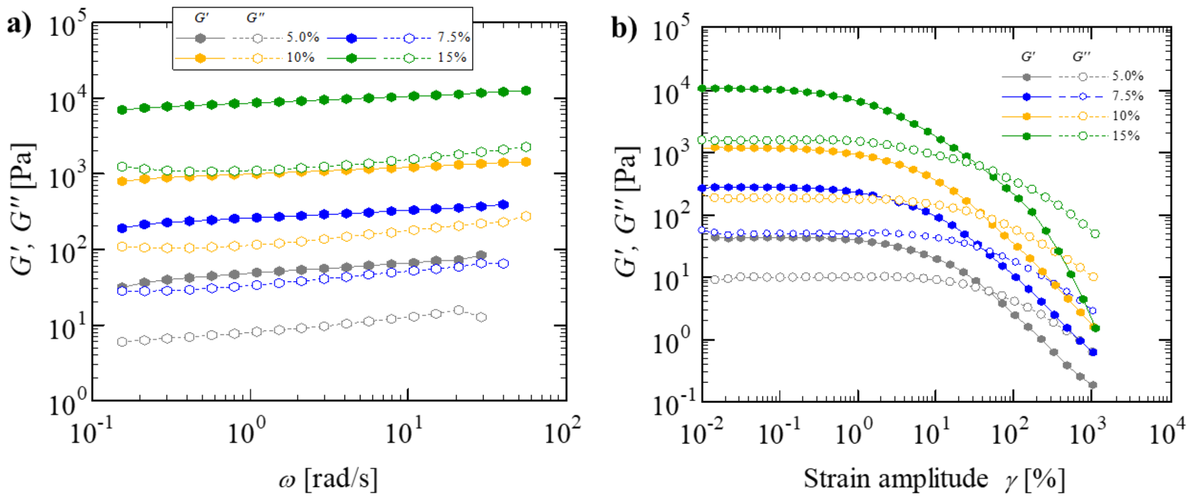

FIGURE 2: Storage modulus and loss modulus for coarse HC MFC grade a) as a function of angular frequency and b) as a function of strain amplitude. The amplitude sweep measurements were performed at an angular frequency of 10 rad/s.

Fig. 3a and **Fig. 3b** present the results of amplitude sweep and shear rate sweep measurements for the coarse and fine HC MFC grades, together with the commercial MFC reference at 2.5% fiber consistency. At the same fiber consistency, clear differences are observed between the samples. The commercial MFC exhibits the highest storage and loss moduli, indicating the formation of a comparatively stronger and more rigid fibrillar network at the same fiber consistency. In contrast, both HC MFC grades show lower moduli, reflecting a less densely connected structure. The fine HC MFC grade consistently shows higher *G'* and *G''* compared to the coarse grade.

The shear viscosity results in **Fig. 3b** show that all suspensions exhibit strong shear-thinning behavior, with viscosity decreasing continuously with increasing shear rate. At the same fiber consistency (2.5%), both HC MFC grades exhibit significantly lower viscosity than the commercial MFC over the studied shear rate range. For example, at a shear rate of 10 s^{-1}, the viscosity of the commercial MFC is about one order of magnitude higher than that of the fine HC MFC grade and about two orders of magnitude higher than that of the coarse HC MFC grade. Overall, the results demonstrate that HC MFC produced by mechano-enzymatic fibrillation provides reduced flow resistance over the commercial MFC produced entirely by mechanical fibrillation, enabling easier handling and more efficient processing.

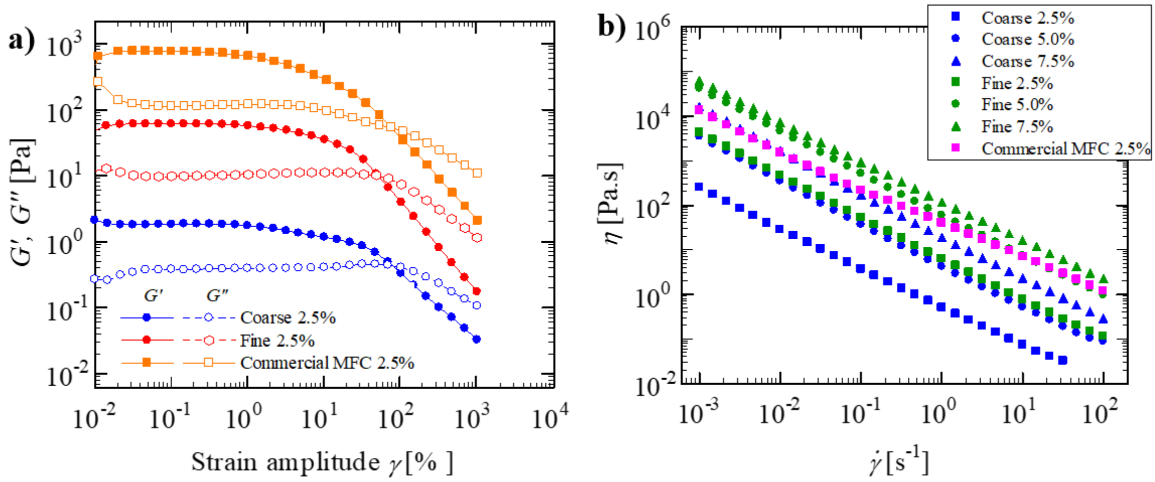

FIGURE 3: a) Storage and loss modulus as a function of strain amplitude for the coarse HC MFC, fine HC MFC, and commercial MFC suspensions. The amplitude sweep measurements were performed at an angular frequency of 3 rad/s. b) Viscosity as a function of shear rate for the three different MFC suspensions.

MFC fiber foams

Following the rheological characterization of HC MFC fiber suspensions, the analysis was extended to fiber foams prepared from the same HC MFC materials. For the fiber foams, CMC was added to improve dispersion and foam stability while reducing granule formation during mixing.

It is important to note that the rheological behavior of fiber foams can differ significantly from that of suspensions. Compared to suspensions, fiber foams contain less free water, which restricts fiber mobility and promotes inter-fiber contacts within the network. Furthermore, the presence of air bubbles alters the network structure and connectivity, effectively increasing the solid volume fraction and influencing the rheological response of the system.[13, 18, 19] These structural differences are expected to influence the viscoelastic and flow behavior of the materials.

Fiber foams represent a multiparameter system, in which not only the fiber properties but also the air content plays a critical role. It is well established that even small variations in air content can significantly influence rheological properties, and therefore careful control and reporting of air content is essential when comparing different samples.[20]

To the best of the authors' knowledge, this study presents the first investigation of high-consistency mechano-enzymatically fibrillated cellulose HC MFC fiber foams and their rheological behavior.

The corresponding air contents of the coarse HC MFC foams were 72.9% at 2.5% consistency, 73.1% at 5.0% consistency, and 64.1% at 7.5% consistency.

In **Fig. 4**, the amplitude sweep measurements revealed that the studied systems exhibit a predominantly elastic response at small deformations, as indicated by the storage modulus (G') values exceeding the loss modulus (G'') for all consistencies. With increasing strain amplitude, a transition from solid-like to liquid-like behavior was observed, marked by G'' surpassing G' at larger deformations. Similarly to the HC MFC suspensions, Type I LAOS behavior is observed at the lowest fiber consistency of 2.5%. However, at higher fiber consistencies of 5.0% and 7.5% Type III LAOS behavior is observed, as the G'' curve shows strain overshoot. At increasing fiber consistency, the fiber network within the foam becomes increasingly dense and hence the number of fiber-fiber contacts increases. These frictional contacts cause an increase in G'' values when the fibers start to slide against each other at increasing strain amplitude.[21] Both G' and G'' increased systematically with increasing consistency, reflecting the formation of stronger and more interconnected fiber networks at higher solids contents. In addition, the flow point, defined by the crossover of G' and G'', shifted to higher strain amplitudes with increasing consistency. This indicates that larger deformations are required to induce flow – i.e. predominantly liquid-like behavior – in more concentrated systems.

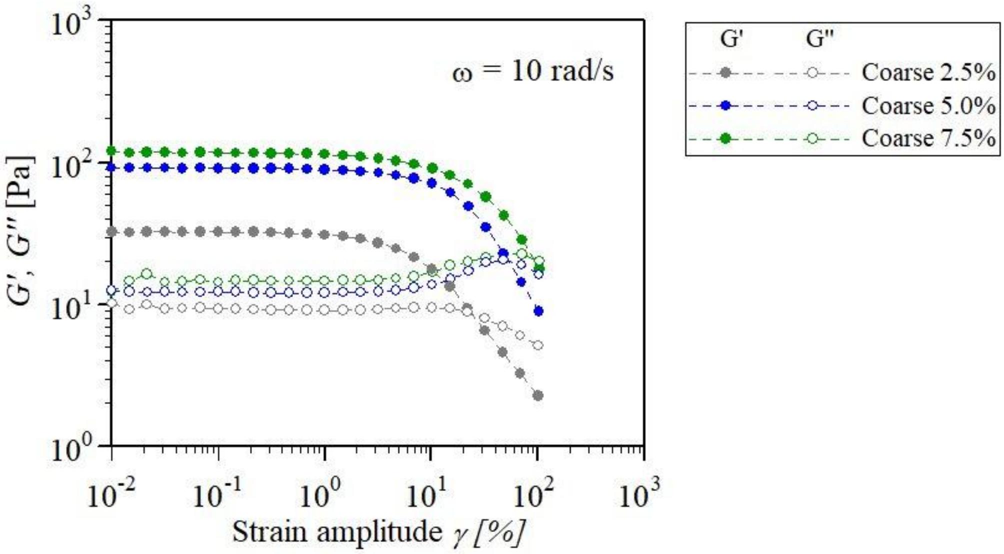

FIGURE 4: Storage and loss modulus as a function of strain amplitude of the coarse grade HC MFC fiber foams at consistencies ranging from 2.5%, 5.0% to 7.5% and air contents of 72.9%, 73.1% and 64.1%, respectively.

Having established the influence of consistency on the rheological behavior of coarse HC MFC fiber foams (**Fig. 4**), the effect of fibrillation degree was further investigated by comparing coarse and fine HC MFC grades, as shown in **Fig. 5**. In addition, the rheological properties were benchmarked against a commercially available MFC reference. The measured air contents were 72.9% for the coarse HC MFC foam, 71.3% for the fine HC MFC foam, and 70.5% for the commercialMFC foam at 2.5% consistency.

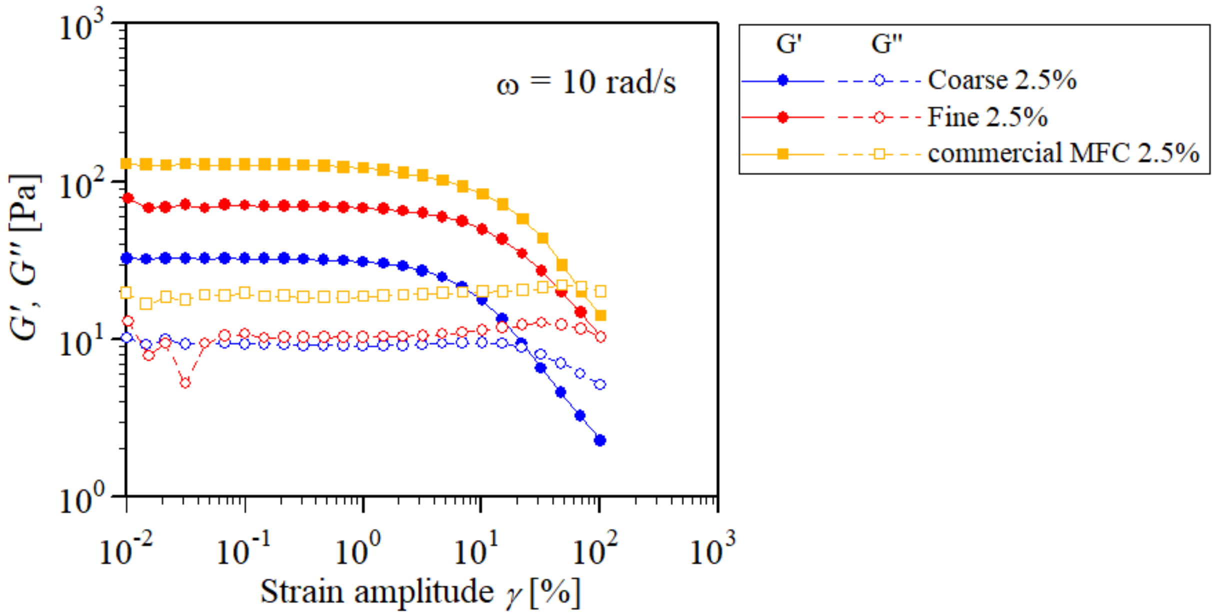

FIGURE 5: Storage and loss modulus as a function of strain amplitude of the coarse and fine grade HC MFC fiber foams, as well as the commercially available MFC reference at consistencies of 2.5% with resulting air contents of 72.9%, 71.3% and 70.5%, respectively.

As shown in **Fig. 5**, the fine HC MFC grade exhibited higher storage (G') and loss (G'') moduli compared to the coarse HC MFC grade, indicating a stronger and more interconnected network structure. Furthermore, the flow point ($G' = G''$) occurred at higher strain amplitudes for the fine grade, demonstrating that larger deformations are required to disrupt the internal structure and induce flow.

In comparison to the commercial MFC reference, HC MFC fiber foams showed lower G' and G'' values at comparable consistencies, indicating a less stiff network. This demonstrates that mechano-enzymatically fibrillated MFC can be used to produce fiber foams with improved handleability and processability. All fiber foams displayed pronounced shear-thinning behavior.

CONCLUSIONS

This study provides the first systematic investigation of the rheological behavior of high-consistency mechano-enzymatically fibrillated cellulose HC MFC fiber foams (previously referred to as HefCel). The results demonstrate that both HC MFC suspensions and foams exhibit characteristic viscoelastic behavior, with a predominantly elastic response at small deformations and a transition to liquid-like behavior at large strain amplitudes.

The rheological properties of HC MFC suspensions and foams were strongly influenced by fiber consistency, with increasing fiber consistency leading to higher storage (G') and loss (G'') moduli. In HC MFC foams, the flow point was shifted to higher strain amplitudes as the fiber concistency increased. This indicates that with increasing fiber consistency a stronger and more interconnected fiber networks forms in HC MFC foams, requiring larger deformations to induce flow. In addition, variations in air content were found to influence the foam structure and therefore the fiber network and should be carefully considered when interpreting rheological data.

The degree of fibrillation further affected the rheological response. The fine HC MFC grade exhibited higher moduli and greater resistance to structural breakdown compared to the coarse grade, reflecting a more developed fibrillar network. When compared to a commercial MFC reference, HC MFC suspensions and foams showed lower stiffness at comparable consistency, although similar qualitative rheological behavior was observed.

Overall, the results highlight that both fiber properties and foam characteristics, such as air content, govern the rheological behavior of HC MFC foams. These findings contribute to the understanding of structure–property relationships in fiber-based foams and indicate improved processability of HC MFC suspensions and foams at high consistencies. Furthermore, the results of this study support the potential of HC MFC as a promising material platform for applications requiring lightweight and porous structures.

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