**Characterizing the dispersion of Cellulose Micro/Nanofibers hydrogels**

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**Highlights**

* Gel point is used to evaluate CMF/CNF dispersion
* Stirring speed modifies CMF/CNF hydrogels network
* CMF/CNF dispersion affect to their application efficiency

**1. Introduction**

Cellulose micro/nanofibers (CMF/CNF) have gained attention due to their desirable properties as high strength, stiffness, high surface area or low toxicity [1,2]. The methods to disintegrate cellulose fibers into substructures are well-known. However, CNF characterization is an area still under development [3]. The history of CNF before its use has a significant effect on their efficiency. Therefore, the dispersion of CNF hydrogels is an important parameter to understand and optimize their effectiveness in several applications, e.g. strength additive or wastewater treatments. Nowadays, there is not a methodology to quantify how stirring speed influence on the CNF network in suspensions. For this reason, the adaptation of a parameter used to measure NC sedimentation, the gel point (øg), is proposed [4-6]. øg, the volume concentration in the boundary between dilute and semi-dilute region, may be considered the lowest volume fraction at which all primary flocs are interconnected throughout the container, and form a self-supporting network [5]. The aim of this work is to show how CNF/CMF hydrogels perform at different stirring speed using the øg as parameter to quantify the dispersion in hydrogels.

**2. Methods**

CMF/CNF were obtained from recycled old newspaper. It was disintegrated in a pulp disintegrator at 30,000 revolutions before soaking. CNF were obtained using TEMPO-mediated oxidation with 10 mmol of NaClO/g dry pulp and 4 steps of homogenization at 600 bars in a homogenizer. CMF were obtained by refining the pulp in a PFI mill at 5,000 revolutions and 6 homogenization steps. Both products were characterized [1]. øg was evaluated with 250 mL of CMF/CNF suspensions at different concentrations (Co), stirred with a 3-bladed propeller stirrer at several speeds for 10 min. Crystal violet was used as dye to visualize suspensions. Then, they were settled for 2 days (CMF) or 8 days (CNF) to claim fibers deposition. The curve Co vs. the relation deposit height / initial height (Hs/Ho) was fitted with a quadratic equation for each speed. The linear term gives the øg [4-6].

**3. Results and discussion**

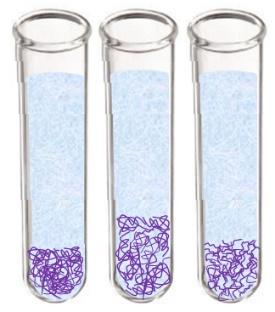
Figure 1 shows Co vs. Hs/Ho for CMF/CNF at different stirring speeds. Gel point values were obtained from the fit of quadratic equations and Figure 2a shows Gel point for each stirring speed.

øg decreases slightly in both cases with stirring speed until a minimum value and then, øg increases. The suspensions with the stirring speed that produce the lower øg, have the higher Hs/Ho relation (Figure 2b). The opening of the network increase with stirring speed until the lower øg in which the fibers start to break and destroy the structure, decreasing Hs/Ho relation. The results were compared using Electronic Microscopy. An opening network would be the more effective state to applicate CNF/CMF hydrogels in applications as strength additive in cardboard or water-based inks treatment.

**b)**

**a)**

**Figure 1.** Gel point representation: a) CNF; b) CMF.



**a)**

CNF

CMF

**b)**

**Figure 2.** a) Influence of CNF/CMF gel dispersion on Gel point; b) CMF network representation with stirring speed.

**4. Conclusions**

øg of CNF/CMF at different stirring speeds have a minimum for each material with a lower stirring speed in CNF due to the high fibrillation and an easy break. Suspensions stirred when øg is minimum have the most opening network and they would be the most effective state to applicate the hydrogel. However, higher stirring speeds break the fibers and destroy the networks. Gel point methodology can be used in other cellulosic materials to obtain the optimal network configuration.

**Acknowledgement**

The authors wish to thank the Economy and Competitiveness Ministry of Spain for the support of the project (CTQ2017-85654-C2-2-R) as well as UCM and B. Santander for the grant of J.L. Sanchez-Salvador (CT17/17).

**References**

1. A. Balea, N. Merayo, E. Fuente, P. Mutje, A. Blanco, C. Negro. Bioresources, 11 2016 3416-3431.
2. D. Klemm, E.D. Cranston, D. Fischer, M. Gama, ... K. Petzold-Welcke. *Materials Today*, 21 2018 720-748.
3. A. Blanco, M.C. Monte, C. Campano, A. Balea, N. Merayo, C. Negro. In Handbook of Nanomaterials for Industrial Applications, Elsevier, 2018, pp. 74-126.
4. W.K. Mosse, D.V. Boger, G.P. Simon, G. Garnier. Langmuir, 28(7) 2012 3641-3649.
5. P. Raj, A. Mayahi, P. Lahtinen, S. Varanasi, G. Garnier, D. Martin, W. Batchelor. *Cellul.* 23 2016 3051-3064.
6. P. Raj, S. Varanasi, W. Batchelor, G. Garnier. *J. Colloid Interface Sci.* 447 2015 113-119.