Stability analysis for different grades of Micro-fibrillated Cellulose (MFC)

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RAW MATERIALS

Introduction

Nano-scale cellulose fibre materials (e.g., Micro-fibrillated Cellulose) serve as a promising candidate for bio-based materials from a sustainable resource thanks to their abundance, high strength and stiffness, low weight and biodegradability.

Micro-fibrillated Cellulose is obtained from a fibrillation process in water which converts cellulose fibres into a volume spanning three-dimensional network of micro fibrils. As a result, a robust product with interesting rheological properties is formed. It is well adapted for application fields such as cosmetics, food, coatings, cements, agrochemicals and nanocomposites...

Characterization of fibrillated celluloses however, remains challenging due to its heterogeneous nature. MFC mostly consist of long, slender fibril aggregates with a high degree of branching. Fibrillated celluloses contain fibrils and fibril aggregates with different dimensions and thus their proper assessment requires methods adapted for multi-dimensional analysis.

KEY **BENEFITS**

FAST

NO DILUTION

SENSITIVE

Reminder on the technique

Turbiscan® technology, based on Static Multiple Light Scattering, consists on sending a light source (880nm) on a sample and acquiring backscattered (BS) and transmitted (T) signal over the whole sample height. By repeating this measurement over time with adapted frequency, the instrument enables to monitor physical stability.

The signal is directly linked to the particle concentration (φ) and size (d) by the Mie theory knowing refractive index of continuous (n_f) and dispersed phase (n_p):

$$\boldsymbol{BS} = \boldsymbol{f}(\varphi, \boldsymbol{d}, \boldsymbol{n_p}, \boldsymbol{n_f})$$

Method

Three water-based suspensions of micro-fibrillated cellulose (MFC) were analysed at the room temperature for 8 days. The size of fibrils and their aggregates are rather distributed with filamentous and branching shape:

- Width: from a few nanometres up to a few micrometres
- Length: approximately 100 times greater than the diameter according to preliminary SEM studies.

The table below gives the details of size and concentrations:

Sample	A	B	C
Fibre width	few micrometers	sub-micrometers	nanoscale range
Concentration	0.017%wt	0.123%wt	0.012%wt

Table 1: Sample description

Results

Raw data

Figure 1 illustrates the delta-transmission signal for the three samples as a function of the sample height over time (from blue to red curves).

The Turbiscan® profiles for sample A and B display an increase at the top of the sample (right part of the graph) and a decrease at the bottom of the sample (left part of the graph). It means that the concentration of scatterers is increasing from the top to the bottom of the sample – sedimentation is occurring.

In other words, particle migration is occurring causing the sample clarification at the top of the sample...

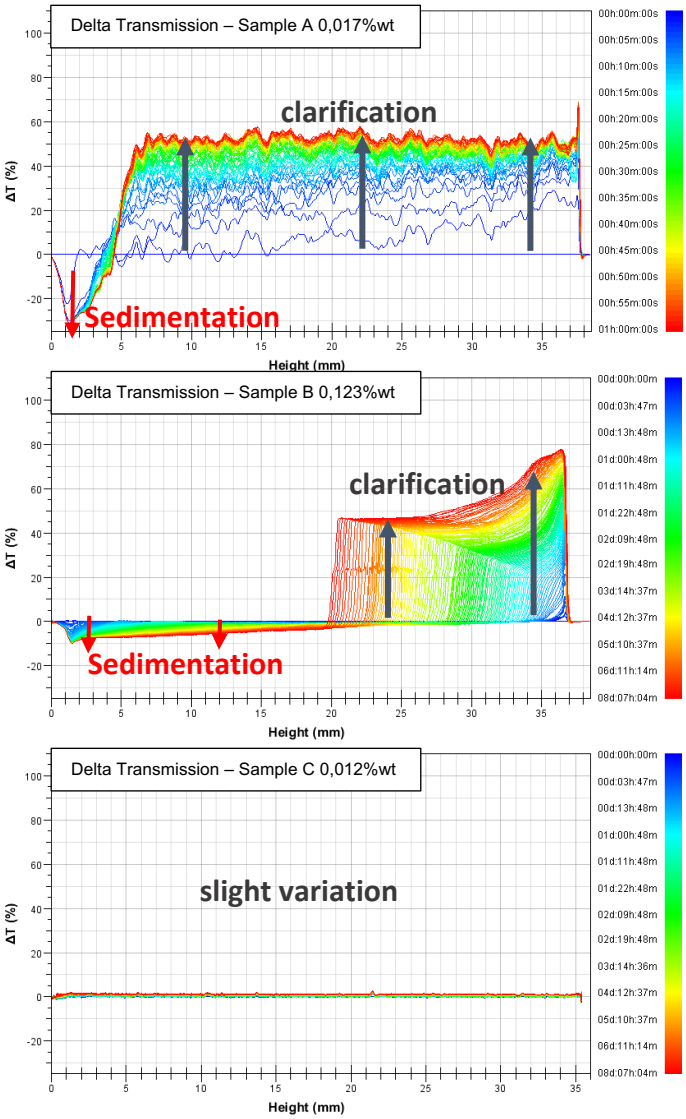

*Figure1. Transmission signal as a function of sample over time (in order Sample **A, B, C**)*

Samples A and B with higher fibre width show a significant destabilization compared to sample C which contains nanoscale fibrils. Sample C shows only a slight variation over the whole height which is linked to size variation in the sample.

Stability measurement

Thanks to the Turbiscan Stability Index (TSI), it is possible to evaluate the destabilization kinetics in the samples with time. The index value is calculated by summing up all the variations of signal detected due to any destabilization phenomena (sedimentation, clarification, size variation, …). At a given time, higher TSI represents more significant destabilization of the sample.

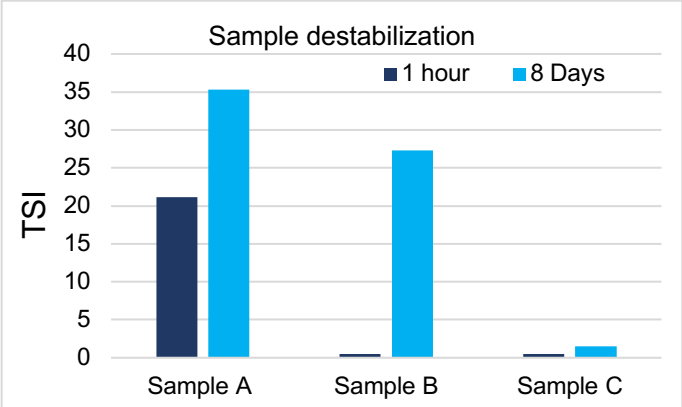

Figure 2: TSI for the three samples after 1 hour and 8 days

The ranking of stability after 1 hour of measurement shows that sample C is more stable than A. Considering that both have similar concentrations, fibril aggregates with a microscale width (sample A) settle very quickly, whereas fibrils with a nanoscale width (sample C) remain suspended.

Assuming that sample B and C have size in a similar scale, C is more stable than B as its concentration is ten times lower.
In addition, sample A gets destabilized very quickly in the first hour as it contains very big fibrils aggregates, but sample B shows little evolution in the first 1 hour but the destabilization is well noticed after 8 days.

CONCLUSION

Turbiscan LAB, based on Static Multiple Light Scattering, can provide a deep insight into the analysis of colloidal stability of micro-fibrillated cellulose. The instrument is sensitive to fibril aggregate sedimentation rate and provides a TSI number that ranks destabilization level between samples. The advantage of the technique is the measurement at rest and with no dilution that characterizes the sample in its real state of application.