

CEPI-CTS NEWSLETTER 2022

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Foreword

Welcome to the ninth issue of the CEPI-CTS Newsletter.

In this very rich issue we will update you on the activities within CEPI's 4evergreen project that aims at achieving a European test method to evaluate the recyclability of paper- and board-based products; we will also present you the recent advances in the development of all-cellulose barrier packaging materials in the framework of the CelluWiz project: both articles are a summary of the presentations given during the CEPI-CTS 2021 Technical Workshop.

Then we will describe a standard to assess the disintegration of tissue papers and products; we will show that measuring the Cobb is not as simple as it looks. Finally we will illustrate two useful statistical tools.

We greatly value your feedback and suggestions to improve the Service: if you have questions, doubts or requests do not hesitate to contact your Distributing Laboratory or myself at the contacts listed on the left.

Fulvio Savagnone
CEPI-CTS Chairman

Harmonising a European method to test paper recyclability

(Heinz Joachim Schaffrath, PMV-TU Darmstadt, Daniele Bussini, Innovhub)

Since 2019 a working group, facilitated by CEPI and consisting of several experts from a number of European countries, is in charge to establish a new, harmonized method to evaluate the recyclability of paper- and board-based products.

Many methods are already used in Europe, such as the PTS-developed RH 021, the EcoPaperloop method, the Italian standard UNI 11743 in connection with ATICELCA 501 assessment scheme, as well as a French method used by CTP.

All these methods take into account the same parameters: coarse reject, flakes, stickies and optical homogeneity of a handsheet made from the recovered fibres. But details in the procedures are different – which water to use for disintegration, which size of pieces to disintegrate, which kind of disintegrator and pulping conditions, which type of screening equipment and settings, etc.

By the end of 2020 the working group published a first draft of a new harmonised method that specifies all these parameters. In this draft, an ISO-standard disintegrator and a Somerville screening device are used, to ensure that most laboratories in Europe would be able to carry out the test.

Right now, as part of CEPI's 4evergreen project workstream 1, a number of laboratories in Europe are executing this method and sharing their experience, in order to test the feasibility of the procedure and get a first evaluation of the reproducibility and repeatability of results.

The experimentation work is still going on: for instance a new parameter describing how the water quality is affected by the tested product is currently being studied for inclusion in the method.

A new draft of the method is presently being discussed by the CEPI 4evergreen experts, as well as its related assessment scheme.

Tables: Comparison of assessment schemes

Total Assessment

Single scores see different sheets

CR = Coarse Reject, FC = Flake Content, MSA = Macra Sticky Area, OH = Optical Homogeneity

<p>UNI 11743 + Aticelca MC 501_19</p>	<p>The lowest level determines the total assessment</p> <p>Total assessment:</p> <ul style="list-style-type: none"> recyclable, level A+ recyclable, level A recyclable, level B recyclable, level C not recyclable 	
<p>EPL (Eco Paper Loop)</p>	<p>In case all criteria are in the green or yellow range, total score may be calculated</p> <p>Score 71 bis 100 = "good recyclability"</p> <p>Score 0 bis 70 = "fair recyclability"</p> <p>CR or MSA in the warning range = "tolerable"</p> <p>CR or FC above threshold = "for specialized purposes"</p> <p>MSA above threshold = "not recyclable"</p>	
<p>PTS</p> <p>RH 021/97:2012 cat. 2 (packaging)</p>	<p>All criteria are fulfilled = "recyclable"</p> <p>Criteria in the yellow range = "conditionally recyclable"</p> <p>One criterium not fulfilled = "not recyclable"</p>	

Base:
Assessment scheme according to Aticelca MC 501_19
placed in this scheme: PTS RH 021-97:2012 categorie 2 (packaging paper) and EcoPaperLoop (EPL)

Coarse Reject	%	< 1,5	1,5 - 2	> 2 - 10	> 10 - 20	> 20 - 30	> 30 - 40	> 40 - 50	> 50
UNI/Aticelca	assessment	recyclable	recyclable	recyclable	recyclable	recyclable	recyclable	not recyclable	not recyclable
	Level	A+	A	A	B	C	C	not recyclable	not recyclable
EPL <small>SCP = sacks (composite materials) LIP = Liquid Packages</small>	Score 1 - general	35	35	< 35 to 19,4	< 19,4 to 0	0	< 0 to -12,5	< -12,5 to -25	< -25 to -87,5
	Score 2 - SCP + LIP	35	35	35	< 35 to 0	0	< 0 to -17,5	< -17,5 to -35	< -35 to -122,5
PTS	assessment	recyclable	recyclable	recyclable	recyclable	conditionally recyclable	conditionally recyclable	conditionally recyclable	not recyclable

Base:
Assessment scheme according to Aticelca MC 501_19
placed in this scheme: PTS RH 021-97:2012 categorie 2 (packaging paper) and EcoPaperLoop (EPL)

Flake Content	%	< 5	5 - 15	> 15 - 20	> 20 - 40	> 40 - 50	> 50
UNI/Aticelca	assessment	recyclable	recyclable	recyclable	recyclable	recyclable	recyclable
	Level	A+	A	B	B	C	C
EPL	Score - general	15	< 15 bis 10,7	< 10,7 bis 8,6	< 8,6 bis 0	< 0 bis - 4,3	< -4,3 bis -25,7
PTS	assessment	recyclable	recyclable	recyclable	conditionally recyclable	conditionally recyclable	not recyclable

Base:

Assessment scheme according to Aticelca MC 501_19

placed in this scheme: PTS RH 021-97:2012 categorie 2 (packaging paper) and EcoPaperLoop (EPL)

MSA, < 2.000 µm	mm ² /kg	< 1.000	1.000 - 2.500	> 2.500 - 10.000	> 10.000 - 20.000	> 20.000 - 30.000	> 30.000 - 50.000	> 50.000
Aticelca	assessment	recyclable	recyclable	recyclable	recyclable	recyclable	recyclable	not recyclable
	Level	A+	A+	A	B	C	C	not recyclable
EPL	Score - general	40	< 40 bis 36,8	< 36,8 bis 21	< 21 bis 0	0	not recyclable	not recyclable
PTS	no determination							

Base:

Assessment scheme according to Aticelca MC 501_19

placed in this scheme: PTS RH 021-97:2012 categorie 2 (packaging paper) und EcoPaperLoop (EPL)

Optical Homog.	-	level 1	level 2	level 3	
Aticelca	assessment	recyclable	recyclable	recyclable	
	Level	A+	A	B	
EPL	assessment	very good	good	tolerable	? not tolerable?
	Score - general	10	5	0	
PTS	assessment	recyclable	conditionally recyclable	conditionally recyclable	

Base:

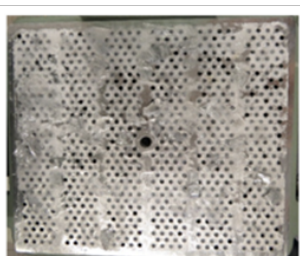
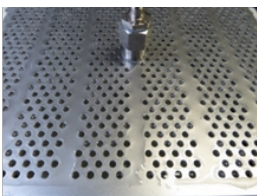
Assessment scheme according to Aticelca MC 501_19

placed in this scheme: PTS RH 021-97:2012 categorie 2 (packaging paper) und EcoPaperLoop (EPL)

Sheet peeling test

Adhesiveness	-	absent	present
Aticelca	assessment	recyclable	not recyclable
	Level	A+	not recyclable
EPL	no determination		
PTS	assessment	recyclable	not recyclable

Coarse (Ø 5mm) and fine screening (// 0.150 µm)



Barrier-effective all-cellulosic packaging materials *(Fleur Rol and Philippe Martinez, CTP)*

CelluWiz is a research project whose overall objective is to develop two processes able to produce a renewable, recyclable/recycled and biodegradable all-cellulose packaging material that can offer a competitive alternative to existing multi-layers plastic materials or multi-materials used in the packaging sector.

Two technologies are combined to produce three proofs of concept of packaging (a clamshell, a cup and a tray) that are bio-based, recyclable and biodegradable, while showing performances at least equivalent to market references:

- **MFC wet lamination process.** This patented process combines, without glue, a board with a layer of Micro-Fibrillated Cellulose (MFC), creating a stratified cellulosic material. The MFC layer is thin enough to be economically viable and thick enough to get the barrier performance to grease, contaminants and oxygen. The first pilot machine was installed at CTP in 2019.
- **Chromatogeny coating and grafting process,** specific version for MFC layers. This ultrafast, efficient and solvent-free chemical grafting process will be revised to increase grafting efficiency and create a micrometre size layer of pure cellulose ester which will bring water and water vapour barrier.

First results are already available on **CelluWiz** newsletters or on www.CelluWiz.eu. The first materials produced, shown to be perfectly recyclable and compostable, already satisfy requirements for clamshell application with a kit test value higher than 11 and a water absorption lower than 20g/m² after 60s.

The **CelluWiz** project (2019-2022) has received funding from the Bio-Based Industries Joint Undertaking, within the European Union's Horizon 2020 research and innovation program under grant agreement No 838056.



A new standard for tissue papers and tissue products *(Sylvie Moreau-Tabiche, CTP)*

The EN ISO 12625 series of standards has been recently enriched with EN ISO 12625-17:2021 “Tissue paper and tissue products - Part 17: Determination of disintegration in water” (*Ed.: in the French version, the specific French word “délitage” is used*).

This standard describes a method to assess the disintegration of tissue papers and products when subjected to mechanical agitation in water. It is applicable to all tissue papers and tissue products, even if mostly toilet paper will be analysed.

The principle is the following: a test piece of tissue paper or tissue product is introduced in a beaker and agitated in water at a constant speed for time durations of 30 s, 2 min and 10 min. After agitation, the beaker content is poured into a sieve, and the remains (if any) are collected and weighed to determine the percentages of disintegration after each agitation time period.

The device and conditions used to disintegrate the sample of tissue paper are the ones defined in the French standard NF Q 34-020:1998 “Sanitary and domestic articles – Toilet paper - Measurement of disintegration”. The determination of the end point has been improved by a gravimetric determination instead of a visual one.

This test could be implemented in our CEPI-CTS test list in a near future.



Cobb: a deceptively easy test (Wilco de Groot, IGT, Khadija Barkani, Celabor)

The Cobb water absorption test is one of these very common and “very easy to perform” tests in the paper industry. But everything which look easy is not always that easy if a certain level of reproducibility is needed or expected. To understand why, it is important to understand the factors influencing the result:

- Water volume



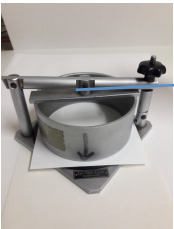
- Water leakage under the ring
- Unwanted drops of water outside the measuring area, filling and emptying the container
- Water passing through the substrate under test
- Timing



- Blotting paper, weight, timing



- Dimensions of the apparatus, pressure of the clamping system



Caution! The grab bar must be balanced in relation to the cylinder.

Some of these are instrument specific, others are substrate dependent. Most are user dependent: here are some examples:

Water leakage under the ring: for a 100 cm² ring, 1 mm penetration under the ring results in 3,6 % increase in wetted area. Do the same with a 50 cm² ring and the result is an error of 5%.

One drop of water weighs about 50 mg. So each “standard” drop of spilled water may result in anything between 3 and 10% difference in Cobb value.

Timing between different steps in the test may have huge influence on the results: will you start pouring out the water 15 s before the end of the nominal test time or exactly when the nominal test time expires? Removing the clamps directly or after these same 15 s?

Then if all these parameters are kept constant, the results within a laboratory should be consistent, but how about between other laboratories? This is what the CEPI-CTS is all about: inter-laboratory testing, see the article below for some useful statistical tools to evaluate laboratories differences.

Two useful statistical tools *(Xavier Joppin, Celabor)*

In many cases a laboratory may underestimate the uncertainty of its results that is associated with the measuring device and the testing process, as explained above for the Cobb test.

In an interlaboratory testing scheme, a better way to estimate the consistency between one laboratory and the other laboratories is to evaluate the Z-score.

$$Z = \frac{x - \mu}{\sigma}$$

Score
Mean
SD

But when the operator intervention can induce several other uncertainties, it is important to also evaluate the Zeta-score.

$$z\acute{e}ta = \frac{x - m}{\sqrt{u_x^2 + u_m^2}}$$

In the Zeta-score formula above m is the calculated mean, u_x is the uncertainty of the laboratory measurement and u_m is the uncertainty of the assigned value. Zeta-score and Z-score are complementary parameters and cannot substitute each other.

A good Z-score (between ± 2) indicates that the laboratory result is consistent with that of other laboratories.

A good Zeta-score indicates that the laboratory has identified and integrated in the analysis of the process all sources of uncertainty, thus allowing to define an interval that includes the true value with a likelihood of 95%.

According to ISO 13528, the limit values of the Zeta-score are the same as for the Z-scores: ± 2 = monitoring limits, ± 3 = action limits. (Ed.: years of experience has lead CEPI-CTS to use narrower action limits ($\pm 2,6$, see CEPI-CTS Handbook 6:1, "Statistical treatment of the measurement data", because the higher natural variance observed in testing paper-based materials can often cause high variances which in turn lead to excessively wide limits).

If $|z\acute{e}ta| > 3$, the measurement uncertainty estimated by the laboratory is underestimated. It does not explain the difference between the laboratory analytical result and the assigned value. A new evaluation of the laboratory uncertainties is needed. The table below shows the possible situations.

Lab value	Lab uncertainties	Z-score	Zêta-score
Correct	Correct	Good	Good
Far away	Correct	Bad	Good
Correct	Underestimate	Good	Bad
Far away	Underestimate	Bad	Bad

Come meet us and share technical ideas!

Every year CEPI-CTS organises a Technical Meeting to discuss paper-testing issues that are relevant to the Service and a Technical Workshop to have a hands-on experience on selected technical topics.

Both the Technical Meeting and the Workshop are open to the public: they are therefore a unique occasion to discuss state-of-the-art technical matters with scientists and technicians from the most important European research institutes that manage a proficiency testing scheme that has no equal in the world with regards to scientific soundness, scope of availability of tests, efficiency, number of satisfied clients and last but not least, historical tradition.

The 2021 CEPI-CTS Technical Meeting and Workshop the meeting was held remotely one the web, due to the COVID-19 pandemic; they were also attended by guests from French paper industries. Scientists from PTS, CTP and PMV gave very interesting presentations on forensic paper analysis for the authentication of documents and works of art, on recent

advances in the development of all-cellulose barrier packaging materials in the framework of the CelluWiz project and on the activities to reach a European harmonised method on the evaluation of paper recyclability in CEPI's 4evergreen project framework (both presentations briefly summed up in the articles above).

To make your attendance even more fruitful, we will strive to organise such meetings in correspondence with national or international fairs, conferences and so on. We will adequately publicise our next Technical Meeting and Workshop, don't miss this opportunity!

What they say about us

"The CEPI Comparative Testing Service enables us to detect possible drifts of our devices."

Personnaz - Fibre Excellence

The next issue of the Newsletter will be out December 2022