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Sustainpack

Innovation and sustainable Development in the Fibre Based Packaging Value Chain

Instrument: **IP**

D2.16 – Basic recyclability testing of paper board

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Dissemination Level		
PU	Public	X
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RE	Restricted to a group specified by the consortium (including the Commission Services)	
CO	Confidential, only for members of the consortium (including the Commission Services)	

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1 Introduction

The overall objective of SP2 is to increase the strength (dry, moist, wet) of fibre based packaging materials, which will enable the design of more cost-effective packaging using less material with a target reduction of 30 %. In this context, Work package 2.2 deals with the development of paperboard while maintaining the structural thickness and critical material parameters that are important during converting and end use. With regard to properties, recyclability tests of the new paperboard materials are to be included in the investigation.

As it was not possible to provide enough sample material for recyclability testing by the partners during the first project phase, it was decided to postpone the recyclability activities and to include recyclability testing in SP2 Demonstrator projects (paperboard demonstrator project). For these recyclability tests, a paperboard with latex addition in the centre ply was chosen as ‘New concept board’. The corresponding test results are described below.

2 Recyclability - General aspects

Recyclability is understood to be the property of a used or ready-to-use paper or paperboard product to be processed in a recovered paper treatment plant that complies with the recognised rules of engineering in such a way that the resultant recovered paper pulp alone or as one of several stock components allows the trouble-free production of a new recycled fibre based paper and that has acceptable quality. In addition, a recovered paper component characterized as ‘recyclable’ will not when repulped cause an excessive increase in waste arisings or circuit and effluent loads.

The principal objective of the material recycling of products of paper and paperboard by the paper industry is to recycle the substances inherent in the paper, the main emphasis being almost entirely to recover the fibres. Fillers and pigments are not being actively recycled as yet. Since it is not a goal of paper recycling to recycle non-paper components such as plastics, metals, printing inks, adhesives, coatings, laminations, upholstery fabrics, impregnation agents, they merely have to comply with the requirement of being ‘recycling-oriented’. The latter term means they must not disturb or disrupt recovered paper treatment or papermaking, they must not unreasonably load process waters and effluents nor may they impair the quality of the new paper produced from the recycled pulp. Although non-paper components are usually considered to be unfavourable since they cause an increase in waste volumes, they are often unavoidable.

Regarding the requirements to ensure recyclability, all paper products - irrespective of their potential use - should be examined to determine whether they can be slushed within a reasonable period of time in a process that complies with the recognised rules of engineering. Furthermore, a recyclable product is expected to yield an RCF pulp (RCF = recycled fibre) after being defibered and subsequently cleaned that will permit trouble-free sheet formation. This then gives rise to the requirement in particular that a sheet produced from treated RCF pulp must not contain any stickies.

3 Recyclability tests of paper and board products

The recyclability of a paper product, i.e. its behaviour during recovered paper treatment as well as the properties of the recycled fibres produced and their subsequent behaviour in fresh paper production, is determined by various different factors such as the share and quality of usable components (fibres and fibre characteristics), amount and type of residues, and the disturbing potential of the pulp and its individual components.

The recyclability evaluations of this project focus on the disturbing potential during and after recovered paper treatment, to answer the question if papers produced with the new materials will cause operational trouble during recovered paper treatment or the production of fresh paper from recycled fibres.

Testing procedure

The samples will be tested according to the test procedure detailed below (Figure 1), to evaluate their recycling behaviour. Testing criteria are as follows:

- defibration characteristics (fragmentation behaviour),
- separating behaviour of disturbing substances by screening,
- sticky-forming potential of recycled fibres / disturbing potential of process water constituents (disturbing potential of substances entering the water circuits, e.g. micro-stickies, potential to form secondary stickies).

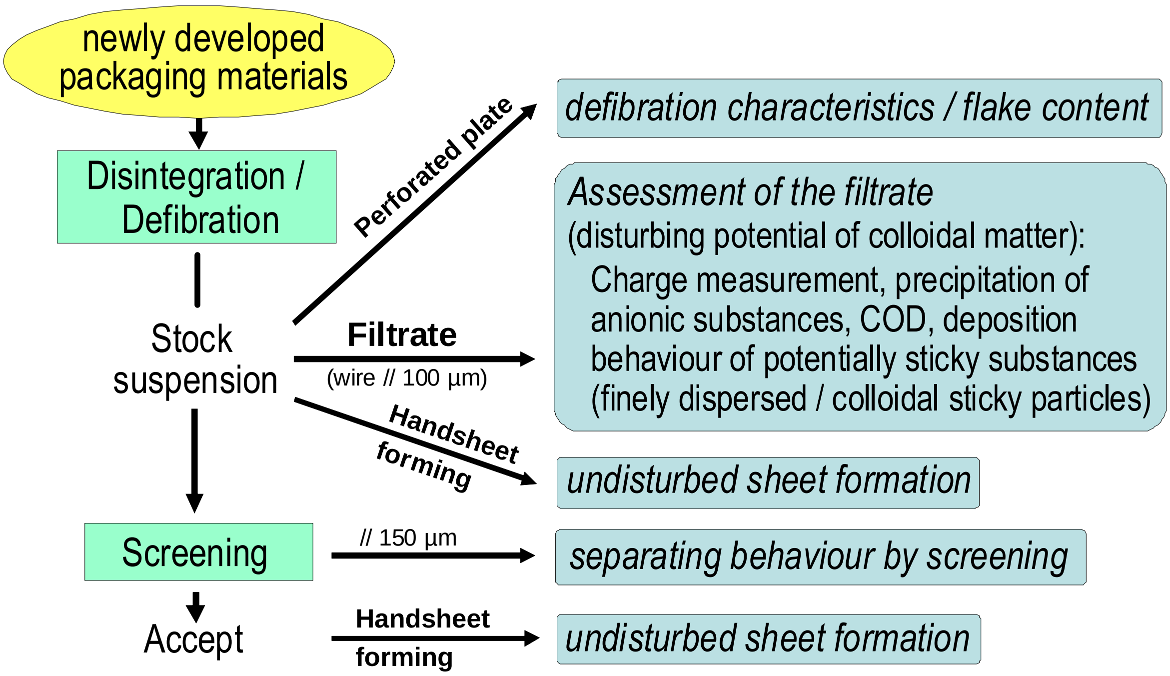


Figure 1. Testing scheme

The test procedure is based on the PTS method PTS-RH 021/97: Identification of the recyclability of paper and board packages and of graphic print products. In this method, the processes of recovered paper treatment are simulated in the laboratory for the products to be tested.

Using scissors, the paper sheets are cut into pieces measuring about 2 cm x 2 cm and conditioned overnight in a standard climate of 23 °C and 50 % relative humidity. The test portions are defibrated for 20 min. in a standard disintegrator at a consistency of 2.5 % o.d. and a volume of 2,000 ml according to DIN EN ISO 5263 (04.97) without preswelling. Tap water of 40 °C is used as dilution water. After disintegrating, the stock suspension is transferred to a standard distributor and diluted with tap water to a total volume of 10 l, which corresponds to 0.5 % o.d. consistency. For the determination of defibration characteristics, a fractionation test is carried out in line with ZM V/18/62 using a Brecht-Holl apparatus (perforated plate of 0.7 mm hole dia). Furthermore the stock suspension from the distributor is screened in a Haindl fractionator in line with ZM V/1.4/86, using a slotted plate (slot width 0.15 mm). Handsheets (approx. 60 g/m²) are tested for undisturbed sheet forming (sheet adhesion test), which are taken from the total stock and from the accept. For sheet forming, a Rapid-Köthen device is used according to DIN 54358 (02.81).

3.1 Testing methods

Defibration characteristics (fragmentation behaviour):

When evaluating the recyclability of packaging products, the focus is to determine, in relation to the possible material composition of packages, the proportion of non-paper components (such as plastic sheets or metal foils) and to evaluate the defibration behaviour under test conditions. Defibration characteristics are ascertained by a quantitative and qualitative (visual) assessment of the residuals on the perforated plate.

Sheet adhesion test:

The disruptive potential due to stickies is also of considerable importance. The sheet adhesion test using handsheets made from the slushed unscreened stock as well as from the screening accept indicates whether or not sticky components are present in the RCF pulp and whether or not they can be separated by mechanical screening processes.

- After drying, the handsheets together with board carrier and cover sheet are removed from the dryer and transferred to a drying cabinet with a temperature of 130 °C. In this cabinet they are placed between two brass plates and subjected to a surface pressure of 1.18 kPa for 2 minutes. Following this, the specimens are left to cool down in a desiccator for 10 minutes after which they will be slowly peeled one by one from the cover sheet first and then from the board carrier. They are checked for picking and adhesion when being peeled off. In addition, handsheet, cover sheet and board carrier are inspected for surface defects.
- The handsheets are examined by transmitted light and inspected for defects. In addition cover sheet and carrier board are visually inspected.

Depending on the test results, the products are classified as being *recyclable, conditionally recyclable* or *non-recyclable*. Products are *conditionally recyclable* if they have a high proportion of non-paper components, if they are not sufficiently defibrated or if the handsheets of the screening accept show optical inhomogeneities (dirt specks, transparent spots) found to negatively affect the optical appearance of the sheet. Products will be rated *non-recyclable* if they share of non-paper components or undefibered material exceeds a limit value or if handsheets formed from the screening accept are sticky and would cause damage in the sheet adhesion test.

Assessment of the filtrate:

During recovered paper treatment, coating components may be dissolved or form finely dispersed/colloidal particles that resist separation by screening. These particles are retained in the pulp or they are carried on with the process water along the various thickening and dewatering stages of recycling plants. The highly or totally closed water circuits of modern mills cause such potentially sticky material to accumulate in the form of finely dispersed and colloidal stickies, the so-called microstickies, which may cause deposits in the paper machine with the final consequences of web breaks or product surface defects.

The filtrates of the slushed RCF pulp are used to characterise the disturbing potential of possible dissolved matter (finely dispersed/colloidal potentially sticky contraries – micro stickies). A screen (mesh width 100 µm) is used to separate the fibres/coarsely dispersed particles from the finely dispersed/colloidal particles. Whereas satisfactory measuring methods are available for measuring the coarsely dispersed stickies > 100 µm, the finely dispersed/colloidal stickies still manage to escape reliable measurement. For this reason, several measuring methods will be used in parallel that indirectly supply information about the content, and which point out tendencies with respect to the disturbing potential:

- **Determination of the anionic trash content through polyelectrolyte titration**

In the presence of cationic aids such as those used in papermaking, colloidal and finely dispersed stickies can combine with each other or with anionic trash particles to form agglomerates leading to deposits. Polyelectrolyte titration (cationic demand) to determine the distribution of electric charges in the course of the process has turned out to be a suitable way to detect the overall loads of anionic trash. It allows quantitative determinations of the charge densities of all colloidal substances dissolved in the process water (anionic oligomers and polymers), but no detection of non-ionic hydrocolloids.

- **Chemical oxygen demand**

The chemical oxygen demand (COD) is a sum parameter of the organic substances capable of oxidation. Finely dispersed and colloidal stickies influence the COD to a certain extent. The parameter is more strongly influenced, though, by dissolved sizing agents or adhesives such as starch, for example. There is no direct relationship between potential stickies and COD.

- **Precipitation of anionic substances**

Process described by INGEDE method 06/97 and PTC method PTC-AAW 0002: A predefined amount of filtrate is precipitated with a cationic precipitant. After precipitation, the precipitate will be separated from the filtrate by filtering. The coacervate obtained by filtration is dried in a drying cabinet up to weight constancy. By deducting the o.d. mass of the filtrate w/o precipitation from the o.d. mass of the coacervate, the mass of substances that may be precipitated by adding a cationic polymer is obtained (m_F).

In addition, the appearance of the precipitate is visually assessed and classified into four groups or levels each of which has been assigned a factor f_A. Moreover, the tackiness of the precipitate is classified into five groups assigned the factors f_K. The precipitate m_F and factors f_A and f_K, may be used to calculate a characteristic for the determination of potential secondary stickies according to the formula:

$$\textbf{PSS-Number} = m_F * f_A * f_K * 10^{-6}$$

The method does not permit selective evaluations of potentially sticky finely dispersed and colloidal constituents of mill waters and recycled pulps, but the relatively simple sum parameters determined yield some information on the anionic substances present in the water or stock systems.

- **Deposition behaviour of potentially sticky substances (finely dispersed and colloidal sticky particles)**

The depositing tendency is determined according to INGEDE method 09/99. The method uses the adsorption behaviour on metal plates to simulate the processes in the dryer sections of paper machines. The filtrate containing potentially sticky particles is mixed with fibres (chemical pulp) to form handsheets over a white ribbon filter placed in a frit. The handsheets are produced to obtain a sticky-laden sheet. To simulate on a laboratory scale typical deposition phenomena in a PM dryer section, the filter side of the suction filter sheet is covered with a chromium-plated flat metal plate with mirror finish. After drying, the sheets were carefully peeled off the plate, a test which leaves the plate with more or less stickies attached to it.

For evaluation, the adsorbed hydrophobic particles on the metal plates are stained with a highly selective fluorescent dye for hydrophobic particles. Following this, the fluorescent particles are visually assessed under ultraviolet light in a fluorescence-detecting stereo microscope. The amount of particles adsorbed at the plates and which are visible under the microscope will be assessed qualitatively. According to the test method, they are classified according to the categories 'high', 'medium' and 'low' depending on the degree of adsorption.

The method yields at least general information as to whether the finely dispersed/colloidal particles contained have a depositing tendency and, thus, the potential to form microstickies.

3.2 Sample material

To obtain meaningful results, the recyclability should be studied on finished products (paper products). Therefore, paperboard has to be manufactured as a first step. In addition recyclability studies should always include the comparison with a reference.

Recyclability tests of this work package were done on samples derived from SP2 demonstrator project. The laboratory board samples tested were:

- ‘New concept board’: paperboard with latex addition in the centre ply. The latex binder is used as strength additive in the middle layer (CTMP middle layer) of a 3-ply paperboard.
- Reference board: paperboard produced without extra additions (based on CTMP).

The 3-ply forming was done at Stora Enso, using a Formette Dynamique. The sheets formed were couched together to produce a multiply board. Table 1 shows the materials tested.

Table 1. Sample material

Sample	*Paperboard*	*Produced / provided by*
A	3-ply paperboard, latex addition in centre ply	Stora Enso
B	3-ply paperboard w/o latex addition *Reference sample*	Stora Enso

4 Results

4.1 Assessment of the recyclability (defibration characteristics, undisturbed sheet forming process)

Defibration characteristics:

Table 2. Residue on the perforated plate after screening

Sample	A	B
Residue on the perforated plate (in relation to the weighed-in o.d. sample) [%]	**3.4**	**1.0**

All samples were easily defibrated. The residuals consisted of few long fibres.

Sheet adhesion test:

Table 3. Sticky forming potential - undisturbed sheet formation (handsheets from the screening accept)

Sample	A	B
Adhesive effect	**no**	**no**
Optical inhomogeneities	**no**	**no**

The sheet adhesion test did not show an adhesive effect for any of the samples tested.

Table 4. Assessment of the recyclability within the scope of the specifications set forth in the PTS Method RH:021/97

Sample	A	B
recyclable		
conditionally recyclable		
non-recyclable due to stickies		

According to the evaluation criteria defined in the recyclability method, samples A and B may be classified as *'recyclable'*.

4.2 Assessment of the filtrate

Anionic trash content:

Table 5. Anionic trash content (polyelectrolyte titration)

Sample	A	B
Consumption of PolyDADMAC [ml]*	**0.19**	**0.20**

Chemical Oxygen Demand:

Table 6. Chemical Oxygen Demand (COD)

Sample	A	B
mg/l	**663**	**299**

Precipitation of anionic substances:

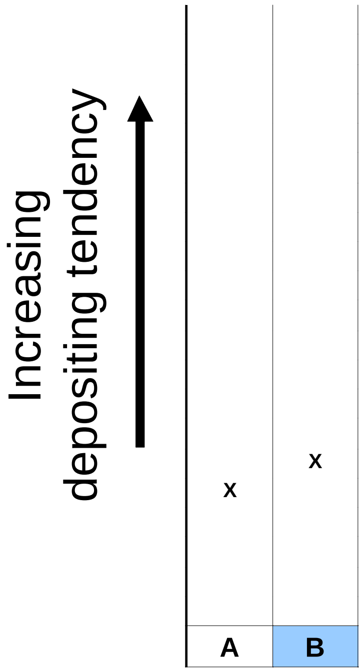
Table 7. Precipitation of anionic substances

Sample	A	B
Precipitate [mg/l]	**13**	**24**

Sample	A	B
PSS-Number	**0.13**	**0.12**

Deposition behaviour of potentially sticky substances:

Table 8. Deposition behaviour



To evaluate the depositing tendency of the samples, the plates were visually assessed and compared with each other. The ranking obtained by these comparative assessments reflects merely the tendency of the samples to form deposits.

There is no difference in depositing behaviour between the new concept board and the reference sample without latex in the middle layer. Latex as a paperboard component has thus no influence on the depositing tendency. All in all, the depositing tendency of the samples tested is rather low.

4.3 Discussion of the results

Table 9. Summary: disturbing potential of the samples - potential to form stickies (sheet adhesion test / assessment of the filtrate)

Sample	A	B
Low disturbing potential		
Medium disturbing potential		
High disturbing potential		

All samples were completely defibered in the recyclability tests. No adhesive effect could be determined in the sheet adhesion tests of the handsheets produced from the screening accepts. According to the evaluation criteria defined in the recyclability method PTS: RH 021/97, samples A and B may be classified as '*recyclable'*.

Filtrate tests were conducted to assess the samples' potential to form finely dispersed and colloidal stickies, or the disturbing potential of substances likely to enter the water flow. Regarding the anionic trash contents there were no differences between the sample with latex addition and the reference. Small differences were determined between the newly developed paperboard and the reference regarding COD. The absolute level, though, was still low.

No significant differences were determined between the sample with latex addition and the reference regarding the precipitation of anionic trash. The precipitation levels found were low in general. Regarding the depositing tendencies there is no difference in depositing behaviour between the new concept board and the reference sample without latex in the middle layer. Latex as a paperboard component has thus no influence on the depositing tendency. All in all, the depositing tendency of the samples tested is rather low.

When comparing the individual results of all tests on the basis of our previous experience, the disturbing potential of the 'new concept board' during recycling may be considered rather small. The recycling behaviour is comparable to that of conventional paperboard.