GUIDELINE DOCUMENT:

RECYCLABILITY OF PAPER BASED PRODUCTS

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Guideline document: Recyclability of paper based products

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1. Introduction

Paper represents one of the best recycled material in Europe and a good example how the circular economy may work promoting proximity recycling thus creating new job opportunities at local level. Currently, the statistics[1] show that at European level 71,7% of this material goes back into new paper products. Nonetheless, the quality of this material is clearly affected by some present mega trends. The sharp decline of newspaper consumption in most of the European countries is reducing one of the best known recycled paper products; meanwhile the concomitant increase in the share of paper based packaging products poses new challenges due to the high diversification of these products. In order to retain the currently high paper recycling rate or even improve it in the future, a clearer definition of recycling oriented eco-design is necessary as well as a further development of the life cycle thinking in the whole paper value chain. The quality of the collected paper for recycling has to be considered equally important as the amount of collected paper by local decision makers. Besides, the extended producer responsibility for an effective material recycling shall become a key driver in the decision process of environmentally focused companies.

The collected paper for recycling in Central Europe (CE) accounts for approximately 16 million tonnes, representing about one third of the amount used by the European paper industry. However, the recycling rates are quite different among the CE countries. Some of them are approaching the theoretical limit in collection whereas others still show a significant potential that must be exploited. Learning lessons from best practices is a key point and communication through suitable expert based guidelines is very much relevant to spread correct information thus helping the paper value chain stakeholders to better contribute to the sustainability of the paper recycling loop.

This document gives a brief insight into paper recycling and quality requirements as well as a comprehensive collection of relevant guidelines, assessment schemes and laboratory test methods.

2. Glossary

Deinkability[2]: Removal of ink and/or toner from a printed product to a high extent by means of a deinking process. This shall restore as good as possible the optical properties of the unprinted product.

[1] CEPI – Confederation of European Paper Industries „Key Statistics European Pulp and Paper Industry 2013“
[2] European Recovered Paper Council, European Declaration on Paper Recycling 2011 - 2015

European Recovered Paper Council (ERPC): A committee of the paper value chain in Europe. Members of the ERPC are associations which are either Signatories or Supporters of the European Declaration on Paper Recycling.

Non-paper product materials: Any foreign matter in paper and board for recycling, which is a constituent part of the product and cannot be separated by dry sorting.

Paper and board for recycling[3] (often referred to as "paper for recycling"): Natural fibre based paper and board suitable for recycling and consisting of

- paper and board in any shape,
- products made predominately from paper and board, which may include other constituents that cannot be removed by dry sorting, such as coatings and laminates, spiral bindings, etc..

Paper product[2]: General term used to cover all paper- and board-based converted products.

Recyclability[2]: Design, manufacturing and converting of paper- and board-based products in such a way as to enable a high quality recycling of fibres and minerals in a manufacturing process in compliance – where appropriate – with current standards in the Community: as a minimum, recyclability requires that sufficient information is exchanged for appropriate risk management and safe re-use of fibres.

3. Quality of paper for recycling

Principally, paper for recycling can be divided in three groups. The two main ones are graphic and packaging grades, often referred to as white and brown grades. White grades are used predominantly for the production of graphic papers, some for hygiene papers and white top plies of packaging paper and board. The brown grades find their utilisation in the production of packaging paper and board. Also the mixed grades which mostly are used for corrugated papers or inner plies of boxboard belong to this group. The third group are special grades which usually require special treatment processes. These special grades are defined in group 5 of EN 643.

Quality of paper for recycling has several aspects. One is the composition and the content of unwanted substances expressed as unwanted papers, non-paper components and prohibited materials. This is mainly a function of the collection system and the subsequent handling of paper for recycling. The European standard EN 643 provides a detailed description and definition of the individual grades and their contents. Physical and optical properties of the paper for recycling and

[3] EN 643 – Paper and board – European list of standard grades of paper and board for recycling, January 2014

the level which can be achieved after treatment are a function of the composition and the recyclability. The form of delivery – loose or baled, original shape or shredded – is mainly relevant for handling but can also be a safety issue. Unnecessary shredding should be avoided since it creates dust and reduces fibre length and strength as well as deinking performance[4]. Moisture is mainly a commercial aspect but can become a quality issue if the paper for recycling is extremely wet. Last but not least the recyclability of individual products in paper for recycling is of particular importance.

The implementation of recyclability criteria in ecolabels, especially the latest EU-Ecolabel for printing products, demonstrates the importance of paper products to become a secondary raw material for papermaking.

4. The paper recycling processes

Depending on the kind of paper and board manufactured from paper for recycling, the processes are different. Common and basic process steps are slushing in a pulper and mechanical separation of impurities by screening through baskets or plates with holes or slots and by centrifugal forces in cleaners.

Brown processes often operate deflakers to separate fibre bundles (“flakes”) into individual fibres and refiners to develop mechanical properties. These processes may be combined with a fractionation in order to treat only the long fibre fraction of the pulp. A slight dewatering is usually installed to provide the proper consistency for subsequent treatment, save volume for pulp storage and to separate the water circuits of stock preparation and paper or board machine. Some optional treatment like dispersing or kneading even require high consistency thickening of at least that portion of the pulp which is treated this way.

[4] Faul, A., Geistbeck, M., Klar, A.-K., Deinking Grades of Paper for Recycling – What determines the quality?, CTP-PTS Deinking Symposium, May 2014

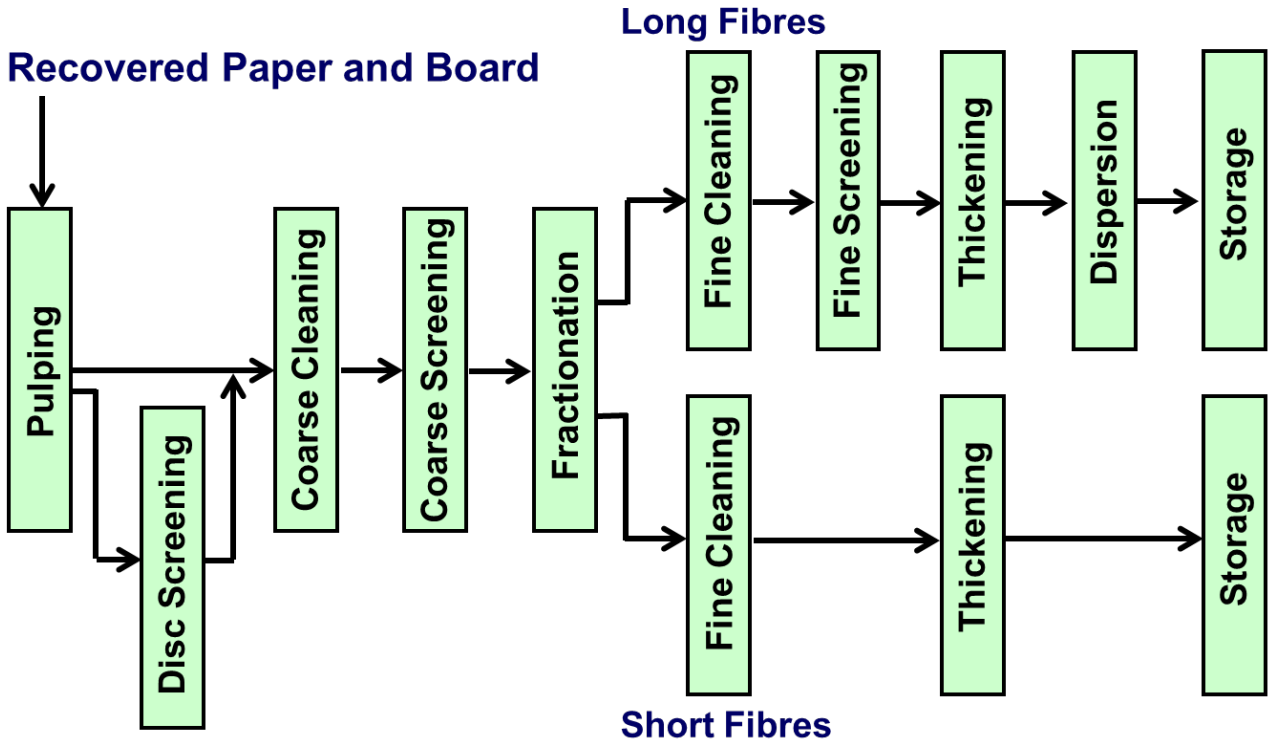

Figure 1: Typical layout for a recycling process to treat mixed and packaging paper for recycling[5]

White processes are typically equipped with a deinking step which is required to remove printing inks and to achieve a bright final pulp. Deinking consists of two stages:

- detachment of inks from the fibres in the pulper, usually with the help of chemical additives (sodium hydroxide, sodium silicate, hydrogen peroxide, soap) and
- separation of the detached ink particles by deinking (in flotation cells or in certain cases in washers).

By far the predominant ink separation process is flotation, mainly due to a significantly higher yield of the process. In the flotation deinking cells the pulp is mixed with air in form of small bubbles which “catch” the ink particles and transport them to the surface where they are skimmed or sucked off. A prerequisite for this flotation process is a hydrophobic character of the ink particles and a certain range of the ink particle size. In European deinking plants, the operation of a disperser for the entire pulp is state-of-the-art, also an internal treatment of the process water. A post-flotation has become common. Deinking plants have at least one high-consistency

[5] Putz, H.-J., Runte, S., Packaging Paper and Board: Raw Materials, Production, Converting and Recyclability, EcoPaperLoop seminar Warsaw, October 2013

thickening in order to separate the water systems of deinking plant and paper machine, which have different pH levels. For higher qualities, one or two bleaching stages are installed.

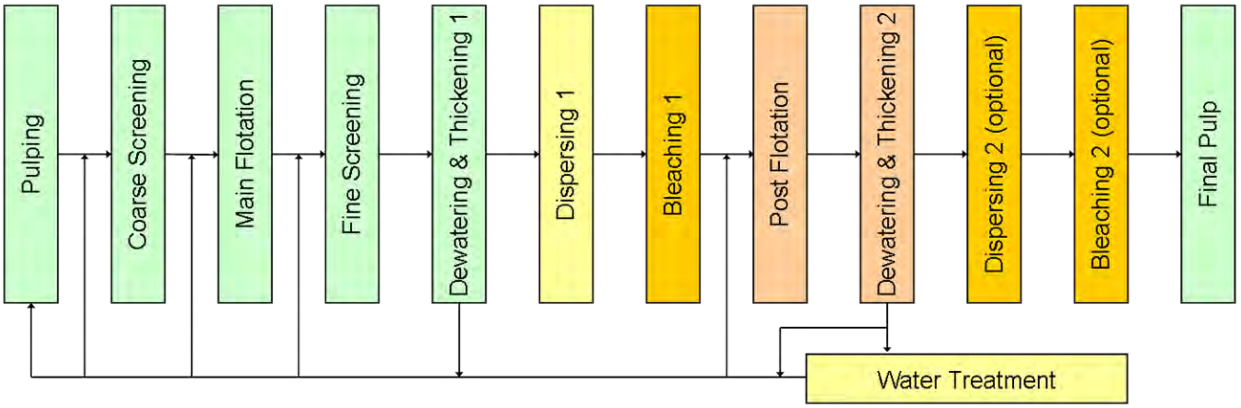

Figure 2: Typical layout for a deinking process (Green: Essential process steps in flotation deinking plants; Green and yellow: 1-loop deinking plant; Green, yellow and brown: Common 2-loop process design for standard grades; Orange: Additional options for higher qualities)[6]

There are some recycling plants utilising specific processes in order to treat special types of paper products which are regarded as detrimental to standard processes or to achieve a special quality of final product. Products which are known as detrimental to standard processes but utilised as raw material for the paper industry are defined as special grades of paper for recycling in group 5 of EN 643.

5. Aspects of recyclability

Recyclability in the context of this guideline document refers to features of individual paper products and should not be confused with the quality of a delivery of paper for recycling (as described in chapter 3).

A good recyclability enables paper and board mills to restore as good as possible the properties of the original paper and board before printing and converting by means of a reasonable process design. “Reasonable” refers to equipment, energy and additives needed as well as to the yield which can be achieved. In addition, the intended use of the recycled products should not be restricted for health & safety reasons.

[6] Faul, A., Oberndorfer, J., The challenge to deink inkjet prints together with recovered paper from households, 9th Research Forum on Recycling, Norfolk, VA (USA), October 2010

The scope of EcoPaperLoop deals with the first aspect, the restoration of the original properties. For all recycled pulps it means that the content of adhesive material ("stickies") should be low. Packaging paper mills are facing issues with high content of non-paper material and insufficient repulpability due to wet-strength additives and laminations. In graphic paper mills the main focus is on the removal of printing inks and varnishes.

6. What the producer can do

This chapter refers to several documents for guidance, assessment, and test procedures. All these documents are attached in full length to this guideline as annex.

6.1 Packaging products

It is obvious that the priority in producing packaging is on the functionality of the product. This objective is not always in line with requirements of a good recyclability. In cases of insufficient recyclability it should be checked whether and how the design can be amended to improve recyclability without affecting functionality. Also an "overdesign" of packaging should be avoided if it has a negative influence on recyclability.

Some paper companies operate plants which are designed to treat paper products which are regarded detrimental to recycling in standard processes. These processes can have a higher tolerance to coarse rejects and to flakes. Or they even utilise the non-paper product materials as valuable by-products.

Stickies, however, are unwanted in every paper recycling process because they can cause problems and downtime of the paper machine as well as quality defects in the manufactured product.

For some applications, a uniform optical appearance is desired. Therefore, this parameter should be considered in an assessment as well.

Chemical ingredients can also play an important role in the usability of the recycled product. A general requirement is to consider alternatives to the use of substances which can act detrimental in downstream recycling processes.

Within the framework of the EcoPaperLoop project, these statements have to remain at a general level. It is recommended that the packaging paper value chain intensifies its dialogue and extends it to recyclability issues in order to develop common guidelines which result to an enhanced recyclability of paper based packaging. The EcoPaperLoop project group drafted a scorecard assessing the parameters coarse rejects, flakes, macrostickies and optical homogeneity. This draft scorecard

was discussed within the value chain and handed over to the European Recovered Paper Council, who will further develop it and possibly adopt a first version in spring 2015.

6.2 Graphic products

The graphic paper value chain started the discussion of recyclability issues in round tables in about 1996. These activities are ongoing in Germany at the Technical Committee Deinking and in Europe at the European Recovered Paper Council. Visible results of this cooperation are the "Guide to an Optimum Recyclability of Printed Graphic Paper" and the scorecards "Assessment of Printed Product Recyclability – Deinkability Score –"[7] and "Assessment of Printed Product Recyclability – Scorecard for the Removability of Adhesive Applications", both published by the ERPC.

The "Guide" describes the graphic recycling process in a more detailed form than this document and points out the obstacles to the process, thus guiding a producer to the materials and procedures which should be avoided. The two scorecards enable everybody in the value chain to assess the deinkability and the removability of adhesive applications of individual graphic paper products. They are based on laboratory tests simulating basic deinking and screening processes.

Producers of print products who apply for an ecolabel on printed products have to prove deinkability and removability of adhesive applications. All relevant ecolabels – the EU Ecolabel (2012/481/EU), the Nordic Swan (Nordic Ecolabelling of printing companies, printed matter, envelopes and other converted paper products), the Austrian Ecolabel (UZ 24 "Druckerzeugnisse") and the German Blue Angel (RAL-UZ 195 "Druckerzeugnisse") – contain criteria on these two characteristics.

7. Test methods on recyclability

The scorecards require test methods on which they are based. For the time being, there are three main methods delivering the results which can be assessed by means of the scorecards. EcoPaperLoop Method 1 is for the assessment of paper based packaging products, the INGEDE Methods 11 and 12 are for graphic products. These main methods need several auxiliary methods for some details of the laboratory procedures.

[7] The Deinking Scorecard was under revision in 2014 and was adopted in October 2014. The published version will describe good deinkable products in an annex which is in the process of being finalised. The layout work and the official publication of the revised scorecard are planned in March 2015. The enclosed "draft" version of the Deinking Scorecard dated on 02 Oct 2014 is – in its contents – identical to the adopted version.

7.1 Main methods

7.1.1 EcoPaperLoop Method 1

For packaging products, the main method is EcoPaperLoop Method 1 “Recyclability Test for Packaging Products”. It requires a large sample size (480 g oven dried packaging product). This ensures that usually an entire product (independent of size) can be tested. The first step after disintegration is coarse screening (10 mm holes). The reject (non-paper product materials and not disintegrated materials) is weighed. The accept undergoes determinations of flake and macrosticky content, the latter according to INGEDE Method 4. The accept of the macrosticky analysis is similar to an industrial final pulp. It is used to form handsheets to assess the optical homogeneity.

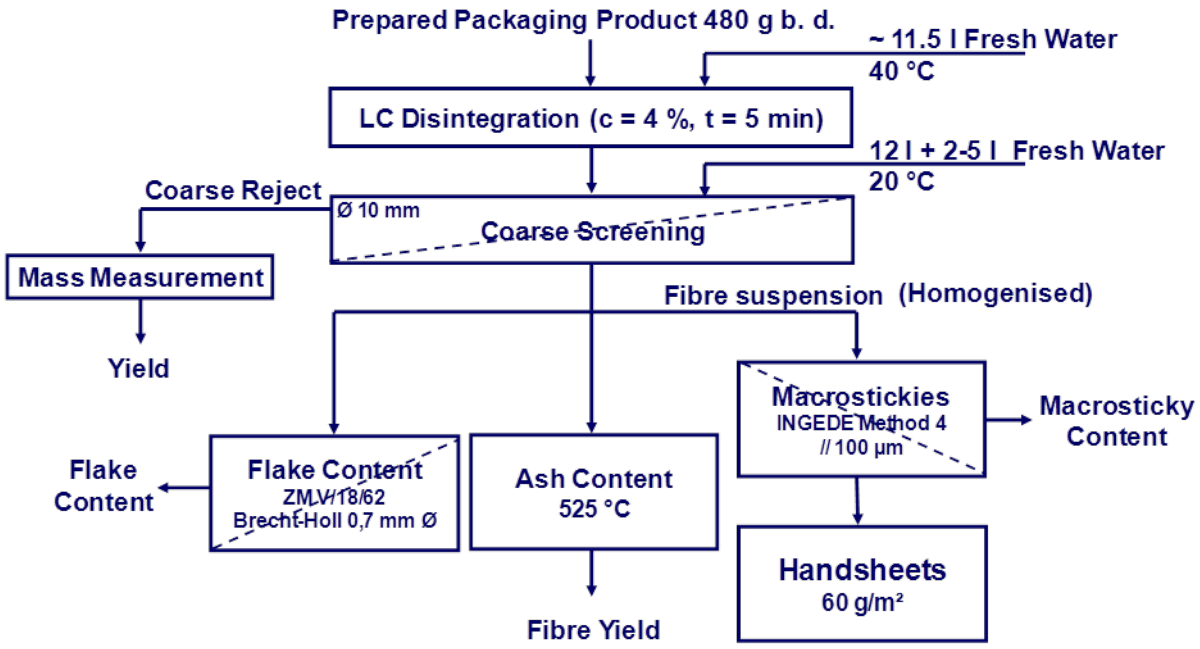

Figure 3: Procedure of the recyclability test for packaging products (EcoPaperLoop Method 1)

7.1.2 INGEDE Method 11

Flotation is the most widely used technology for ink removal in the paper recycling process. This INGEDE Method in a laboratory scale defines the essential steps of the flotation deinking process: pulping and flotation. In order to simulate the average age of paper recovered from households, an accelerated ageing step is part of the procedure. Special care was taken to define a procedure without the need to test unprinted paper. The whole laboratory procedure is shown in Figure 4.

The deinkability is assessed by three quality parameters of the deinked pulp and two process parameters.

Quality parameters	Process parameters
Luminosity	Ink Elimination
Colour shade	Filtrate darkening
Dirt specks (in two different size categories)	

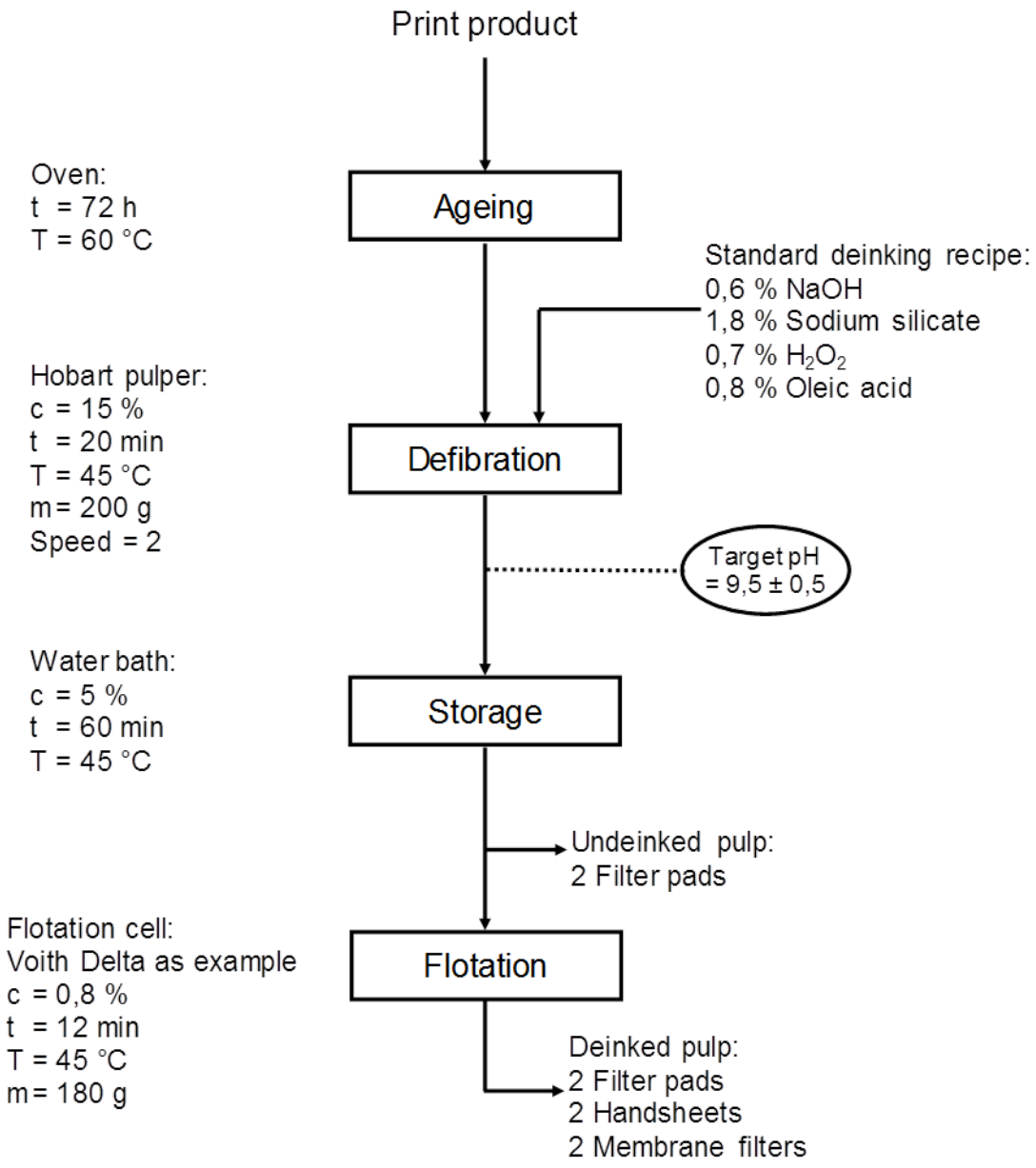

Figure 4: Procedure for testing deinkability with standard deinking recipe

7.1.3 INGEDE METHOD 12

This method is determined to simulate the screening ability of adhesive applications in a deinking process. The two essential process steps are pulping and screening.

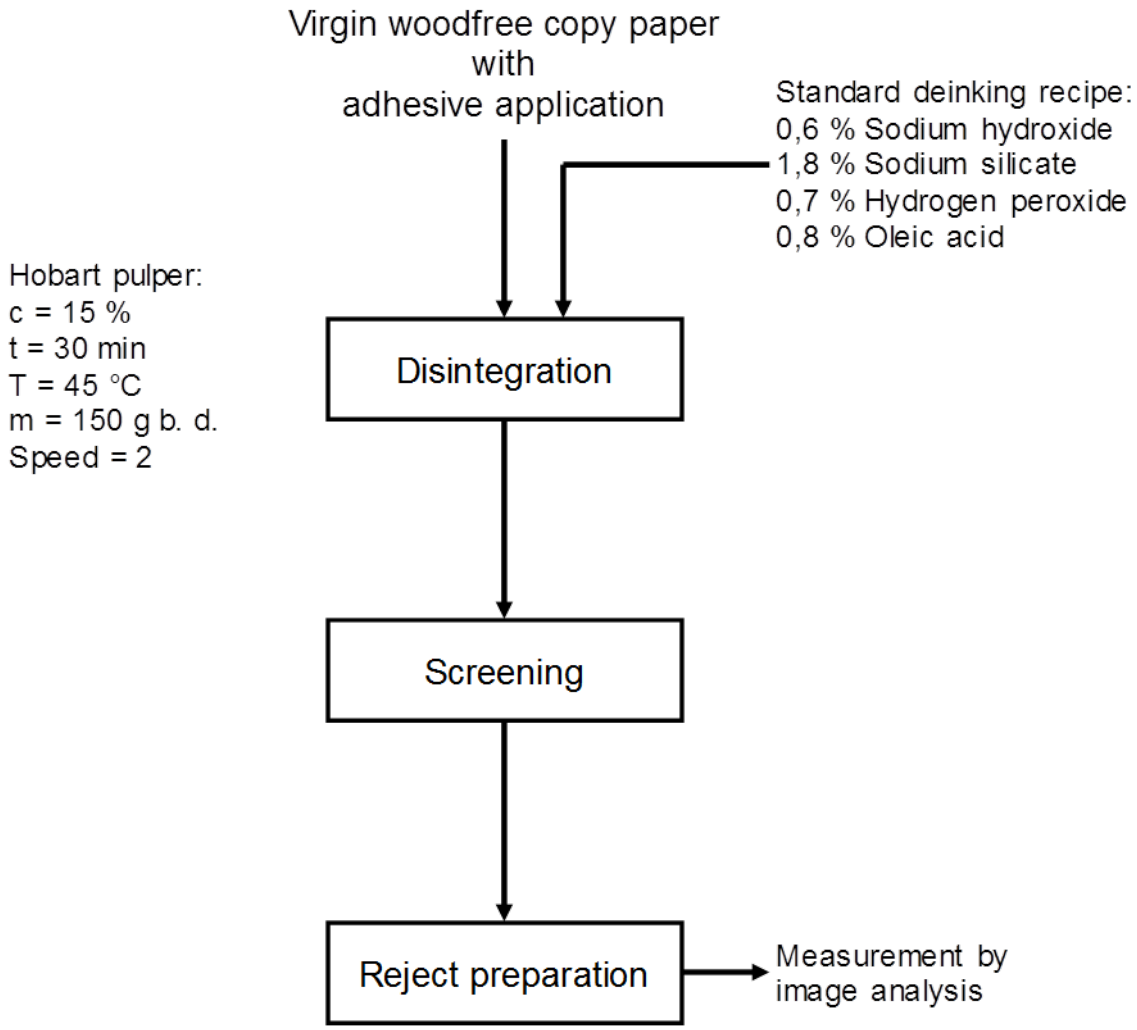

Figure 5: Testing fragmentation behaviour of adhesive applications

The separation of adhesive applications from the pulp is done by screening according to INGEDE Method 4.

The particle size distribution of the macrostickies is measured, thus allowing the assessment of the screening ability of the adhesive applications in an industrial process.

The setting of the screening ability limit of < 2 000 µm equivalent circle diameter was proven in semi-industrial pilot plant trials and confirmed by test results from industrial processes.

7.2 Auxiliary methods

7.2.1 INGEDE METHOD 1

For testing purposes, filter pads are prepared from industrial or laboratory pulp samples using a Büchner funnel and defined filter paper. Handsheets are prepared with the Rapid-Köthen method from industrial pulps under defined conditions. The filtrate samples are drained over a membrane filter and compared with a reference membrane filter made with tap water.

Optical measurements are conducted according to INGEDE Method 2.

7.2.2 INGEDE METHOD 2

Industrial or laboratory samples of pulp and filtrates in deinking processes are transformed to filter pads and handsheets by means of INGEDE Method 1. INGEDE Method 2 describes and defines the parameters and the settings of the measurement devices to obtain results for optical characterisation of the samples. The calculation of the ink elimination is also part of this method and allows an assessment of the deinking process.

7.2.3 INGEDE METHOD 4

The method describes a laboratory screening procedure for pulps of a paper recycling process. The reject of this screening procedure is prepared in such a way that macrostickies can be determined by means of an image analysis system.

8. Annex (with relevant documents):

EcoPaperLoop Method 1 (2014)

Guide to an Optimum Recyclability of Printed Graphic Paper (2008)

Deinking Scorecard (2014)

Removability Scorecard (May 2011)

INGEDE Method 11 (2012)

INGEDE Method 12 (2013)

INGEDE Method 1 (2014)

INGEDE Method 2 (2014)

INGEDE Method 4 (2013)

Recyclability Test for Packaging Products	Leaflet
	Issue: Nov. 2014

1 Introduction

In order to minimise the problems occurring during recovered paper processing, it is essential that packaging products are manufactured considering a good recyclability. In favour of this, the packaging products have to be manufactured for the most part from fibres and must be easy to disintegrate. This increases fibre yield and reduces energy demand as well as the amount of rejects to be disposed. Alike, adhesive applications used for packaging products have to be shear resistant to withstand shear forces during stock preparation processes, and to fragment mostly into particles of adequate size which can be removed during the process.

The following laboratory method defines a procedure to assess the processing of packaging material. For this purpose the content of non-paper product materials, the content of difficult to disintegrate material, the flake content, the macrosticky potential and also the ash content and fibre yield after a disintegration step are investigated. The determined data can be used to assess the packaging product's recyclability. Currently, such a general assessment scheme is not available.

2 Purpose and Application

The purpose of the method is to simulate the behaviour of packaging material during stock preparation in a paper mill. During the investigation, the packaging material is probed considering the content of non-paper product materials, content of difficult to disintegrate material, flake content, macrosticky potential, ash content and fibre yield.

The content of non-paper product materials as well as the content of difficult to disintegrate material and the flake content allow the evaluation of the disintegration behaviour of the packaging material. The non-paper product materials and the content of difficult to disintegrate material form coarse impurities which can stress the coarse screening process in a paper mill. The flake content detects impurities like small plastic parts and primarily fibre bundles which have to be removed during the fine screening steps of a paper mill. The flake content therefore gives information about the load of the industrial fine screening process.

The macrosticky potential is analysed by measuring the macrosticky area. The macrosticky area reflects the load of adhesive impurities within the industrial stock preparation.

The fibre yield is calculated with the yield and the ash content after coarse screening. It allows evaluation of the fibre content of the packaging material.

Handsheets are made from the accept of the macrosticky analysis. They give information about the optical properties of the stock.

3 Definitions

Non-paper product materials
Packaging materials are designed for different functions. For this reason, they are manufactured using a combination of paper and different other materials like plastics or metals. These non-paper product materials can disturb, hamper or avoid the material's recyclability.

Content of difficult to disintegrate material
As several packaging products show a certain water resistance and are more robust during disintegration in water, it is not possible to suspend certain fibre materials into single fibres, instead, fibre bundles remain. Such water resistant packaging materials disturb or hamper the preparation process and the material's recyclability.

Disintegration behaviour
The disintegration behaviour describes how the packaging material can be suspended into single fibres. The disintegration behaviour is analysed by considering the content of non-paper product materials, the content of difficult to disintegrate materials and the flake content.

Flake content
The flake content describes impurities like small plastic parts and primarily fibre bundles.

Yield
The yield describes the amount of usable solid stock material which passes the coarse screening step. By using the ash content a fibre yield could be calculated.

Ash Content
The ash content describes the inorganic content after incineration (525 °C) of the solid stock material which passes the coarse screening step.

Fibre Yield
The fibre yield describes the fibre content of the solid stock material which passes the coarse screening step. It is calculated by using the yield and the ash content.

Macrosticky potential
The macrosticky potential describes the macrosticky area after disintegration of the packaging material.

Handsheets
Handsheets from the accept of the macrosticky evaluation are prepared for visual inspection of the optical properties of the pulp.

4 Principle

This leaflet describes the preparation and investigation with its main steps of sample preparation, disintegration, coarse screening, ash content evaluation, flake content evaluation, macrosticky potential evaluation, yield and fibre yield calculation. For this purpose, a defined amount of the packaging material has to be prepared and then disintegrated at low consistency. The generated suspension has to be screened using a hole screen. The reject on the screen has to be evaluated gravimetrically and the yield has to be calculated. The screening accept has to be homogenised and analysed for flake content using Zellcheming Leaflet ZM V/18/62 [1] or alternatively by an adapted method suitable for the Haindl Classifier. For the macrosticky area the determination follows a macrosticky method based on INGEDE Method 4 [2]. From the accept of the macrosticky screening step handsheets have to be prepared according to ISO 5269-2 [3]. Furthermore from the accept of the coarse screening step the ash content has to be measured according to ISO 1762:2001 [4] and the fibre yield has to be calculated.

The flow chart of the procedure is given by **Figure 1**.

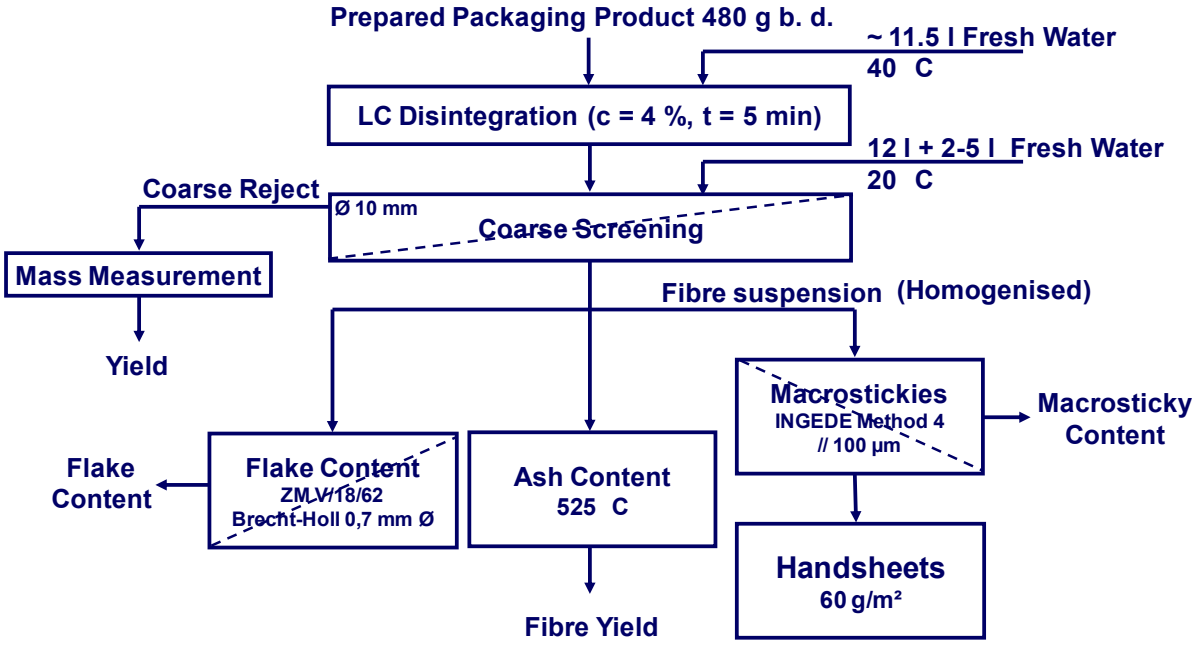

Figure 1: Flow chart of the procedure for the assessment of packaging material recyclability

5 Equipment and Tools

5.1 Disintegration equipment

The disintegration shall be carried out using a low consistency laboratory pulper that can handle a suspension volume of 12 l with a stock consistency of 4 %.

5.2 Coarse screening equipment

The coarse screening is performed utilising a screening device with a 10 mm hole screening plate at the bottom and a volume of 12 l in minimum has to be used. The accept stream of the screen has to be interruptible by an outlet valve. The screening holes have to be kept free during the screening process by using a stirrer. The stirrer blade has to be positioned 10–20 mm above the screen plate and has to run at 200 rpm. As the stirrer has to overcome high resistance forces if excessive coarse rejects are accumulated, the motor has to transmit high moment of torque to the stirrer. For this application the driving motor of a pillar drilling machine is suitable.

5.3 Screening equipment for flake content test

The flake content shall be measured with a Brecht-Holl screening device. The device is described in [5]. Alternatively, a Haindl Classifier can be used.

5.4 Ash determination

The ash content is determined according to ISO 1762:2001(E). - Paper, board and pulps – Determination of residue (ash) on ignition at 525 °C [4].

5.5 Screening equipment for macrosticky test

The macrosticky test must be performed using the screening equipment as described in INGEDE Method 4 [2]. A screening plate with 100 µm slot width is necessary. Using of a Haindl screening device according to ZELLCHEMING Leaflet V/1.4/86 [6] is recommended.

5.6. Equipment for handsheet preparation

Laboratory handsheets are prepared according to ISO 5269-2 [3] using a standard sheet former (model: Rapid-Köthen) with dryer (vacuum 95 kPa, 94 ºC).

5.7 Other Tools

- Distributor for suspension homogenisation
- Garden pump sprayer with atomised spray function
- Analytical balance
- Drying cabinet
- Büchner funnel 150 mm diameter
- Filter paper 150 mm diameter (e. g. Munktell Grade 12/N)
- Filter paper 240 mm diameter (medium to large pores, medium filtration speed, machine finished, good wet strength, white (e. g. Macherey-Nagel MN 617≡Nr.4))
- One sided, silicone-coated release paper (60 g/m²)
- Black water-based ink, e. g. Pelikan No. 4001
- Specially fused alumina powder: white, sharp-edged particles, grain size 220 according to FEPA Method.

6 Sampling and Sample Preparation

6.1 Determination of the adherend proportion

Before disintegration in the laboratory pulper, the dry content of the packaging sample has to be determined as well as the proportion of the adherend. To determine the mass ratio of the adherend, the mass of the air-dry packaging sample has to be measured. Afterwards the entire adherend is cut out tight with all adhesive material and weighed. The ratio between the mass of adherend (plus adhesive) and the mass of the total sample is defined as adherend ratio.

$$X_{Adherend}\ [\%] = \frac{m_{Adherend}\ [g]}{m_{Packaging\ Sample}\ [g]} * 100\ \%$$

$X_{Adherend}$: Adherend ratio in %
$m_{Adherend}$: Adherend mass (adhesive and glued packaging paper) in g
$m_{Packaging\ Sample}$: Total packaging sample mass in g

6.2 Sample preparation

480 g oven-dry material is needed for one investigation. By using the dry content of the samples, the respective amount of packaging products is determined. If a packaging product has to be divided to reach sufficient amount of material, the correct ratio between adherend and non-adherend material has to be maintained. Therefore, parts of the adherend and non-adherend material should be added following the adherend ratio.

Afterwards the complete sample material has to be cut to palm size.

7 Procedure

7.1 Disintegration of the sample material

The palm size cut material has to be filled into the pulper completely adding water of 40 °C temperature. The amount of water has to be calculated in order to reach a disintegration stock consistency of 4 %. The disintegration time is 5 min. After disintegration, the complete sample is removed from the pulper. The sample with a volume of approx. 12 l will be processed further using the coarse screening device.

7.2 Coarse screening

The coarse screening is used to separate large and difficult to disintegrate paper parts as well as large non-paper product materials. The objective is to achieve a nearly fibre free reject. The device consists of a 10 mm hole screen and is defined in Chapter 5.2.

Before starting the process, a container with a capacity of 30 l in minimum is placed below the screening device to collect the screening accept. The outlet valve below the screen is closed. The stirrer is agitated with 200 rpm and has to be operated during the complete screening process. The suspension with the volume of 12 l is

filled in the screening device completely and agitated for 3 more seconds. Then the outlet is opened to start the screening process.

When the suspension is drained completely, the outlet valve is closed, then 12 l tap water are filled into the device. After agitating for 3 more seconds the outlet is opened and the device is drained again.

Then, free fibres still attached to the screening plate or the surface of the device, are drained through the screen using 2–5 l tap water, sprayed using the garden pump sprayer. The water-jet is arranged like spray. The objective is a nearly fibre free reject. Otherwise excessive spraying might dilute the suspension after the coarse screening too much; a very low stock consistency might be problematic for the following tests. Here a good compromise must be found. Therefore it is recommended to use 2–5 l tap water for this step. In exceptional cases up to 10 l tap water can be used for the benefit of a fibre free coarse screening reject.

Then the stirrer is stopped, and the reject on the screening plate is transferred to a weighted and heat resistant case in order to dry the reject until constant weight. The temperature during the drying should be 105 °C. Afterwards the reject mass is determined gravimetrically.

7.3 Yield calculation

The yield can be calculated using the coarse reject as following:

$$Yield\ [\%] = \frac{Packaging\ Product\ used\ [g\ oven-dry] - \ Coarse\ Reject\ [g\ oven-dry]}{Packaging\ Product\ used\ [g\ oven-dry]} * 100\ \%$$

7.4 Homogenisation of screening accept

The accept of the coarse screening must be mixed gently by hand to guarantee a well mixed suspension in order to ensure a homogenous sampling for flake content evaluation, macrosticky determination and ash content measurement. A minimum of 70 g oven-dry pulp sample should be filled directly into a distributor to have a sufficient amount of material for all trials. The pulp is then diluted to a stock consistency of approximately 1 %. After gentle mixing of two minutes minimum, samples for the respective trials can be taken. The distributor stirs until all samples are taken.

7.5 Determination of flake content

The homogenised accept of the coarse screening has to be tested for flake content acc. to ZELLCHEMING Leaflet V/18/62 [1]. In contrast to this method, non-paper product materials like small plastic parts are not removed from the reject on the screen plate but examined as part of the flake content. As screening plate a metal plate with a hole diameter of 0,7 mm has to be used, complying with the requirements of the method. 5 samples with 2 g oven-dry sample material each have to be classified for 5 min using 100 double strokes per minute.

Alternatively to the Brecht-Holl screening device a Haindl Classifier could be used. If a Haindl Classifier is used, a water volume flow of 3,33 l/min or 0,2 m³/h has to be applied.

In the case of a high filter mass and low flake content, negative results for the flake content can occur due to scales accuracy. In such cases, the use of filters with lower mass (e. g. with smaller filter diameter) is recommended.

7.6 Ash content determination

From the homogenised accept of the coarse screening, filters for stock consistency measurement should be prepared and incinerated (525 °C) for ash content determination, following the conditions of ISO 1762:2001(E) [4].

7.7 Fibre Yield calculation

By a combination of yield and ash content the fibre yield could be calculated as following:

$$Fibre\ Yield\ [\%] = \frac{(100\ \% - \ Ash\ Content\ [\%]) \cdot Yield\ [\%]}{100\ \%}$$

7.8 Determination of macrosticky area

The homogenised accept of the coarse screening has to be tested for macrosticky area according to INGEDE Method 4 and to be determined as macrosticky area per kg of packaging material [2]. Therefore, four suspension samples of 10 g oven-dry material are screened over a 100 µm slotted plate.

The screening period per sample is 5 min. The screening is performed in a Haindl device with 480 double strokes per minute. Prior to screening, the suspension samples have to be diluted to a stock consistency of max. 1 %. The complete sample is filled into the Haindl device continuously within the first 5 seconds of the screening.

The reject on the screening plate is then transferred to a paper filter following INGEDE Method 4, stained and visualised. If an overlapping of the residue occurs on the filter, the test has to be repeated, and the residue has to be divided and transferred to several filters. Alternatively the suspension mass can be reduced. In that case more than four samples have to be prepared to maintain sufficient sampling mass. After that the filters have to be finished and evaluated using image analysis, as described in INGEDE Method 4. Also a microscopic inspection of the samples prior to the measurement is necessary. White particles or plastics which are definitively no stickies must be detected and removed or painted over in black so they are not visible any more for the macrosticky image analysis system.

The accept of the macrosticky screening step is used to prepare handsheets.

7.9 Handsheet preparation

An appropriate volume of material for a preparation of handsheets with 60 g/m² should be taken from the accept of the macrosticky screening. As the screening is done with 10 l/min water flow and using 10 g oven-dry pulp, it can be enough to collect the first 15 l accept for the consistency measurement and the two handsheets. After standard laboratory handsheet formation according to ISO 5269-2 [3], drying takes place in the Rapid-Köthen dryer between carrier board and a cover sheet. The

drying time should be 7 minutes. In total, a minimum of two handsheets has to be produced.

Afterwards the handsheets are inspected visually for optical inhomogeneities. The observations should be noted.

8 Report

The results for the reject of the coarse screening step, the calculated yield, the flake content, the macrosticky area, the sticky area size distribution, the ash content and the fibre yield as well as the handsheet observations are summarised in a report. The report must consist of the single results as well as the arithmetical means. All results have to be scaled per kg packaging material. Additionally, the mass of the packaging material, the adherend ratio report and the observations of the handsheets have to be mentioned in the report. Photographs from the used packaging material and the coarse screening reject should be made with a scale for documentation always. If deviations from the above mentioned procedure are conducted, reasons and type have to be noted.

9 References

1. Prüfmethode: ZELLCHEMING Merkblatt V/18/62. (Fachausschuss für Physikalische Halbstoff- und Papierprüfung). Prüfung von Holzstoffen für Papier, Karton und Pappe: Gravimetrische Bestimmung des Stippengehaltes von Stoffsuspensionen.
2. Prüfmethode: INGEDE Method 4. (INGEDE e.V.). Analysis of Macro Stickies in Deinked Pulp (DIP).
3. Norm: ISO 5269/2: Pulp - Preparation of laboratory sheets for physical testing, Part 2: Rapid-Köthen Method.
4. Norm: ISO 1762:2001(E): Paper, board and pulps – Determination of residue (ash) on ignition at 525 °C.
5. Brecht, W.; Holl, M.: Stippengehaltsbestimmung und Faserfraktionierung in einem Gerät. In: Das Papier, 2(1948) Nr. 5-6, S. 85-91
6. Prüfmethode: ZELLCHEMING Merkblatt V/1.4/86. (Fachausschuss für Physikalische Halbstoff- und Papierprüfung). Prüfung von Holzstoffen für Papier, Karton und Pappe: Gleichzeitige Bestimmung des Gehaltes an Splittern und Faserfraktionen.

10. Source of Supply

Pulper, coarse screening device and alumina powder:

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Technische Universität Darmstadt
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64283 Darmstadt
Germany
pmv@papier.tu-darmstadt.de

11. Annexe

Annex 1: Description of the equipment used in this method
Annex 2: Remark to labels with integrated electronic

Annex 1: **Recyclability Test for Packaging Products**	**Leaflet**
	Issue: Nov. 2014

1 Introduction

Subsequently, the equipments to perform the experiments are described in detail, which are used to assess the recyclability of packaging products within the method.

2 Disintegration equipment

As disintegration equipment a low-consistency pulper (LC pulper) has to be used, that can handle a suspension volume of 12 l in minimum with a stock consistency of 4 %. Suitable for this purpose is the disintegration equipment shown in **Figure 1**, which originally belonged to the Escher-Wyss laboratory refiner. The sideways arranged rotor is driven by a 1.5 kW motor with 3,000 min^{-1}. **Figure 2** and **Figure 3** show the pulper and the rotor as dimensioned drawings. Other disintegration equipment must enable comparable fibre separation behaviour.

Figure 1: Picture of LC pulper

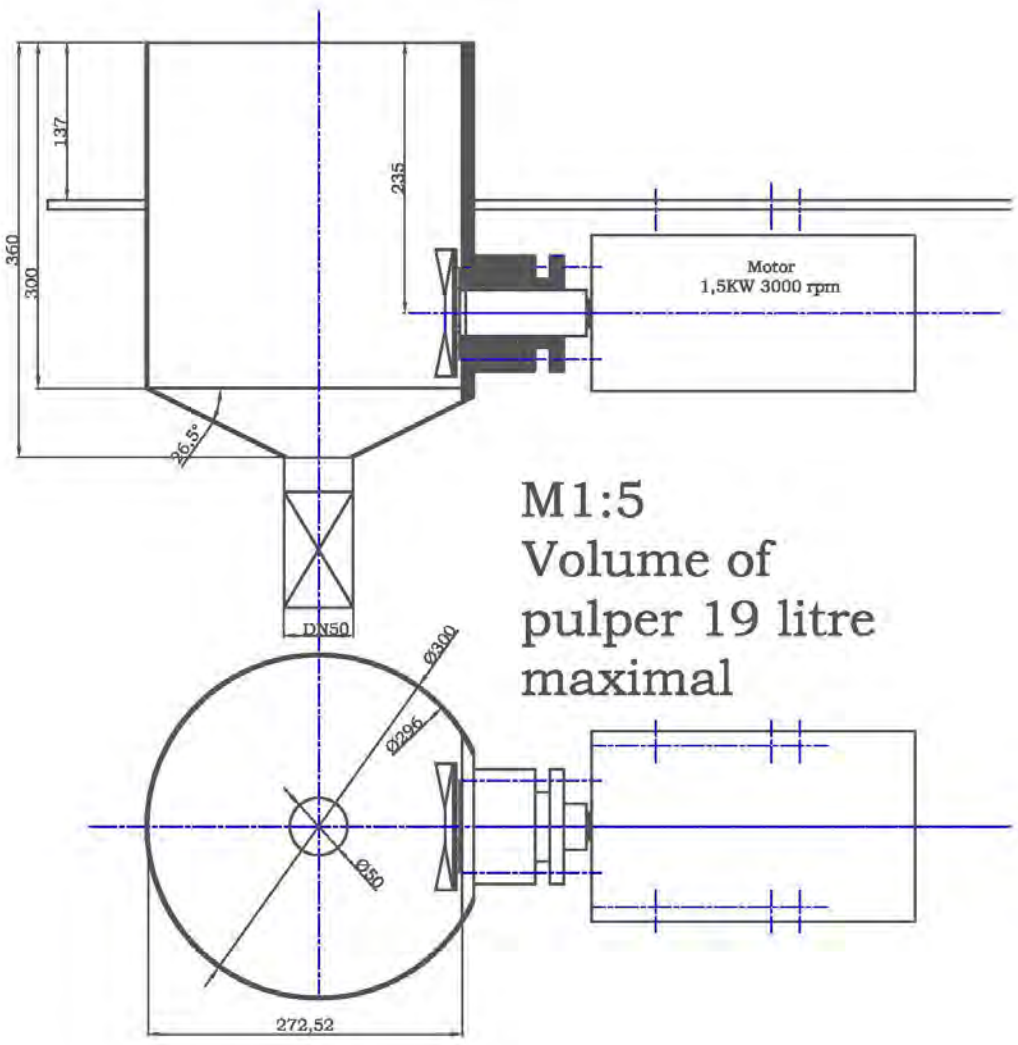

Figure 2: Dimensioned drawing of LC pulper

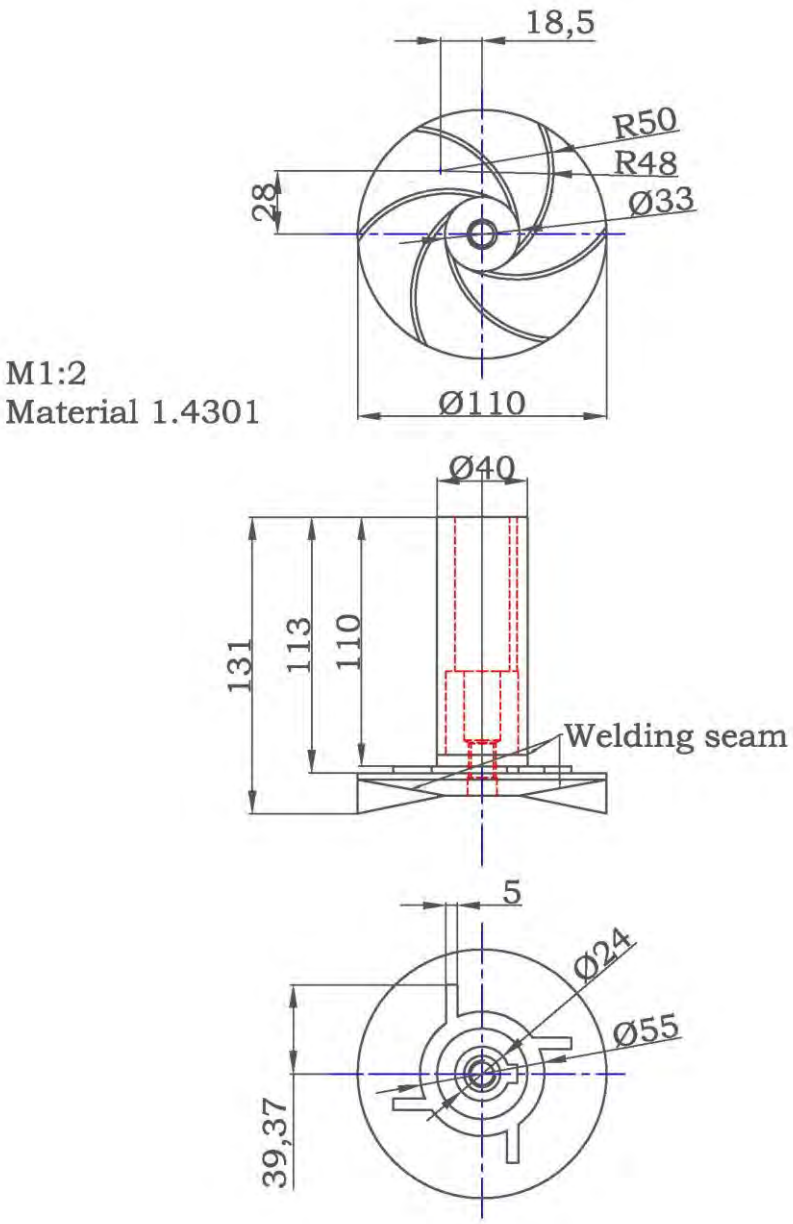

Figure 3: Dimensioned drawing of rotor from LC pulper

3 Coarse screening equipment

For coarse screening a cylinder with a 10 mm hole screening plate at the bottom and a minimum volume of 12 l has to be used (**Figure 4**). The flow through the hole plate must be interruptible by a ball valve. The screening holes have to be kept free during the screening process by using a stirrer. The dimensions of the blade stirrer are shown in **Figure 5**. The stirrer blade has to be positioned 10 – 20 mm above the screen plate and has to run at 200 rpm. As the stirrer has to overcome high resistance forces if excessive coarse rejects are accumulated, the motor has to transmit high moment of torque to the shaft of the stirrer. Therefore the driving motor has to have power of 1.5 kW in minimum. For this application the driving motor of a pillar drilling machine is suitable for example. The basically experimental setup is shown in **Figure 6**.

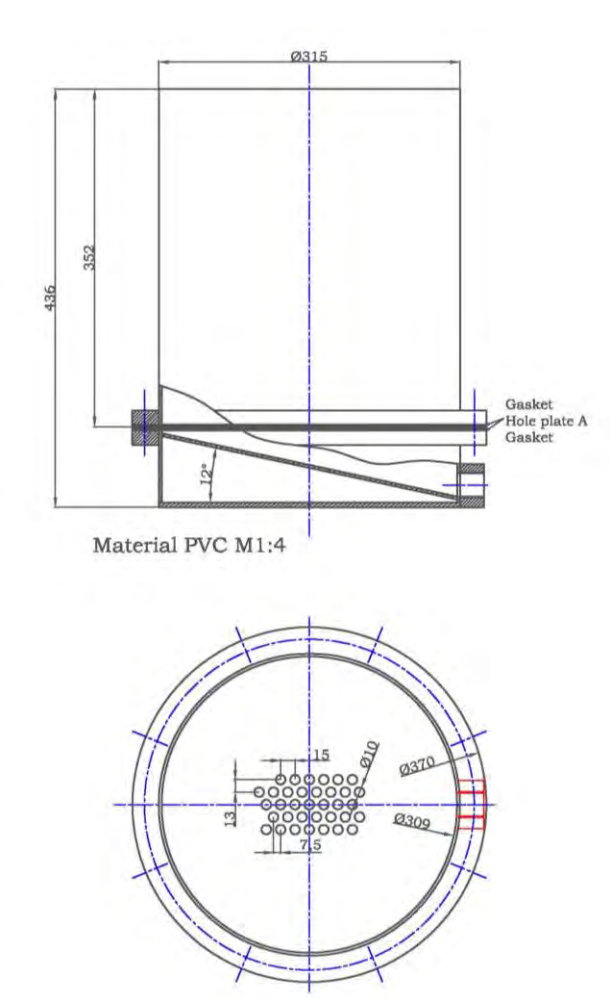

Figure 4: Dimensioned drawing of coarse screening equipment

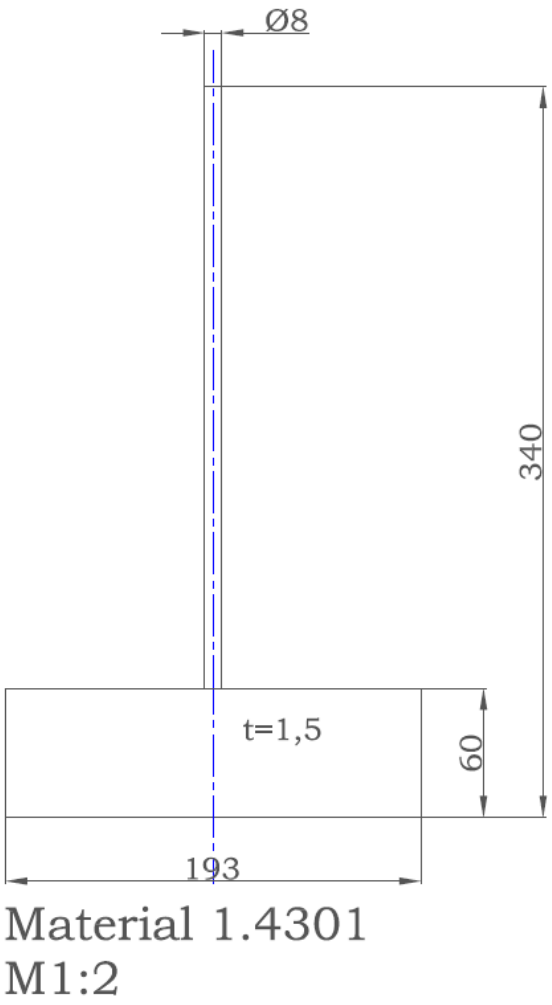

Figure 5: Dimensioned drawing of blade stirrer for coarse screening

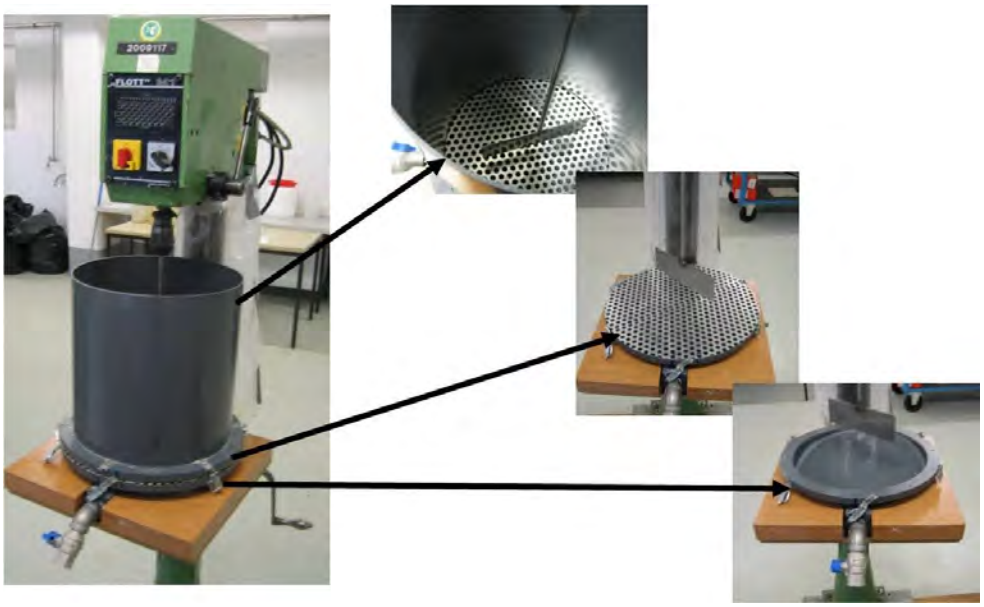

Figure 6: Basic experimental setup in pictures

4 Screening equipment for macrosticky determination

The macrosticky test must be performed using the screening device according to ZELLCHEMING Leaflet V/1.4/86. Using of a Haindl Sorter is recommended. For the intended maximum membrane stroke of 480 double strokes per minute, the device has to be equipped with an extension to operate as a splash guard (**Figure 7**). As screening plate a 100 µm metal slot plate has to be used because plastic slotted plates are not able to hold the mechanical stresses permanently due to the high stroke frequency.

Figure 7: Haindl screening device

5 Screening equipment for flake content determination

The flake content has to be measured with a Brecht-Holl screening device, which is shown in **Figure 8**. A plate with holes of of 0.7 mm diameter has to be used. Alternatively, a Haindl classifier with appropriate hole plate and operating conditions can be used.

Figure 8: Brecht-Holl screening device

Annex 2: **Recyclability Test for Packaging Products**	**Leaflet**
	Issue: Nov. 2014

1 Remark on labels with integrated electronic

Labels with integrated electronics may contain harmful substances, which particularly can be discharged in the disintegration step. Especially with regard to the production of food packaging products, unknown or toxicologically harmful substances should not attain into the paper loop in principle. However, the determination of such substances is not the subject of this leaflet.

Applications, such as labels, shall be examined generally as a whole, insofar their mass fraction has to be rounded up proportionately. The testing of label material has to be done according to INGEDE Method 12 [1]. The labels are applied to 480 g oven-dry packing material. Selection of the packaging material, the amount of applied labels and the application form are based on the real ratio for the concrete purpose.

Subsequently, the packaging material has to be cut into palm size. Labels, especially those with electronic components may not be cut, bent or otherwise damaged prior to the disintegration. Deviations have to be documented in the test report.

Applications, such as labels, affect the recyclability of packaging products in different ways. As a non-paper product material component they can lead to increasing rejects after coarse screening or they can affect the flake content. Adherend parts may cause macrostickies. Applications with harmful substances can disturb sustainable the recyclability of packaging products. Preferably, for the protection of the paper recycling loop this non-paper product material components with harmful substances have to be discharged completely after the first disintegrating step. With regard to a recycling-oriented design of paper and cardboard packaging products, it has to be mentioned in the test report to which extent the applied applications are not damaged after disintegration and are separated as reject after coarse screening. It must also be mentioned in the report, which applications or parts of it attain into the screened pulp suspension.

2 Literature

1. INGEDE Method 12. - Assessment of the Recyclability of Printed Paper Products - Testing of the Fragmentation Behaviour of Adhesive Applications.

Guide to an Optimum Recyclability of Printed Graphic Paper

I. Introduction

This paper deals with the recycling of recovered graphic paper, for the production of graphic paper and other white papers. For brown packaging, other recycling techniques apply.

In recent years the recycling of recovered paper in the production of graphic papers and other white papers has increased considerably.

Today recovered paper is, in terms of quantity, the most important raw material for the European paper industry. Now, in particular newsprint consists increasingly of recycled graphic paper. The treatment of recovered paper starts with the separation of non-paper components, and is followed by the removal of the printing ink in the flotation deinking process. The share of printing ink in average recovered paper mixtures amounts to about 2% by weight. However yields of de-inked pulp (DIP) are only between 75% and 85%, because besides the printing ink and adhesives, fragments of paper fibres and parts of the mineral fillers and coating pigments are also removed.

The result of the recovered paper treatment depends on many factors (e.g. quality of the paper, type of printing process, properties of the printing ink, etc). Moreover the ageing process and climatic conditions during the life cycle of the print products can influence the result.

In many countries it has recently become increasingly difficult in deinking pulp mills to maintain the customary standards of yield and brightness of DIP. The reasons for this are manifold:

- **The increasing collecting rates throughout Europe and the systems used for the collection of used paper destinated to be deinked are a challenge for the deinking industry. There is a danger that the requirements of recovered paper quality are not met; e.g. due to higher shares of board or aged products.**
- **The increase in the recycling of recovered paper leads to lower shares of virgin fibres in recovered paper.**
- **The trend in newspaper printing to apply growing quantities of ink onto ever thinner paper brings an unfavourable quantitative ratio of ink / paper.**

To make up for these unfavourable developments, equipment used in deinking plants is constantly being extended. However to maintain the achieved standard, it is also necessary that everyone involved in the paper chain - including parties placing the order and designers of print products - give due consideration to the requirements of recycling. In the European Declaration on Paper Recycling 2006 – 2010 all major stakeholders in the paper value chain committed themselves to act accordingly.

II. Processes

Various process steps must be evaluated in the technical process of graphic recovered paper treatment.

1. Separation of non-paper components

As a matter of principle, operators of deinking plants see non-paper components in an unfavourable light, because they increase waste quantities. However, quite often, such components cannot be avoided. To impede the de-inking process as little as possible, the following requirements are important:

- Non-paper components should be dimensioned and mechanically stable in such a way that they survive as large particles, without being comminuted, in the conditions of pulping and allow mechanical separation by means of punched screens, slot screens and centrifugal purifiers. Relevant examples are cover foils, staples, thick adhesive layers, various product samples.

- Materials applied in very small dimensions or disintegrating into very small parts are unfavourable because they cannot be removed using today's conventional sorting methods.

2. Detachment of the printing ink film

The next step is to remove the printing ink film from the paper fibres. In the case of prints on coated paper there is, of course, no contact between printing ink and paper fibres. Here in general no problems arise, because the paper coating disintegrates as the recovered paper is pulped and fragments of the ink film are released. On uncoated paper the adhesion of printing ink to paper depends, firstly, on paper properties such as surface structure, fibre type, ash contents, etc and, secondly, the drying mechanism of the chosen printing process.

Printing inks, which form firmly sticking, tenacious printing ink films are more difficult to remove from the fibre. Examples are inks drying by polymerisation (oxidative drying, radiation curing). The ageing of offset inks based upon oxidative drying materials can also significantly reduce the deinkability.

3. Soluble and redispersable components

Components in the recovered paper, which dissolve in the process under standard conditions of deinking (pH 8 - 10) and reach the process water, pose a risk of unintended spreading to all parts of the paper machine. Problems occur when sticky residues - stickies - form upon redrying. In principle, these stickies have to be removed by tedious manual work, causing downtime, or by additional cleaning equipment, reducing the lifetime of equipment and paper machine clothing. A typical way in which stickies form is the agglomeration of dispersed or dissolved auxiliary materials, e.g. water-soluble or redispersable adhesives, paper-coating binders, coatings, varnishes and printing ink constituents. A similar – albeit very rare – problem arises when dyes from paper or printing ink dissolve initially in water and subsequently move onto clean paper fibres.

The requirement therefore is that recovered paper should contain as few components as possible, which dissolve or disperse in weakly alkaline medium and form sticky residues or cause discolourations.

4. Flotation

Flotation, which is the most common process currently used in Europe, is the essential step to remove printing inks. Supported by surface-active substances, printing ink particles gather on the surface of air bubbles. This process works at an optimum with printing ink particles sized between 20 - 100 µm. Thus, the loaded air-bubbles streams upward through the paper pulp. On the surface of the flotation cell, a dark foam segregates, which contains printing ink, fragments of paper fibre, fillers and paper-coating pigments. Particles smaller or bigger than the optimum particle size are floated with less efficiency.

In some cases water-based printing inks are used for flexo-newspaper printing. These inks may contain binders soluble in the alkaline range. Consequently in deinking, such inks do not break up into fragments of printing ink film but into pigment particles, smaller than 1 µm in size. These particles are much too small for flotation.

Printing ink particles too large for the flotation process occur in cases of tenacious, crosslinked ink films in thick layers on coated paper. For example, this problem can arise in connection with coated papers and UV inks or conventional sheet-fed offset inks coated with UV varnishes. When such coarse printing ink particles are obtained, the paper mill still has the option of comminuting them in a disperser and floating them once again. However, this 2nd deinking loop makes the process more complex and increases the rejects.

Likewise, paper mills whose furnish contains a proportion of water-borne flexo newsprint and therefore particles too small to float, often utilise an optional washing cycle. However this is usually only necessary when the proportion of water-borne flexo newsprint exceeds 5% of the total recovered paper, but washing is not reasonable for recovered papers with high mineral content, e. g. magazines.

III. Recyclability assessment

Development and design of printed products are dynamic. Materials and processes, too, are subject to technical innovations. Therefore it is necessary that all parties involved evaluate their products as to good recyclability if major changes are made to materials and processes.

Solutions are available to the various problems highlighted in this guide. These solutions must be examined in each individual case. In this examination, additional criteria, e.g. production quality, economic efficiency, environmental protection, occupational safety, etc have to be included in the assessment.

Institutes and paper mills throughout Europe have developed assessment methods. With the help of these methods it can be estimated whether printed products meet the criteria of recyclability. ERPC recommends using its assessment scheme “Deinkability Scores”. Harmonisation of schemes to assess the removal ability of adhesive applications is recommended.

When assessing whether the criteria of recyclability have been fulfilled, the relevance of the quantity of the examined print product must be taken into account with regard to its deinking performance and the final properties of the recovered substrate.

IV. Recovery of residues from the deinking process

The paper industry is eager to reuse residues generated in recovered paper treatment or to find external possibilities of reuse. Technical and economically feasible options are available. Here it is important that individual constituents do not impair the reuse of residues.

V. Updating

Statements made in this guide will be reviewed and revised if necessary.

Addendum

Non Impact Printing Inks

The quantity of printed office paper in collected paper for recycling is growing at a rate of 20% per annum. Most of this paper is printed by non-impact printing methods such as photocopiers, laser printers and inkjet printers.

Inks used in photocopiers and laser printers are often referred to as ‘toners’ and are often in a dry fine powder form.

Toners are coloured thermoplastic polymers that are usually based on pigments (not dyes). They contain low levels of additives used to help confer electrostatic properties, but essentially their fusing/fixing properties are of greatest interest in the recycling process, and are dominated by the thermoplastic polymer.

In normal use, particles of the dry toner are developed onto a photoreceptor and transferred to paper. At this stage the toner is still in the form of discrete particles, ~10µm in size. The paper then passes through some form of high temperature fusing system and this is where the problem arises, in terms of eventual recycling. During the fusing process the toner polymer melts, wetting and adhering to the paper fibres. At the same time the discrete particles merge forming much larger solidified ‘lumps’ depending on the size of the image. The toner is then well bonded to the paper fibres.

Some toners bond large numbers of paper fibres together which do not float in the flotation process and consequently are retained in the DIP causing a ‘speckling’ problem much like in the case of UV inks. Likewise, paper-mills whose furnish contains a proportion of recovered paper from offices have the option to break them down in a disperser and repeating the flotation process again.

Ink jet inks, commonly used on paper and found in office waste are usually water based dye types. The inks contain little or no resin component and the dye is completely water-soluble. In the flotation cell the dye redissolves and cannot be separated and subsequently moves onto the paper fibres as described in section 2.3. The recommendation is therefore the same, that recovered paper should contain as few components as possible that may cause discolouration.

[September 2008]

SIGNATORIES

June 2009

www.cepi.org
www.paperrecovery.org

www.citpa-europe.org

www.erpa.info

www.europeantissue.com

www.ingede.org

www.intergraf.org

www.fepe.org

SUPPORTERS

www.eadp.org

www.eupia.org

www.enpa.be

www.feica.org

www.faep.org

www.radtech-europe.com

www.finat.com

European Recovered Paper Council
c/o CEPI aisbl
Confederation of European Paper Industries
250 Avenue Louise, Box 80
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Tel: +32 2 627 49 11 Fax: +32 2 646 81 37
mail@cepi.org
www.cepi.org www.paperonline.org
www.paperrecovery.org

If you want to help us develop paper recycling in Europe, why not include the following email tagline in your own email signature:

"When you print this email, please recycle it. Paper is renewable, recyclable and the natural support of ideas. www.paperonline.org"

Design by Ben Timmers (CEPI)

European Recovered Paper Council Draft 2 October 2014	**Assessment of Printed Product Recyclability – Deinkability Score –**	**ERPC Logo**

1 Introduction

Deinking, the removal of printing inks, is a major step in the recycling process of printed graphic products to produce a bright pulp suitable for a wide range of recycled papers and board.

For an efficient functioning of the circular economy it is valuable that products can be recycled to similar quality levels as the original products. It is therefore desirable that printed products are deinkable. If they are not deinkable, printed products can be recycled to lower grade paper and board.

The deinkability of a printed product as a whole can be assessed by only looking at its Deinkability Score, which can range from -100 to +100. For individual products this is done by using the rating of the results given in this specification or by comparing the Deinkability Scores of several printed products.

If a more thorough technical / scientific evaluation has to be made, the individual scores or the measured values of the deinkability parameters can be used.

Ecolabels for printed matter on European and National levels require a positive deinkability result. In many cases, this can be achieved by choosing the proper printing technology and material combination. For these cases, exemptions for deinkability tests are defined in an annex to this Scorecard. This annex is subject to review and revision according to new knowledge gained.

2 Scope

This document of the European Recovered Paper Council provides an assessment of the deinkability of a printed product by evaluating results of a laboratory deinking test procedure. It is applicable to all kinds of printed graphic products on white paper.

3 Principle

Results of deinkability tests achieved by means of INGEDE Method 11 are converted into Deinkability Scores. For each of the five parameters – luminosity, colour, cleanliness, ink elimination and filtrate darkening – threshold and target values are defined. Cleanliness is measured as dirt speck area in two particle size classes. The threshold and target values are depending on the category of the printed product. If the result meets the target value or is better, it scores the maximum points allocated to this parameter. The maximum points achievable for each parameter are different thus indicating the importance of each individual parameter. A score below 0 in one or more parameters leads to the overall assessment “not suitable for deinking”.

4 Determination of the Deinkability Score

In this chapter, particularly in the tables, abbreviations for the assessment parameters are used:

Y: Luminosity
a*: Colour a* (green – red) of the CIELAB system
A: Dirt particle area
A_{50}: Dirt particle area for particles larger than 50 µm (circle equivalent diameter)
A_{250}: Dirt particle area for particles larger than 250 µm (circle equivalent diameter)
IE: Ink elimination
∆Y: Filtrate darkening

Rounding of the parameters: Y, IE and ∆Y to whole numbers, a* to one decimal and A to one decade. The individual scores of each parameter are rounded to whole numbers as well. Method: financial rounding.

4.1 Source of the deinkability results

The results of deinkability tests have to be obtained according to INGEDE Method 11. The fibre yield of the laboratory flotation, determined as yield of organic components, should be at least 65%. . If that value is not reached, the test has to be repeated with reduced flotation time.

For the determination of IE the parameter R_{700} has to be used with the term $\left(\frac{(1-R_{\infty,unpr})^2}{R_{\infty,unpr}}\right)$ set to 0.

For the image analysis, DOMAS or Simpatic are allowed.

4.2 Weighting of the parameters

The assessment of deinkability consists of five parameters. Three of those – luminosity, colour and cleanliness – refer to the quality of the deinked pulp, the other two – ink elimination and filtrate darkening – are process parameters. The quality parameters have a higher maximum score than the process parameters, which serve as a kind of backup for the assessment. The split of the evaluation of cleanliness in two size classes of the dirt speck area gives a total of six single scores.

Table 1: Maximum score for each parameter

Parameter	Y	a*	A_{50}	A_{250}	IE	∆Y	Total
Maximum Score	35	20	15	10	10	10	100

4.3 Threshold values

For a good deinkability, the values for Y and IE have to be high, the values for A and ΔY have to be low. The parameters with a desired high value have a lower threshold, the parameters with a desired low value an upper threshold. The a* value has both thresholds because the result should be within a target corridor. Falling below a lower threshold, exceeding an upper threshold, as well as falling out of a threshold corridor, results in a negative score for this parameter.

The thresholds are not comparable to the actual industrial quality requirements; they are by far less challenging due to a wide safety margin. This is because INGEDE Method 11 is not a complete simulation of the industrial deinking process; the assessment is to determine the relative challenge a printed product means for a flotation deinking plant. This margin also takes variations in the test procedure into account.

Printed products in the category "Low ink coverage products (Brightness of base paper > 75)" typically are produced using woodfree uncoated or coated papers. They usually end in grades of paper for recycling of groups 2 and 3 – medium and higher grades according to EN 643. These grades are used by mills producing deinked pulp with high optical quality requirements. Products in the categories "Newspapers", "Magazines, uncoated", "Magazines, coated" and "Low ink coverage products (Brightness of base paper ≤ 75)" typically are produced using mechanical pulp based or recycled papers. After use, these products predominantly end in grades of paper for recycling which are used in deinking plants with lower optical quality requirements. Therefore it is possible to have the same threshold in these four categories but necessary to increase the thresholds for the high quality requirements.

Table 2: Threshold values for "Newspapers", "Magazines, uncoated", "Magazines, coated" and "Low ink coverage products (Brightness of base paper ≤ 75)"

Parameter	**Y** [Points]	**a*** [-]	**A_{50}** [mm²/m²]	**A_{250}** [mm²/m²]	**IE** [%]	**ΔY** [Points]
Lower Threshold	**47**	**-3.0**			**40**	
Upper Threshold		**2.0**	**2.000**	**600**		**18**

Table 3: Threshold values for "Low ink coverage products (Brightness of base paper > 75)"

Parameter	**Y** [Points]	**a*** [-]	**A_{50}** [mm²/m²]	**A_{250}** [mm²/m²]	**IE** [%]	**ΔY** [Points]
Lower Threshold	**67**	**-3.0**			**40**	
Upper Threshold		**2.0**	**2000**	**600**		**18**

4.4 Target values

Each parameter has a target value depending on the product category.

Table 4: Target values

Category of printed product	Y [Points]	a* [-]	A_{50} [mm²/m²]	A_{250} [mm²/m²]	IE [%]	ΔY [Points]
Newspapers	≥ **60**	≥ **-2.0** to ≤ **+1.0**	≤ **600**	≤ **180**	≥ **70**	≤ **6**
Magazines, uncoated	≥ **65**				≥ **70**	
Magazines, coated	≥ **75**				≥ **75**	
Low ink coverage products (Brightness of base paper ≤ 75)	≥ **70**				≥ **70**	
Low ink coverage products (Brightness of base paper > 75)	≥ **80**				≥ **75**	

Note: Brightness measurement is done according to ISO – R457 (without UV).

Definitions and examples for the product categories:

Newspapers:

Written publication containing news, information and advertising, usually printed on low-cost paper called newsprint

Inserts, flyers & brochures – with an ash content of less than 22%: Leaflets for advertising.

Directories: Telephone books and similar types of printed products.

Magazines:

This category comprises a variety of printed products. They are distinguished in two sub-categories, depending whether the base paper is uncoated or coated.

Magazines: Publications which are generally published on a regular schedule, containing a variety of articles, generally financed by advertising, by a purchase price, by pre-paid magazine subscriptions, or all three.

Inserts, flyers & brochures – all coated ones; if uncoated, with an ash content of 22% or higher: Leaflets for advertising.

Catalogue: Publication containing a list of merchandise from a company, often in a similar fashion as any magazine.

Books with high ink coverage

Low ink coverage products:

Products, which are typically printed on high grade paper and/or with significantly lower ink coverage than magazines.

In this category belong text only prints, transactional and transpromotional prints, formal or personal correspondence, one side printed products, low ink coverage books and the like.

In case of doubts whether a printed product is a Low ink coverage product, the determination can be made by measuring the grey scale value, if necessary as average of several pages which should be representative for the printed product. If the grey scale value is above 200 (on a scale of 0 to 255), the product is regarded as Low ink coverage product. Procedure: A print sample is scanned by the scanner used for DOMAS with the equipment's scan software. For the scan 24 bit and 600 dpi (all other settings: standard settings) will be used and the file is saved in jpeg format. The median grey value of the complete scan (sample with paper margin but no scanner header) is measured (e.g. with the freeware "imagej"). Calculation of the grey scale value is done by arithmetic average of the RGB values.

4.5 Determination of the Deinkability Score

It is recommended to use spreadsheet software to calculate the score. The INGEDE Office can provide the formulae in Microsoft Excel® format.

4.5.1 Calculation of the score per parameter

Results of the individual parameters which meet or exceed the target values receive the maximum scores for these parameters (according to Table 1). "Exceeding the target values" means:

- In case of Y and IE: higher than the target value
- In case of A and ΔY: lower than the target value
- In case of a*: between higher and lower target value

If this is not the case, the score has to be calculated. For each individual parameter, the ratio of units better than the threshold value, divided by the range between threshold and target values, multiplied by the maximum score for this parameter, gives the Deinkability Score for this parameter. All individual scores are rounded to whole numbers by financial rounding.

Calculation for one individual parameter:

$$DS_P = \frac{(R_P - TH_P)}{(T_P - TH_P)} \cdot MS_P$$

Where

The index letter P stands for one of the six results Y, a*-value, A_{50}, A_{250}, IE and ΔY

DS_P is the Deinkability Score of the parameter P

RP is the result of the parameter P

TH_P is the threshold value of the parameter P (according to Table 2 or Table 3)

TP is the target value of the parameter P (according to Table 4)

MS_P is the maximum score of the parameter P (according to Table 1)

Example: Deinkability Score DS_Y for the luminosity of DP from newspapers

Luminosity Y of DP: 55

Threshold TH_Y: 47

Target T_Y: 60

Maximum score MS_Y: 35

$$DS_Y = \frac{(55-47)}{(60-47)} \cdot 35 = 22$$

The DS is limited to the maximum score MS for each individual parameter, even if the calculation gives a higher result. In that case it is not possible to compensate a weak deinkability in one parameter with a very good deinkability in another parameter.

If the result is worse than the threshold, the score is negative for this parameter. In that case the absolute number is limited to the same value as the maximum score for this parameter.

If the value a* is above the higher target value, the upper thresholds and targets have to be used in the formula – and vice versa if it is below the lower target value.

4.5.2 Calculation of the Deinkability Score

For a complete evaluation of the deinkability, the six individual scores are added. If one or more individual scores are negative, the assessment of the printed product is “not suitable for deinking”. However, the product may be well recyclable for a process without deinking.

If a product is assessed as “not suitable for deinking” due to negative scores of one or more parameters, the scores of the parameters with positive results are not displayed.

Note (Ink Elimination):

In case of Low ink coverage products the determination of the Ink Elimination IE can become inaccurate. If IE is the only parameter which causes a printed product to fail, the ink coverage should be artificially increased and the test repeated. Increased ink coverage with analogue prints can be achieved by cutting unprinted portions off from the test samples In case of digital printers a print pattern with higher ink coverage should be chosen. In seldom cases in which the ink coverage cannot be increased, e. g. at note pads with ruling only, the assessment should be done with the help of the other parameters. In these cases the Score for IE will be set to 10 points.

Table 5: Examples (Newspapers)

Parameter	Y	a*	A_{50} (DOMAS)	A_{250} (DOMAS)	IE	ΔY	Deinkability Score / Assessment
Threshold	47	-3 / +2	2.000	600	40	18	
Target	60	-2 / +1	600	180	70	6	
Maximum score	35	20	15	10	10	10	
Sample A							
Result	55	-2.5	450	220	60	8	good
Score	**22**	**10**	**15**	**9**	**7**	**8**	**71**
Sample B							
Result	45	-2.0	200	120	32	12	**not suitable for deinking**
Score	**-5**	**20**	**15**	**10**	**-3**	**5**	**-8**
Sample C							
Result	60	-1.6	150	90	75	5	good
Score	**35**	**20**	**15**	**10**	**10**	**10**	**100**

5 Rating of the results

In order to give the user an idea of the relevance of the Deinkability Scores, they should be assessed according to the following table:

Table 6: Rating of the Deinkability Scores

Score	Evaluation of deinkability
71 to 100 Points	Good
51 to 70 Points	Fair
0 to 50 Points	Tolerable
negative (failed to meet at least one threshold)	Not suitable for deinking (may be recyclable without deinking)

Experience has shown that in cases of poor deinkability not all results of the individual parameters are bad. If the most critical parameter is just slightly better than the threshold, the scores of the other parameters usually give already a sum of about 50 points. Therefore a Deinkability Score of up to 50 points is regarded as “tolerable”.

In charts, coloured backgrounds as in the table above should be used whenever possible. In order to reflect the assessment above, the colours should be set as follows:

- Below 0 points: red
- 0 to 40 points: orange
- 40 to 50 points: transition orange to yellow
- 50 to 70 points: yellow
- 70 to 80 points: transition yellow to green
- 80 to 100 points: green

6 Generic testing

Typically for the assessment of print product recyclability in the case of the EU Ecolabel licencing and similar (including R&D purposes), it is not always possible or appropriate to provide a genuine print product for testing. A generic testing can therefore be performed on dummies, i.e. a reference product case[1]. Results of the assessment for the reference product case will be valid for all related print products bearing the same features as the tested dummy, i.e. the same technical data and material

[1] *European Commission’s Ecolabel User’s Manual for the application for printed paper of March 2013 refers to a “reference case, which will allow to submit further orders under the limit set by the reference case”.*

combination with the same or lower ink coverage (for each ink). The related printed products will therefore not require further laboratory deinking test procedures.

For the use of the tested printed product as a dummy, all certificates will state the following:

"These test scores are also valid for printed products with the same or lower ink and varnish coverage."

Generic tests can be performed on combinations of inks and types of paper, allowing printers to select pre-tested combinations suitable for deinking according to the ERPC scorecard.

7 Exemptions to the deinkability test

Many printed products are deinkable and will pass the deinkability test. The criteria for which printed products can be exempted from testing are defined in an annex of this document. This annex is subject to review and revision according to new knowledge gained.

8 Report

The report should contain detailed data of the printed product, the printing process and the deinking test:

- Identification of printed product as to name, publishing company, date of issue, product category, print process and paper quality.
- Printing parameters and press settings.
- Name and exact identification of inks or toner.
- Results of the deinking test according to INGEDE Method 11.
- The laboratory equipment used for the deinking test and deviations from INGEDE Method 11, if any.
- Deinkability Scores for every parameter and total (total only if all six individual scores are 0 or higher). The results can be provided either numerical or as graphics. For a graphic presentation column stacked charts are recommended. If at least one element of the stacked columns points to the negative side, this product is rated "not suitable for deinking", even if the other elements are positive. In order to avoid confusion, in case of "not suitable for deinking", only the negative columns are displayed in charts.
- Assessment of the deinkability according to Table 6.
- Optional but desired: Any interpretation of the result which is possible with the help of the technical data.

9 References

- EN 643 – European list of standard grades of paper and board for recycling
- INGEDE Method 11 – Assessment of Print Product Recyclability – Deinkability Test –

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Assessment of Printed Product Recyclability

Scorecard for the Removability of Adhesive Applications

1 Introduction

The assessment of recyclability of printed paper products has several aspects. Two major ones are removability of adhesive applications and deinkability. The removability of adhesive applications of a printed product can be assessed by looking at its Removal Score, which can range from -20 to +100.

The sufficient removal of adhesive applications is one of the challenges for the paper manufacturers using recovered paper. In the recovered paper treatment process, adhesive applications disintegrate during pulping to “stickies”. Stickies is a broad term for all tacky components in recovered paper pulp. Depending on their size and their behaviour they are called macrostickies, microstickies or potential secondary stickies. Mechanical screening with slotted screens is the most efficient tool for sticky removal. High removal efficiency can only be achieved if adhesive applications disintegrate into particles of large size. The smaller the particles are, the lower their removal efficiency is. In addition, they can re-agglomerate later in the papermaking process and thus form secondary stickies which lead to major problems in paper production and/or converting processes.

In this regard, the removability depends not only on the composition of the adhesive but also on the type of application, such as the shape of the application and the thickness of the layer. The larger and thicker the layer of a given adhesive, the less disintegration into small particles occurs. In any case, the particle size of stickies has a certain distribution. The applied method for testing – INGEDE Method 12 – can detect particles of 100 µm size and larger.

Investigations have proven that macrostickies above a particle size of 2 000 µm are completely removed in state-of-the-art paper recycling processes. It is the objective to have a low total area of macrostickies which has to be expected after industrial screening. This area therefore can achieve up to 80 points in this assessment scheme. The higher the share of macrostickies below 2 000 µm, the higher the danger is of having many stickies below the detection limit of the method. Therefore the share of macrostickies below 2000µm has a threshold at 50%. Lower shares are rewarded with up to 20 points.

Literature: see concluding remarks.

2 Scope

This ERPC document provides an assessment of the removability of adhesive applications of a printed paper product as one aspect of its recyclability. The assessment is done by evaluating results of a laboratory test procedure. It is applicable to all kinds of printed paper products containing any adhesive applications.

3 Principle

This assessment scheme deals with the fragmentation of adhesive applications and their removability by a laboratory screening process. It serves as an evaluation for potential sticky problems at the paper machine and quality defects in the produced paper or board.

The assessment refers to complete printed products, disregarding which number and type of adhesive applications it contains. INGEDE Method 12 defines the details of the test procedure.

Results of macrosticky measurements achieved by means of INGEDE Method 12 are converted into Removal Scores. There is a threshold defined for the share of the macrostickies below 2 000 µm (equivalent circle diameter). A share above this threshold results in a negative score and is assessed as "insufficiently removable". The area below 2 000 µm particle size has a scoring limit. By allocating removal efficiencies to the different sticky size classes in industrial screening the theoretical macrosticky content of the pulp after screening is calculated. If this value exceeds the scoring limit, the parameter macrosticky area receives 0 points. This happens also if the share of the macrosticky area fails to meet the threshold.

For both parameters – share and area – target values are defined. If the result meets the target value or is better, it scores the maximum points allocated to this parameter.

4 Determination of the Removal Score

In this chapter, particularly in the tables, abbreviations for the assessment parameters are used:

A_t	**Theoretical macrosticky area in the pulp after industrial screening**
S_{2000}	**Share of macrosticky area below a particle size of 2 000 µm (equivalent circle diameter)**
T_A	**Target value for the theoretical macrosticky area A_t**
T_S	**Target value for the share of macrosticky area S_{2000}**

Rounding of the parameters: A_t to one decade, S_{2000} to whole numbers. The individual scores of both parameters are rounded to whole numbers as well. Method: financial rounding.

4.1 Source of the removability results

The test results have to be obtained according to INGEDE Method 12. For the image analysis, DOMAS or Simpatic are allowed.

4.2 Removal efficiency of the different size classes

One of the factors which defines the efficiency of industrial screening processes is the particle size distribution of macrostickies. The larger the macrostickies, the better their removal efficiency by screening is. Based on results of research projects and evaluation of industrial samples the screening efficiency can be determined as in Table 1.

Size class of macrostickies (equivalent circle diameter)	Removal efficiency
< 600 µm	0 %
600 µm to 1 000 µm	20 %
1 000 to 2 000 µm	80 %

Table 1: Removal efficiency as function of the macrosticky particle size

4.3 Weighting of the parameters

The assessment of removability consists of two parameters. It is beneficial for the paper recycling industry that the total amount of macrostickies is low. Therefore the amount receives a significantly higher score than the share.

Parameter	S_{2000}	A_t	Total
Maximum Score	20	80	100
Minimum Score	-20	0	-20

Table 2: Maximum score for each parameter

If printed products contain adhesive applications but no macrostickies can be detected, the reject of the laboratory screening has to be assessed. There are two extreme cases thinkable – either the adhesive is still attached to a medium (label, tape, etc.) or it is not detectable. In the first case the product will receive the full score for both parameters. In the second case it has to be assumed that all stickies are of low particle size. These products receive scores of -20 for S_{2000} and 0 for A_t.

4.4 Threshold value and scoring limit

Exceeding the upper threshold of S_{2000} results in a negative score for this parameter. Exceeding the scoring limit of A_t results in the score of 0 for this parameter.

Parameter	S_{2000} [%]	A_t [mm²/kg product]
Scoring limit	n/a	5 000
Upper Threshold	50	n/a

Table 3: Threshold value and scoring limit

4.5 Target values

Both parameters have target values.

Parameter	T_S [%]	T_A [mm²/kg product]
Target values	≤ 10	≤ 500

Table 4: Target values

Special case: Labels

Normally, adhesive applications can only be assessed properly after their application on a printed product. Exceptions are labels which do not represent a final product but their application can be easily simulated. See INGEDE Method 12 for details. Since a label is usually not recycled as a pure material, this assessment defines a projection of the macrostickies content originating from label applications. In cases of sticker covers of magazines and of address labels on envelopes, the share of the complete labels (paper plus adhesive) is about 2,5 % of the complete product. Based on this, the amount of macrostickies in mm²/kg determined by the test according to INGEDE Method 12 has to be divided by 40.

This calculation tool should only be used if no real finished product is available.

4.6 Determination of the Removal Score

It is recommended to use spreadsheet software to calculate the score. The INGEDE Office can provide the formulae in Microsoft Excel® format.

4.6.1 Calculation of the score per parameter

Results of the individual parameters which meet or exceed the target values receive the maximum scores for these parameters (according to Table 2). “Exceeding the target values” means that the result has to be lower than the target value.

If this is not the case, the score has to be calculated by linear interpolation. For both parameters individually, the ratio of units better than the scoring limit respective threshold value, divided by the range between scoring limit respective threshold and target values, multiplied by the maximum score for this parameter, gives the Removal Score for this parameter. The individual scores are rounded to whole numbers by financial rounding.

Calculation of the Removal Score for the share of macrostickies below 2 000 µm:

$$RS_S = \frac{(50 - S_{2000})}{(50 - T_S)} \times 20 \qquad \textit{(Formula 1)}$$

Where

RS_S is the Removal Score for the macrosticky share

50 is the threshold value of the share of macrostickies below 2 000 µm (equivalent circle diameter; according to Table 3)

S_{2000} is the share of the macrosticky area below 2 000 µm (equivalent circle diameter)

T_S is the target value of the share of macrostickies below 2 000 µm (equivalent circle diameter; according to Table 4)

+20 and *-20* are the maximum and minimums score of the share of macrostickies below 2 000 µm (according to Table 2)

Calculation of the theoretical macrosticky area in pulp after screening:

Note: The following calculations are only necessary if the score RS_S for the share of macrostickies is 0 or higher. If the score RS_S for the share of macrostickies is below 0, the score RS_A for the macrosticky area is set to 0.

$$A_t = A_{600} + A_{1000} \times 0{,}8 + A_{2000} \times 0{,}2 \qquad \textit{(Formula 2)}$$

Where

A_t is the theoretical macrosticky area after industrial screening in mm²/kg printed product

A_{600} is the macrosticky content in the size classes below 600 µm (equivalent circle diameter)

A_{1000} is the macrosticky content in the size classes between 600 µm and 1 000 µm (equivalent circle diameter; 0,8 corresponds to a screening efficiency of 20 %)

A_{2000} is the macrosticky content in the size classes between 1 000 µm and 2 000 µm (equivalent circle diameter; 0,2 corresponds to a screening efficiency of 80 %)

Calculation of the Removal Score for the macrosticky area:

$$RS_A = \frac{(5000 - A_t)}{(5000 - T_A)} \times 80 \qquad \textit{(Formula 3)}$$

Where

RS_A is the Removal Score for the macrosticky area

5000 is the scoring limit of the macrosticky area (according to Table 3)

A_t is the theoretical macrosticky area after screening in mm²/kg printed product

T_A is the target value of the macrosticky area in mm²/kg printed product (according to Table 4)

+80 and *0* are the maximum and minimum scores for the macrosticky area (according to Table 2)

If the value A_t is higher than the scoring limit (5 000 mm²/kg), the score RS_A is set to 0.

The Removal Score is limited to the maximum score for each individual parameter, even if the calculation gives a higher result. In that case it is not possible to compensate a weak recyclability in one parameter with a very good recyclability in another parameter.

If the result is worse than the threshold, the score is negative for this parameter. In that case the absolute number is limited to the same value as the maximum score for this parameter.

4.6.2 Calculation of the Removal Score

If the score RS_S for the macrosticky share is negative, the score RS_A for the macrosticky area is set to 0 and the assessment of the printed product is "insufficiently removable". If the score RS_S for the macrosticky share is 0 or higher, both individual scores RS_S and RS_A are added.

5 Rating of the Results

In order to give the user an idea of the relevance of the Removal Scores, they should be assessed according to the following table:

Score	Evaluation of removability
71 to 100 Points	**Good**
51 to 70 Points	**Fair**
0 to 50 Points	**Tolerable**
Negative (failed to meet the threshold)	**Insufficient**

Table 5: Rating of the Removal Scores

In charts, coloured backgrounds as in the table above should be used whenever possible. In order to reflect the assessment above, the colours should be set as follows:

- Below 0 points: red
- 0 to 40 points: orange
- 40 to 50 points: transition orange to yellow
- 50 to 70 points: yellow
- 70 to 80 points: transition yellow to green
- 80 to 100 points: green

6 Report

The report should contain detailed data of the printed product, the process for applying the adhesives and the laboratory screening test:

- Identification of printed product as to name, publishing company, date of issue, product category, type of adhesive applications and paper quality.
- Technical data and settings of the adhesive application device.
- Name and exact identification of adhesives.
- Results of the recyclability test according to INGEDE Method 12.
- The laboratory equipment used for the recyclability test and deviations from INGEDE Method 12, if any.
- Removal Scores for both parameters and total (total only if both scores are 0 or higher). The results can be provided either numerical or as graphics. For a graphic presentation column stacked charts are recommended. If the score of the share of macrosticky points is negative, this product is rated as “insufficiently removable”, even if the score for the macrosticky area is positive.
- Assessment of the recyclability according to Table 5.
- Optional but desired: Any interpretation of the result which is possible with the help of the technical data.

7 Concluding remarks

This assessment was developed with results from INGEDE Project 129 09 PMV which was also supported by bvdm, FEICA and FINAT. The data collected was from books, brochures, catalogues and labels.

There are numerous literatures on stickies, their origin, their behaviour in the paper recycling process and their impact on runnability and quality. One example is: Putz, H.-J., Stickies in recycled fiber pulp, chapter 11 of: Göttsching, L. and Pakarinen, H. (editors), Recycled Fiber and Deinking, Fapet Oy 2000, ISBN 952-5216-07-1.

8 References

- INGEDE Method 12 – Assessment of the Recyclability of Printed Paper Products – Testing of the Fragmentation Behaviour of Adhesive Applications
- Terminology of Stickies, ZELLCHEMING Technical Leaflet RECO 1, 1/2006

Annex: Examples

Characterisation of the examples:

A. Book with protein, EVA and PVAc adhesives

B. Telephone directory with EVA hotmelt adhesives

C. PSA paper label with UV acrylic, non tackified adhesive

D. Book with PVAc dispersion adhesive

Parameter / Sample	Example A	Example B	Example C*	Example D	Remarks
Macrosticky area by size class [µm]	mm²/kg	mm²/kg	mm²/kg	mm²/kg	
100 - 200	143	33	35	10	
200 - 400	340	59	23	11	
400 - 600	442	68	459	15	
600 – 1 000	1 000	208	4 315	16	
1 000 – 2 000	836	599	33 058	30	
2 000 – 3 000	127	388	30 162	0	
3 000 – 10 000	3 402	1423	15 419	0	
10 000 – 20 000	0	19 043	0	0	
20 000 – 200 000	0	13 244	0	0	

Table 6: Macrosticky size distribution (determined by INGEDE Method 12)

* Example C: Results obtained by means of INGEDE Method 12 are already divided by 40 (see chapter 4.5)

Size distribution of macrostickies (according to INGEDE Method 12)

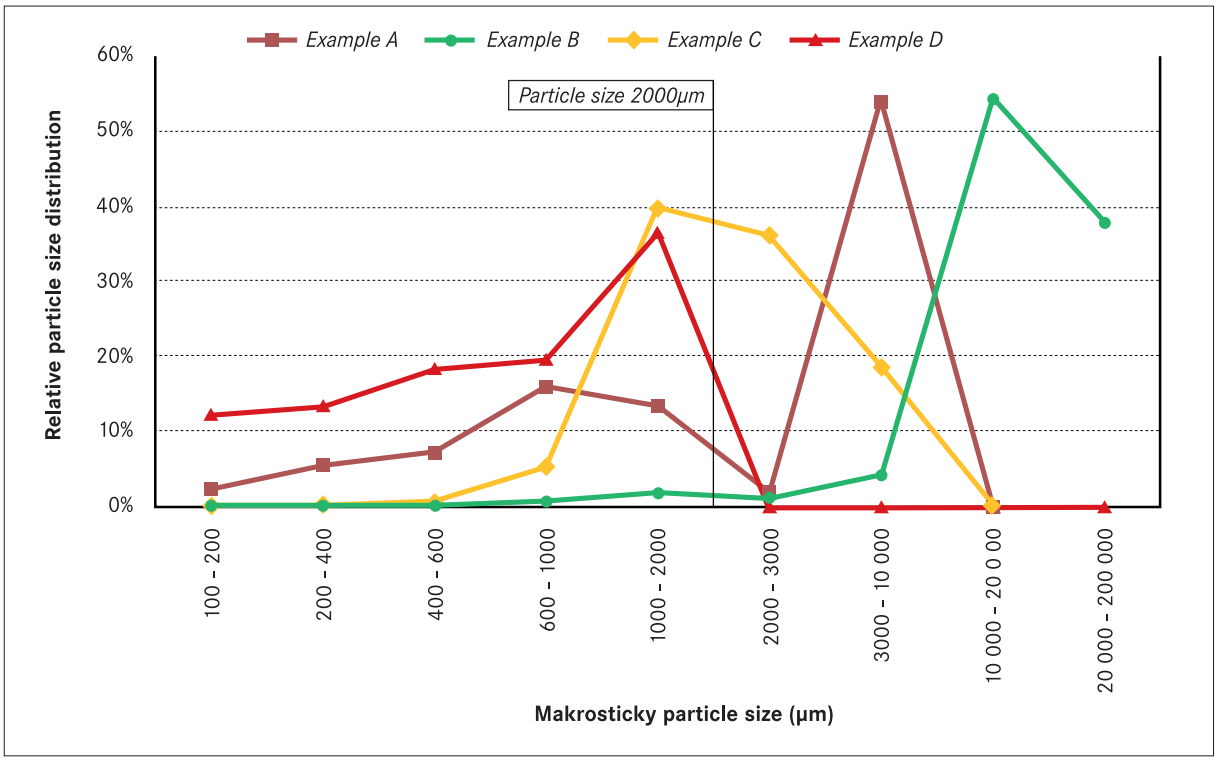

Figure 1: Macrosticky size distribution

Parameter / Sample	Example A	Example B	Example C	Example D	Remarks
Macrosticky area by size class [µm]	mm²/kg	mm²/kg	mm²/kg	mm²/kg	
100 - 600	925	160	517	36	A_{600}
600 - 1 000	1 000	208	4 315	16	A_{1000}
1 000 - 2 000	836	599	33 058	30	A_{2000}
100 - 2 000	2 761	967	37 890	82	***Subtotal 2000***
100 - 200 000	6 290	35 065	83 471	82	***Total***

Table 7: Subtotals and totals of Table 6

Parameter / Sample	Example A	Example B	Example C	Example D	Remarks
Share of macrosticky area below 2 000 µm S_{2000}	44%	3%	45%	100%	Calculation: ***Subtotal 2000*** divided by ***Total*** (Table 7)
Theoretical macro-sticky area after screening A_t [mm²/kg]	1 892	446	10 581	55	See Formula 2

Table 8: Calculation of auxiliary parameters

Parameter / Sample	Example A	Example B	Example C	Example D	Remarks
Share					
Threshold for the share	50%	50%	50%	50%	
Target for the share T_s	10%	10%	10%	10%	
Maximum score for the share	20	20	20	20	According to Table 2
Score for the share RS_S	3	20	2	-20	See Formula 1
Area					
Scoring limit for the area	5000	5000	5000	5000	
Target for the area T_A	500	500	500	500	
Maximum score for the area	80	80	80	80	According to Table 2
Score for the area RS_A	55	80	0	0	
Total Score					
Removal Score	58	100	2	-20	$RS_S + RS_A$

Table 9: Score calculation

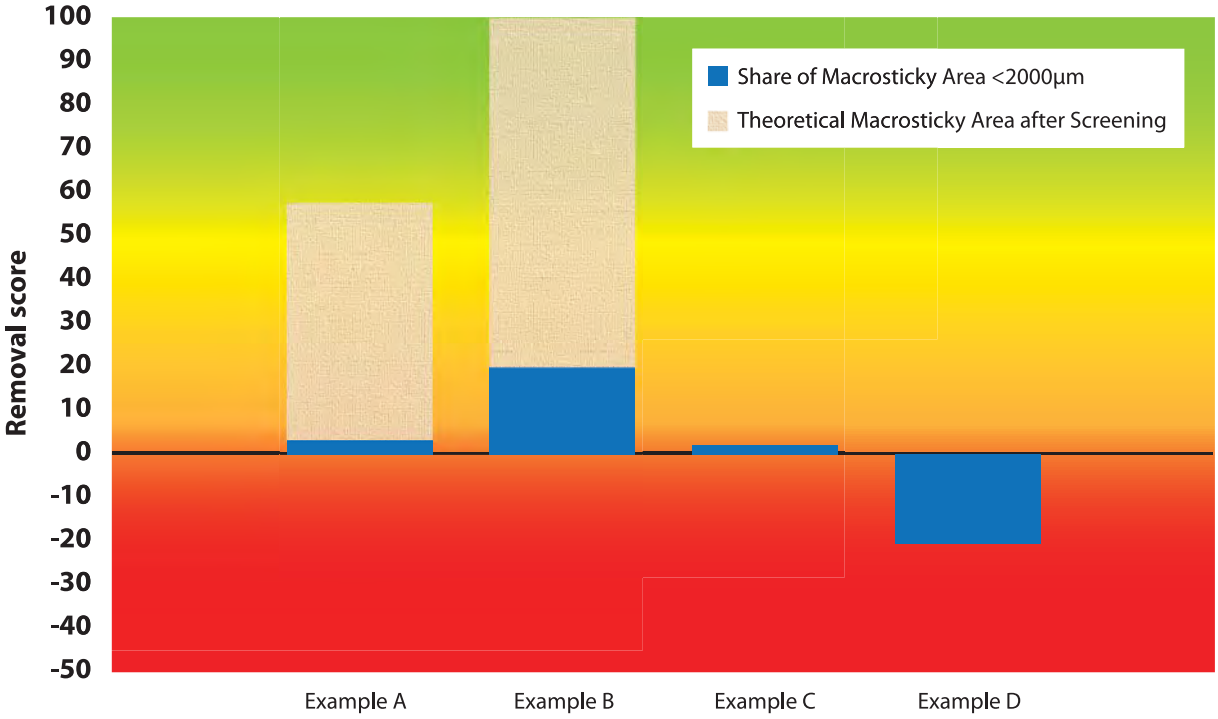

Figure 2: Removal Scores for Examples A to D

Interpretation of the results of Examples A to D:

A. Share S_{2000} close to threshold and area A_t on average level results in an average Removal Score.

B. Low share S_{2000} and low area A_t results in the maximum Removal Score.

C. Share S_{2000} close to threshold and very high area A_t results in a low Removal Score.

D. The very high share S_{2000} leads to the assessment “insufficiently removable” despite the very low area A_t.

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If you want to help us develop paper recycling in Europe, why not include the following email tagline in your own email signature:

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Design by Ben Timmers (CEPI)

INGEDE Method 11

July 2012

Assessment of print product recyclability – Deinkability test

This document was originally developed and launched by INGEDE, its members and its research partners. In the frame of the EcoPaperLoop Project INGEDE Method 11 was translated into several languages. However, in case of any discrepancies the only valid version is the one in English language.

INGEDE Method 11	Assessment of print product recyclability – Deinkability test –	INGEDE International Association of the Deinking Industry
July 2012 13 Pages		

Introduction

A good recyclability of printed products is a crucial feature for the sustainability of the graphic paper loop. It belongs to the focal work of INGEDE to safeguard and improve recyclability.

One of the measures is to provide tools for the assessment of the recyclability in the two aspects:

- Deinkability
- Screenability of adhesive applications.

Therefore a set of methods has been developed to simulate the common operating conditions of relevant process steps in an industrial deinking plant under standard conditions in a laboratory scale. This allows to estimate the relative challenge a printed product means to a deinking plant. Deinking plants producing deinked pulp for newsprint, publication and other printing & writing papers predominantly use paper for recycling with a significant content of mechanical pulp based grades. These papers usually are deinked in an alkaline environment. This is meant by the term “common operating conditions”. Printed paper products recovered by household collection together with newspapers and magazines are also treated under these common operating conditions.

This method has been developed for the assessment of the deinkability of individual printed products.

1 Scope

This INGEDE Method describes a procedure to evaluate the deinkability of printed paper products by means of alkaline flotation deinking. It can be used for any kind of printed paper product.

2 Terms and definitions

Deinked Pulp (DP):

- Pulp consisting of printed products deinked according to this method.

Undeinked Pulp (UP):

- Pulp consisting of printed products disintegrated mechanically with added deinking chemicals, prior to flotation.

3 Principle

Flotation is the most widely used technology for ink removal in the paper recycling process. This INGEDE Method in a laboratory scale defines the essential steps of the flotation deinking process: pulping and flotation. In order to simulate the average age of paper recovered from households, an accelerated aging step is part of the procedure. Special care was taken to define a procedure without the need to test unprinted paper. The whole laboratory procedure is shown in Figure 3.

The deinkability is assessed by three quality parameters of the deinked pulp and two process parameters.

Quality parameters:

- Luminosity
- Colour shade
- Dirt specks (in two different size categories).

Process parameters:

- Ink Elimination
- Filtrate darkening.

4 Equipment and auxiliaries

4.1 Equipment

- Warming cabinet with free or forced ventilation or with air turbulence according to ISO 287.
- Analytical balance up to 1 000 g with an accuracy of at least 0,001 g
- Analytical balance up to 3 000 g with an accuracy of at least 0,1 g
- Hobart pulper N 50, available from Hobart GmbH. Use the type of stirrer and a comparable cover, shown in the following figures. Additionally, it is possible to install a revolution counter, which stops the device automatically.

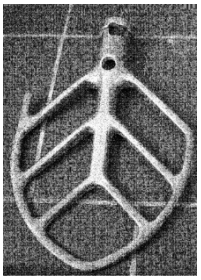

Figure 1: Stirrer for the Hobart pulper

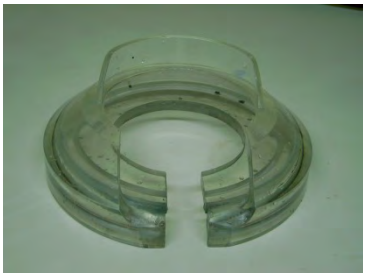

Figure 2: Cover for the Hobart pulper

- Temperature-controlled water bath
- Heating plate equipped with magnetic stirrer, or commercial-grade hot-water heater
- Laboratory flotation cell (references: PTS cell, Voith Delta 25™)
- Plastic scraper (in case of PTS cell)
- Beakers
- Muffle furnace which can be adjusted to an incineration temperature of 525 °C
- pH measuring system with an accuracy of 0,1 points.

If different equipment is used, this has to be mentioned in the test report.

4.2 Chemicals

- Sodium hydroxide (NaOH), pro analysis, CAS # 1310-73-2
- Sodium silicate 1,3–1,4 g/cm³ (38–40 °Bé)
- Hydrogen peroxide (H_2O_2), e.g. 35 %
- Oleic acid ($C_{18}H_{34}O_2$), extra pure, CAS # 112-80-1, e.g. Merck Article No. 1.00471
- Calcium chloride dihydrate ($CaCl_2 \cdot 2\ H_2O$), CAS # 10035-04-8

5 Procedure

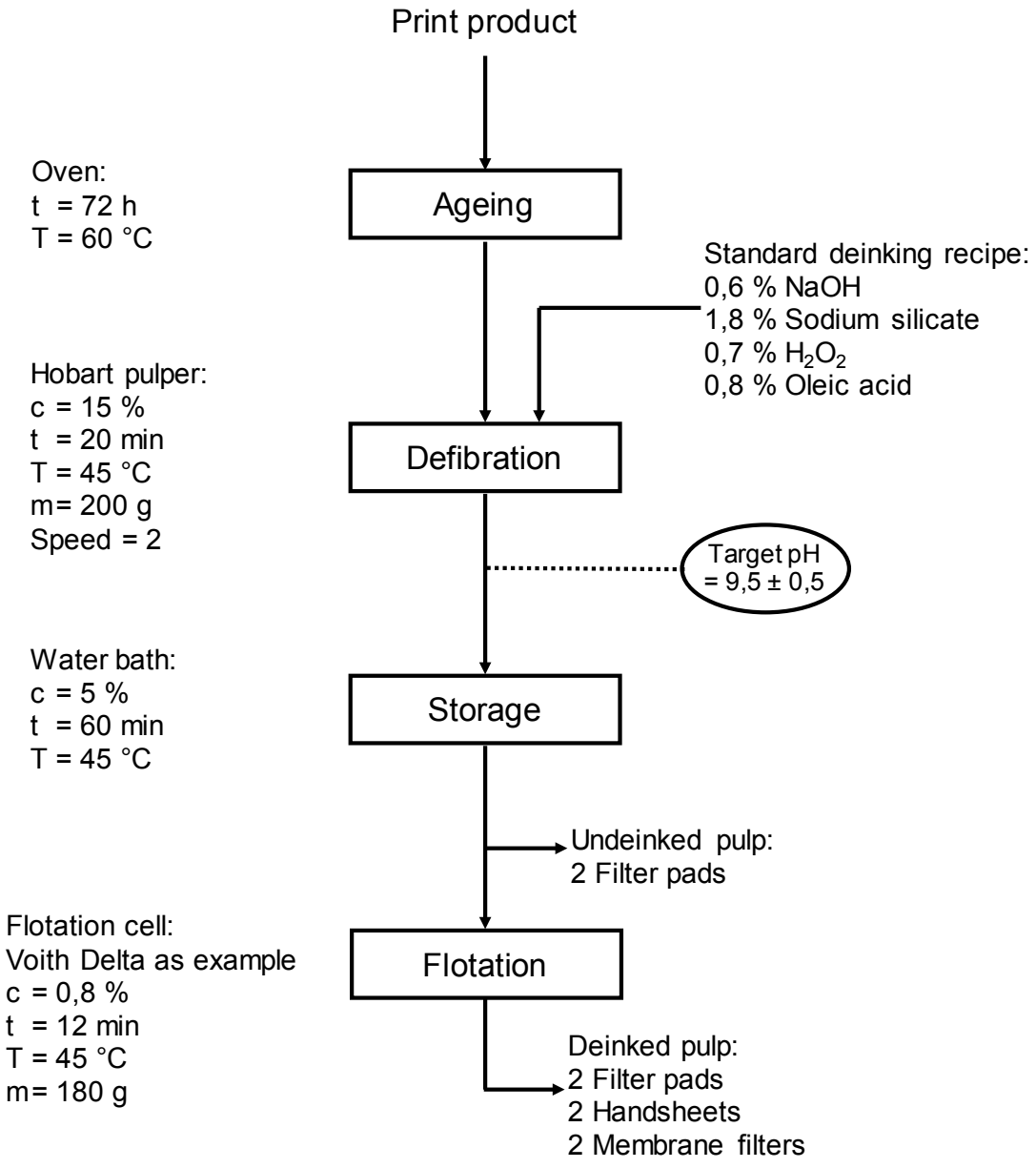

Figure 3: Procedure for testing deinkability with standard deinking recipe

5.1 Sampling

The printed samples used in the tests must not be split up. The minimum amount of each printed sample is 250 g oven-dry.

5.2 Identification

Each print product is designated by its title, publishing house, issue date, product category, printing method and paper grade, if available. Determine the ash content of the paper sample.

Weigh the complete printed product. After weighing, remove any inserts and non-paper components from the printed product to determine their share in the total mass of the product.

5.3 Separation of adhesive applications

To allow the sticky-forming potential of the printed product to be assessed independently, separate all evident adhesive applications from the paper, mark them according to their use, and store them separately.

Glued backs of magazines or catalogues shall be separated according to INGEDE Method 12.

5.4 Accelerated ageing

Place the samples in a warming cabinet for accelerated ageing at 60±3 °C for 72 hours

Accelerated ageing of the samples is necessary because the storage of the papers for recycling can influence their deinkability. These accelerated ageing conditions correspond to 3–6 months of natural ageing.

5.5 Breaking up of samples

Accelerated aged samples are torn into pieces of about 2x2 cm² and acclimatised. A part of the acclimatised samples is used to determine the moisture content according to ISO 287 with at least one sample of about 50 g minimum. Based on the obtained results, calculate the appropriate air-dry weight of the samples which corresponds to the oven-dry weight prescribed.

5.6 Weighing the samples

After homogenising the samples by hand, weigh out samples of 200 g oven-dry.

5.7 Preparation of dilution water

During laboratory treatment of the printed products (5.9 to 5.13), use only water which has been treated to obtain the prescribed hardness values.

To obtain the desired water hardness, add calcium chloride dihydrate ($CaCl_2 \cdot 2\ H_2O$) in deionised water until the concentration of calcium ions reaches 3,21 mmol/l, equivalent to 472 mg/l. This is equivalent to 128 mg Ca^{2+}/l.

If tap water is used, this shall be mentioned in the test report indicating the respective hardness.

During sample preparation, a constant temperature of 45 °C should be maintained. The dilution water should therefore be stored in a water bath whose temperature can be controlled accordingly. It is also possible to heat part of the dilution water to a considerably higher temperature by means of a hot-water heater, and successively add cold dilution water until the desired temperature has been reached. It is not advisable to separately heat the individual stock solutions (dilution water, chemical stock solution, peroxide solution).

5.8 Preparation and dosing of chemicals

The standard formulation is as follows:

Table 1: Standard deinking recipe

Chemical	Dosage (related to oven-dry paper)
Sodium hydroxide	0,6 % (100 %)*
Sodium silicate	1,8 % (1,3–1,4 g/cm^3)*
Hydrogen peroxide	0,7 % (100 %)
Oleic acid	0,8 % (extra pure)

* Only if the pH is either too low or too high after pulping or if it is too low before flotation, the dosages of sodium hydroxide and of sodium silicate have to be adapted (see 5.10).

Make sure that the chemicals are dosed with a relative tolerance not exceeding ±1 %.

It is useful to prepare a total amount of 2 l stock solution which will be sufficient for 5 tests. Dissolve 6 g sodium hydroxide in deionised water, heat slightly to approx. 60 °C and proceed by adding 8 g oleic acid. Stir until the solution is clear, then add 18 g sodium silicate and fill up with deionised water to 2 litres. The formation of soap reduces the alkalinity. 0,114 % sodium hydroxide is needed to neutralise the oleic acid.

In addition, prepare 100 ml hydrogen peroxide solution for each test, using deionised cold water.

5.9 Defibration

Fill the Hobart pulper with the prescribed sample quantity (200 g oven-dry). Take 400 ml of chemicals solution and fill up to a total volume of 1233 ml with appropriately heated dilution water. Add this deinking liquor into the vessel and run the Hobart pulper for some seconds. Then stop it, brush down any scrap of paper from the vessel wall. Repeat this step as often as necessary.

After the first stop, add the peroxide solution (100 ml). The stock consistency is now 15 %. Immediately after, disintegrate the stock for 20 min at approx. 45 °C, using rotor speed 2.

To maintain a constant temperature and avoid splashing losses, cover the vessel during disintegration, for example with a suitably sized, tight-fitting plastic lid (see Figure 2).

5.10 pH value after defibration

At the end of pulping, measure the pH. For a precise measurement of the pH after pulping it is necessary to create a small amount of filtrate by pressing out a pulp sample.

The target pH value is 9,5.

Using the standard formulation from chapter 5.8 the permitted range of pH is 9,5 ± 0,5. If the pH is beyond this range, the sample has to be discarded and the test repeated with an adapted dosage of chemicals. In case of too low pH after pulping the dosage of sodium hydroxide has to be increased. In case of too high pH, both sodium hydroxide and sodium silicate have to be reduced by the same ratio. The minimum dosage of sodium hydroxide is 0,2 %.

Beginning with a non standard chemical formulation, while not proving to be in the range with the standard formulation, the accepted pH is 9,5 ± 0,2.

Figure 4 describes the procedure when starting with standard or non standard chemical formulation.

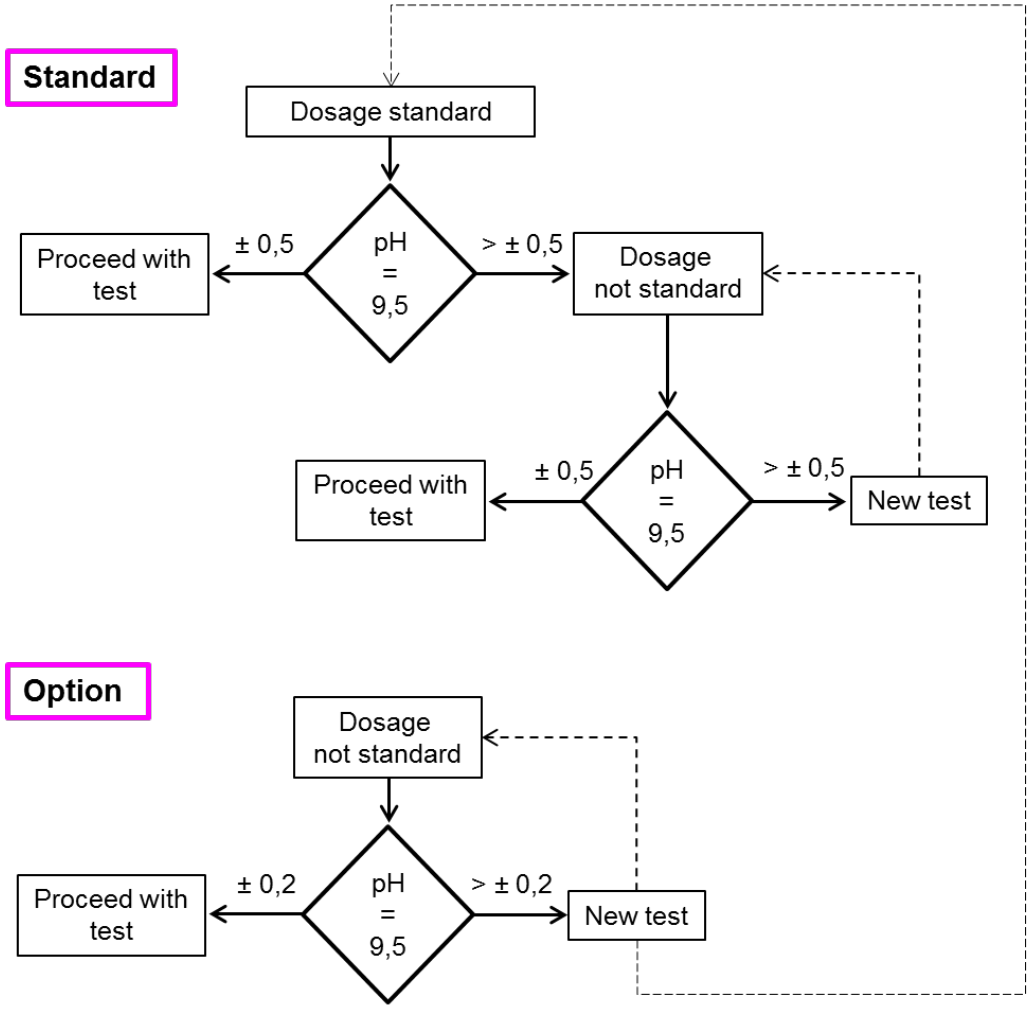

Figure 4: pH value tolerances

ANNEX A describes a method to pre-test the pH after storage with a smaller sample amount. It gives an idea whether a too low or too high pH has to be expected. This principle of pre-testing is applicable also with other chemical dosages, but does not compensate the original defibration with 200 g oven-dry pulp. The requirements of pH tolerances must be fulfilled regardless the pre-test result.

5.11 Storage

The amount of pulp needed for the subsequent treatment steps depends on the quantities required for the final handsheet and filter pad formation (see 5.14). Stock quantities of 12 g oven-dry undeinked (UP) and approx. 15 g oven-dry deinked pulp (DP) are needed in minimum. Stock losses will vary depending on the print products used and can amount up to 50 % during flotation.

Store the amount of stock required for subsequent treatment for 60 min in a water bath at 45 °C and 5 % consistency. The dilution water has been brought to a temperature of 45 °C and to the desired level of hardness.

Measure the pH before and after the storage time. The pH can be measured with reasonable accuracy in the pulp at storage consistency. However, it is recommended to measure the pH before and after the storage in a filtrate without fibres in order to increase the accuracy of the measurement. This filtrate can be generated by pressing a small cullender onto the surface of the pulp. The pH electrode can then be dipped into the filtrate which forms inside the cullender.

5.12 Dilution

After storage the stock samples must be diluted with 45 °C warm water to terminate any chemical reaction before the treatment continues. Use tap water for the UP sample. For the pulp sample to be deinked, use water that has been brought to a temperature of 45 °C and to the desired level of hardness. The consistency after this dilution should be around 1 %; it can be the consistency required for flotation.

Measure the pH. At flotation consistency it should be equal or higher than 7,5, provided that the defined range of the pH after pulping is met. If the pH before flotation is below 7,5, discard the sample and repeat the test with a higher dosage of sodium hydroxide.

Start the flotation before preparing the UP specimens.

5.13 Flotation

Heat up the cell with hot water if the cell has big metal parts. After some minutes pour out the heating water and fill in first some of the prepared dilution water of 45 °C to prevent the “concentrated” pulp from staying in dead corners later. Add the diluted sample into the flotation cell. Fill up with dilution water, and proceed as the instructions of the flotation cell describes. The starting point for the flotation time is when the air supply is started. The process time is set in the following instructions for the recommended flotation cells. For other cells, the process should run until the status of hyper-flotation is reached.

5.13.1 PTS flotation cell

Use the following settings for flotation: air supply rate 60 l/h, stirrer speed in suspension 1200 min^{-1}, flotation period 10 min, suspension temperature approx. 45 °C, consistency approx. 0,8 % at the beginning with 12 g oven dry pulp.

During the entire flotation process, use the scraper to remove the froth without stock, if possible. Collect the skimmed-off flotation rejects in a tank. Continually add dilution water to compensate for the drainage, keeping the suspension level constantly up at the edge of the overflow for the duration of the flotation.

After a flotation period of 10 min switch off the air supply and the stirrer. Use dilution water to bring down any rejects from the overflow into the collecting tank and then dewater the froth. Determine the amount of the overflow oven-dry according to ISO 4119 and use this amount to calculate the flotation yield.

5.13.2 Voith Delta 25TM

The air supply has to be set to approx. 7 l/min. Use the supplier's calibration sheet to find the corresponding point on the scale. The other parameters are: flotation period 12 min, suspension temperature approx. 45 °C, consistency approx. 0,8 % at the beginning with 180 g oven-dry pulp.

During the flotation process add the necessary amount of 45 °C warm water several times in order to maintain the level of the aerated suspension in the cell. In case of low foaming tendency, increase the level in order to guarantee the overflow of foam.

After the flotation period switch off the air supply. Use dilution water to bring down any rejects from the overflow into the collecting tank, and then dewater the froth. Determine the amount of the overflow oven-dry according to ISO 4119, and use this amount to calculate the flotation yield.

5.13.3 Other laboratory flotation cells

Use flotation parameters and conditions similar to the standard conditions applied during the laboratory treatment of deinked recycled pulps.

The flotation should run until the status of hyper-flotation is reached. Set the flotation time in order to get maximum luminosity and ink elimination.

5.14 Specimen preparation

For undeinked pulp two filter pads and for deinked pulp two filter pads and two laboratory handsheets are required to permit an optical evaluation. In addition, two membrane filter specimens are prepared from the filter pad filtrate of the deinked pulp so as to be able to assess filtrate quality. INGEDE Method 1 is used to prepare the specimens.

5.15 Analysis

The following optical characteristics of air conditioned filter pads, laboratory sheets and filtrate filters are determined using INGEDE Method 2.

- Luminosity Y of deinked pulp
- L*, a*, b* colour coefficients of deinked pulp
- Ink elimination IE_{700} and/or IE_{ERIC}
- Filtrate Darkening ΔY of deinked pulp
- Dirt particle area A of deinked pulp

Measure the stock consistency to maintain required conditions, e.g. for storage and flotation. Use the filter pads of stock consistency measurements to determine the ash content of undeinked and deinked pulp in accordance with ISO 1762.

In order to calculate yield values (overall yield and fibre yield) make sure to measure the feed and the overflow of the flotation. Maintain the correct amount of oven dry pulp for the flotation process.

The flotation yield is calculated as follows:

Yield (Overall yield):

$$Yield = \frac{(c_{UP} \cdot m_{UP}) - (c_{froth} \cdot m_{froth})}{(c_{UP} \cdot m_{UP})} \cdot 100\,\%$$

Where:

c_{UP} in g/kg	stock consistency of undeinked pulp
m_{UP} in kg	feed amount flotation, undeinked pulp
c_{froth} in g/kg	stock consistency of overflow
m_{froth} in kg	overflow mass

Fibre Yield:

$$Fibre\,Yield = Yield \cdot \frac{(1 - Ash_{DP})}{(1 - Ash_{UP})}$$

Where:

Ash_{DP}	Ash content of deinked pulp in decimal
Ash_{UP}	Ash content of undeinked pulp in decimal (e.g. 0.03)

6 Report

The following should be recorded in the test report:

- Identification of print product as to name, publishing company, date of issue, product category, print process and paper quality, ash content.
- Mass-related proportion of supplements and non-paper material in %.
- Number and type of adhesive applications.
- pH after pulping, before and after storage and before flotation.
- Chemical dosage for pulping.
- Ash content of undeinked and deinked pulp.
- Flotation yield in %.
- Fibre yield in %.
- Overflow mass m_{froth} .
- Overflow stock consistency c_{froth} .
- Luminosity Y of deinked pulp.
- L*, a* and b* of deinked pulp.
- Ink elimination IE_{700} in %, $R_{\infty,UP}$, $R_{\infty,DP}$ at 700 nm.
- Alternatively to IE_{700}, the ink elimination using ERIC values (IE_{ERIC}) may be determined.
- Filtrate darkening ΔY of the deinked pulp sample filtrate.
- Dirt particle area of deinked pulp in mm²/m² in two categories with the dirt particle area > 50 µm and the dirt particle area > 250 µm.

Deviations from the conditions stipulated for this test method, if applicable (e.g. pulping device, specification of the laboratory flotation cell, conditions of flotation).

Any further optical characteristics of undeinked and deinked pulp yielded as well as their respective filtrate quality may also be noted in the test report.

7 References

7.1 Cited Standards and methods

- INGEDE Method 1 – Test sheet preparation of pulps and filtrates from deinking processes
- INGEDE Method 2 – Measurement of optical characteristics of pulps and filtrates from deinking processes
- INGEDE Method 12 – Assessment of the recyclability of printed paper products – Testing of the fragmentation behaviour of adhesive applications
- ISO 287 – Paper and board - Determination of moisture content of a lot - Oven-drying method

- ISO 1762 – Paper, board and pulps — Determination of residue (ash) on ignition at 525 °C
- ISO 4119 – Pulps – Determination of stock concentration
- ISO 5263-1 – Pulps – Laboratory wet disintegration
- ISO 5269-2 – Pulp – Preparation of laboratory sheets for physical testing. Part 2: Rapid-Köthen method

7.2 Literature and other related documents

- European Recovered Paper Council, Assessment of Print Product Recyclability – Deinkability Score – User's Manual, March 2009, www.paperforrecycling.eu/

7.3 Sources

This method has been published for the first time in 2001. A major revision was done in 2007 according to the definitions made in INGEDE Project 85 02 CTP/PMV/PTS – European Deinkability Method. In 2009 criteria for the pH after pulping and before flotation were added. After gaining some experiences, procedures related to the pH criteria were added to this version.

Annex A:

Testing the pH of a smaller sample amount

In case of not having a sufficient amount of sample paper for repeating the disintegration, test a small amount of your sample beforehand. Use 20 g oven dry sample, pour in 40 ml of the preheated standard chemical formulation and fill up to 123 ml with preheated dilution water. Prepare 10 ml of the peroxide solution. Disintegrate the sample with a dispersing device (e.g. hand blender, laboratory dispersing machine), stop after some seconds and add the prepared peroxide solution. Then disintegrate until the sample is pulped. Store the pulp at 45 °C for 20 minutes and determine the pH.

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INGEDE Method 12

January 2013

Assessment of the Recyclability of Printed Paper Products – Testing of the fragmentation behaviour of adhesive applications

This document was originally developed and launched by INGEDE, its members and its research partners. In the frame of the EcoPaperLoop Project INGEDE Method 12 was translated into several languages. However, in case of any discrepancies the only valid version is the one in English language.

INGEDE Method 12	Assessment of the Recyclability of Printed Paper Products	INGEDE International Association of the Deinking Industry
January 2013 11 Pages	**Testing of the fragmentation behaviour of adhesive applications**	

Introduction

A good recyclability of printed products is a crucial feature for the sustainability of the graphic paper loop. It belongs to the focus of INGEDE activities to safeguard and improve recyclability.

One of the measures is to provide tools for the assessment of the recyclability in the two aspects:

- Deinkability
- Screenability of adhesive applications

Therefore a set of methods was developed which simulate unit operations of a deinking plant and allow conclusions about the behaviour of a printed product and the adhesive applications in a deinking plant.

This procedure deals with the fragmentation behaviour of adhesive applications after pulping as one aspect of recyclability assessment. The method is based on the general requirement that it should be possible to separate adhesive applications mechanically. The fragmentation behaviour determines the screenability (see ERPC Scorecard “Assessment of Print Product Recyclability – Scorecard for the Removability of Adhesive Applications”).

1 Scope

This INGEDE method describes a procedure for testing the fragmentation behaviour and screenability of adhesive applications on paper products. It is suitable for known and for unknown amounts of adhesives in the recycled paper sample.

2 Terms and definitions

Macrostickies:

ZELLCHEMING Technical Leaflet RECO 1, 1/2006 “Terminology of Stickies”, determined by means of INGEDE Method 4.

Stickies is the term for adhesive (tacky) particles that occur when recycled fibres are utilised. Macrostickies is commonly the term for the tacky residues on the screening plate after a fractionation.

Adhesive Applications:

Adhesive spine

Are the adhesive back binding of printed books, magazines, journals and catalogues.

Side glue

One or two pages at the front and one or two pages at the back side of a printed product are part of the binding. The adhesive spine and the side glue form together the adhesive binding.

Glued-in inserts

These are adhesive applications to glue samples or leaflets mostly for commercial purposes into or at printed products.

PSA

Is the abbreviation for pressure sensitive adhesives, typically used for labels and stickers.

3 Principle

This method is determined to simulate the screening ability of adhesive applications in a deinking process. The two essential process steps are pulping and screening.

This method describes the laboratory pulping process by defining the physical conditions and the addition of deinking chemicals (Figure 1).

The separation of adhesive applications from the pulp is done by screening according to INGEDE Method 4.

The particle size distribution of the macrostickies is measured, thus allowing the assessment of the screening ability of the adhesives application in an industrial process.

The setting of the screening ability limit of < 2 000 µm equivalent circle diameter was proven in semi-industrial pilot plant trials and confirmed by test results from industrial processes.

One driving force to develop this assessment method is the fact that normally the amount of adhesives in a printed product is unknown. If it is known, the test can be combined with INGEDE Method 13.

4 Equipment and auxiliaries

4.1 Equipment

- Analytical balance up to 1 000 g with an accuracy of ± 0,001 g
- Analytical balance up to 3 000 g with an accuracy of ± 0,1 g
- Hobart pulper model N 50, supplied by HOBART GmbH, equipped with a blade type stirrer (see INGEDE Method 11)
- Haindl classifier in accordance with ZM V/1.4/86 or Somerville tester according to TAPPI T 275 sp-07 or Pulmac Master Screen-type instrument according to TAPPI T 274 sp-08
- Slotted plate with a slot width of 100 µm
- Rapid-Köthen sheet former in accordance with ISO 5269/2 respectively
- Drying cabinet in accordance with ISO 287

- Scanner-based image analysis system with a minimum resolution of 600 × 600 dpi, e. g. DOMAS, SIMPALAB

4.2 Test material

- Woodfree, virgin fibre based copy paper with an ash content of 20 ± 3 % ash determined at 525 °C
- Test material for sticky visualisation according to INGEDE Method 4

4.3 Chemicals

The required standard deinking chemicals are listed in INGEDE Method 11:

- Sodium hydroxide p. A.
- Sodium silicate, density 1,3–1,4 g/cm³
- Hydrogen peroxide, e. g. 35 %
- Oleic acid, extra pure

5 Procedure

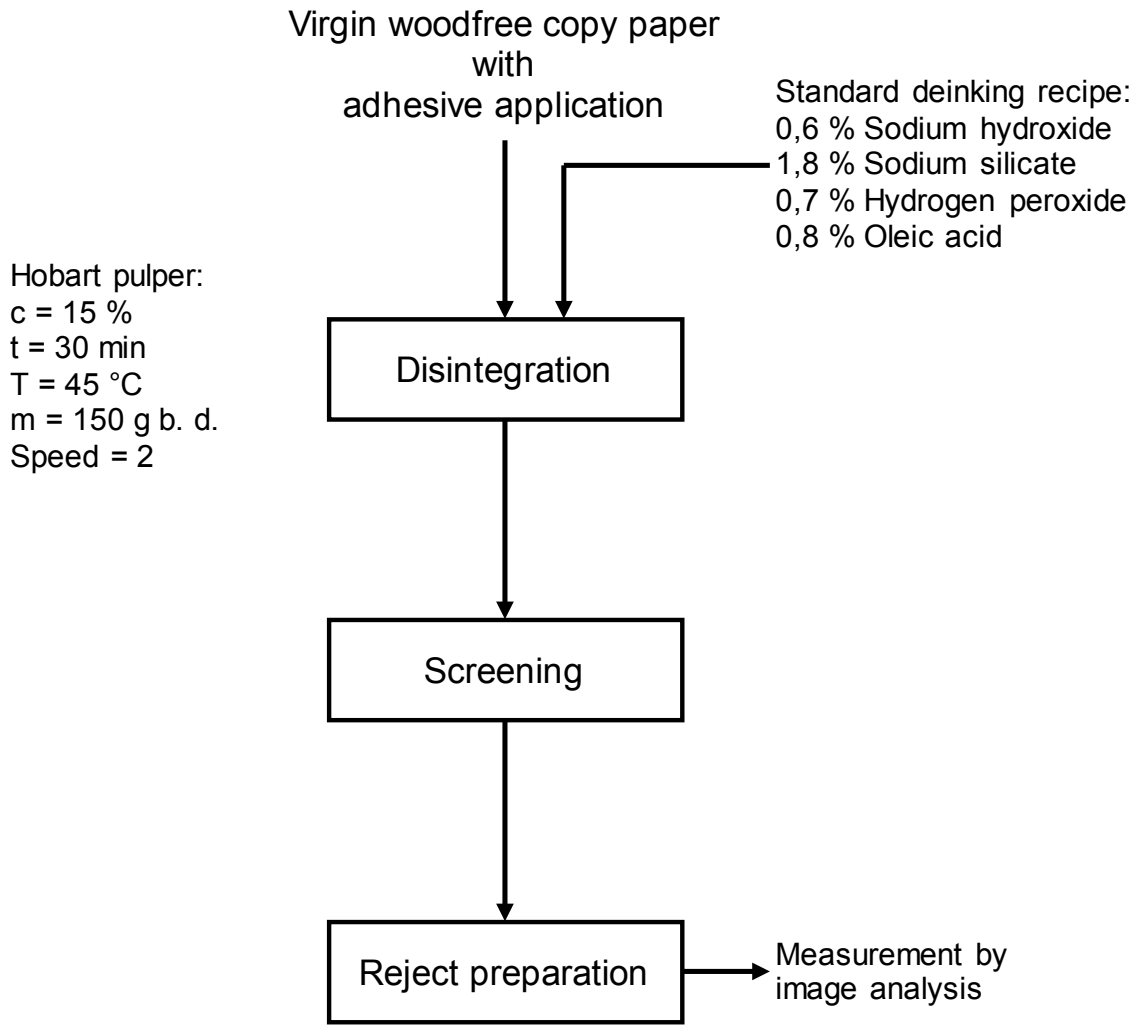

Figure 1: Testing fragmentation behaviour of adhesive applications

5.1 Preparation of adhesive applications

It is recommended to store the samples under climate conditions according to ISO 187 for 24 hours. Use the recommended amount of adhesive applications as it is described below otherwise state the utilized amount or area in the report. Vary the amount of adhesive applications only for the case not reaching representative results or reduce the amount of adhesive applications if stickies heavily overlap on the reject filter.

Adhesive spine

The evaluation of a printed product includes the testing of all adhesive applications. The different adhesive applications of one printed product are tested separately and the results are added weight-proportional (mm^2/ kg). Bookbinding backs which may consist of different adhesive types are tested as one compound as far as no further information is required. The spine and side binding is normally tested together in one test. The both pages at the front and at the end of the printed product should not be separated from the adhesive back unless they contain adhesive applications which should be evaluated independently.

Adhesive magazine and catalogue backs should be separated by means of a saw, leaving approximately 4 cm of the page width attached to the adhesive back. The following use of individual pieces is recommended for the test.

Table 1: Recommendation for the use of magazine/ catalogue back

Width of magazine or catalogue back	Length of each piece	Number of pieces
< 4,5 mm	2,5 cm	5
4,5–6,9 mm	2,5 cm	4
7,0–9,9 mm	2,5 cm	3
10,0–19,9 mm	2,5 cm	2
20,0–30,0 mm	1,0 cm	4
> 30,0 mm	1,0 cm	3

Side glue

If the side glue is of special interest, it is tested separately. The glued sides of the spine of a printed product are prepared similar to the adhesive backs: After separating the first two pages at the front and at the back from the spine, cut a stripe from these pages of 4 cm width including the glued area. Then cut the stripe into pieces of the recommended length in the table.

Glued-in inserts

Glued-in inserts have to be tested separately from spine and side glue.

Inserts made of fibrous material (paper) should not be separated from the printed product page to avoid adhesive losses. The adhesive stays covered with paper on the bottom and on the top side. Cut out the adhesive leaving a frame of paper of approximately 2 cm around the adhesive. Then cut pieces with maximal 2 cm length (which might possibly mean to cut the adhesive).

It might be necessary to use several samples of glued-in inserts to generate a sufficient amount of stickies, e.g. 5 applications. Record the amount for subsequent calculation and report.

Inserts made of plastic materials are detached from the sample without removing the adhesive or with transferring the adhesive carefully back to the printed product page. Cover the adhesive with a clean part of the printed product page and then cut out the adhesive while leaving a frame of paper of 2 cm around it. Then cut pieces with maximal 2 cm length (which might possibly mean to cut the adhesive).

PSA application in printed products

Finished paper label products, e.g. in special editions, journals or magazines may contain huge flat PSA applications. Use 100 cm^2 of the PSA application and stick them on woodfree copy paper. Then cut them into 1–2 cm^2 pieces. If one printed product contains less than 100 cm^2 PSA application use those of several issues of the printed product. Record the number of products for subsequent calculation and report. After performing the test, express the test result in mm^2/kg printed product.

PSA applications – not a final product

Not finally applied PSAs, stickers or labels, are stuck on woodfree copy paper and pressed one time with a press roll (2 kg). It is recommended to use 100 cm^2. This area has to be cut after attaching to the woodfree copy paper into 1–2 cm^2 small pieces before pulping. Record the grammage of the PSA in g/m^2.

If possible, the area and mass of all tested individual adhesive applications should be recorded. That will allow calculating the test result in relation to these figures.

5.2 Sample preparation

For pulping purposes, virgin fibre based copy paper (20 ± 3 % ash) is used. The total mass of copy paper and adhesive application to be tested is 150 g oven-dry. The paper should be provided in 1–2 cm^2 sized pieces.

5.3 Disintegration

On order to simulate an industrial sticky fragmentation, it is necessary to use a Hobart pulper under the following conditions. The total mass of copy paper and adhesive application tested is 150 g oven-dry. The total suspension volume in the vessel is 1000 ml.

In the beginning, the vessel of the Hobart pulper is filled with hot water of about 50 °C. After removing the water from the vessel, the copy paper is added as well as 300 ml of the basic chemical solution prepared in accordance with INGEDE Method 11 and tempered dilution water to a total of 925 ml. The dilution water should be heated to such a level that, after the addition of all pulp components, the temperature in the pulper is 45 °C. Directly after the start of the pulping process, set in motion by switching on the rotor at speed 2, the peroxide solution (75 ml), also prepared in accordance with INGEDE Method 11, is added. The prepared adhesive application to be tested is then stirred in immediately afterwards.

Especially during the first five minutes of the disintegration process, any solid particles that attach to the wall of the vessel should be pushed back in to ensure a complete treatment of all solid material. The pulper can be stopped briefly for this purpose.

The pulping time is 30 minutes in total. In order to keep the temperature constant during pulping and to prevent stock losses, the pulper should be fitted with a tightly closing lid.

5.4 Screening

In order to ensure that all generated sticky fragments are taken into account in the evaluation, the entire prepared stock (150 g oven-dry) is screened in portions. For this purpose, the pulped stock is filled up with water to a total volume of 3 000 ml, and the dilution water is used at the same time for rinsing out the pulper vessel. After its homogenisation, the pulp suspension is divided into three equal parts of 50 g oven-dry each. Depending on the contaminant concentration the operator can also decide to screen in portions of 25 g oven dry pulp. For this, divide the pulp suspensions into six equal parts of 25 g oven-dry (500 ml) each and dilute them to 1 liter.

The screening procedure follows INGEDE Method 4.

5.5 Specimen preparation

After each individual screening, the residue is treated according to INGEDE Method 4. It is recommended to prepare one filter sample from each individual screening. Pay attention that no overlapping of stickies occurs on the filters. In the case of presence of larger sticky fragments which can occur especially when testing adhesive backs, check visually if smaller stickies are overlaid before drying the dewatered residue. Try to separate them carefully on the filter or transfer the larger sticky fragments on an additional filter. Big, cubic sticky particles must be transferred on an additional filter (in a later step smaller and flat particles are better covered by the alumina powder).

Besides dewatering, the preparation of the residue includes the part steps of drying and sticky visualisation. The visual check of the contrasted filter preparations is omitted, as there are no other hydrophobic particles than those to be tested contained in the residue. All hydrophobic particles that occur as a result of the adhesive application are taken into account in the following measurement by image analysis.

5.6 Measurement by image analysis

The treated filter preparations are then evaluated with the aid of a scanner-based image analysis system at a resolution of 600 dpi. The area to be measured should be selected in such a way as to ensure that all macro stickies are recorded.

Ensure that one class limit is fixed at the identical equivalent circle diameter of 2 000 µm when defining the class limits. The lowest measuring limit, in view of the method concerned, is 100 µm. When defining the upper limit, it must be ensured that no large sticky fragments are excluded.

DOMAS or SIMPALAB systems can be used. The following defined class limits have to be set:

100 µm, 200 µm, 400 µm, 600 µm, 1 000 µm, 2 000 µm, 3 000 µm, 5 000 µm, 10 000 µm and larger than 10 000 µm.

5.7 Evaluation

Add up the results of single measurements which were obtained due to the screening of individual portions. The final test results are expressed in mm^2/kg printed product air dry, for this, calculate as follows.

Adhesive spine

After the image analysis, the results for adhesive backs are obtained in mm^2/analysed filter area. Calculate the sticky area for the overall back length and divide it by the mass of the printed product (catalogue, magazine...) in kg. The result is mm^2 stickies per kg printed product.

Side glue

Proceed similar to the adhesive spines.

Glued-in inserts

The result of the glued-in insert is divided by the number of used inserts for one test. Divide then by the mass of printed product mass. The result is mm^2 stickies per kg printed product.

PSA application in printed products

The results of PSA application are obtained in mm^2 stickies per 100 cm^2 which have been tested. Based on that, calculate the sticky area for the effective PSA area present in the printed product. Divide then by the mass of printed product and get the result in mm^2 stickies per kg printed product.

PSA applications – not a final product

Calculate the theoretical mass of the tested 100 cm^2 ($m_{100\,cm^2}$):

$$m_{100cm^2} = w_{PSA} \cdot 0{,}01 m^2$$

w_{PSA} grammage of the label, g/m^2

- "Scorecard for the Removability of Adhesive Applications" (ERPC):

Calculate the amount of macrostickies per kg label product. This value is expressed in mm^2/kg label and is transferred to the Scorecard.

- Macrostickies per kg printed product:

It is assumed that the share of the complete label (paper plus adhesive) is about 2,5 % of the complete printed product. Based on this calculate the factor

$$Factor = \frac{25g}{m_{100cm^2}}$$

Multiply the sticky area in mm^2/100 cm^2 with this factor. The result is the macrosticky area per kg printed product under the approval.

The following characteristic quantities from the accumulative result of the measurement of the three individual preparations are used for evaluation purposes:

A_{total} in mm^2 /kg printed product: Total area of macrostickies

A_{MS} in mm²/kg printed product: Total area of macrostickies < 2000 µm identical equivalent circle diameter

S_{2000} in %: Share of macrosticky area below a particle size of 2000 µm identical equivalent circle diameter

A_{600} in mm^2/ kg printed product: The macrosticky content in the size classes below 600 µm identical equivalent circle diameter

A_{1000} in mm^2/kg printed product: The macrosticky content in the size classes between 600 µm and 1 000 µm identical equivalent circle diameter

A_{2000} in mm^2/kg printed product: The macrosticky content in the size classes between 1000 µm and 2 000 µm identical equivalent circle diameter

Presupposing knowledge of the adhesive mass or its application area respectively, it is possible to put the measured area of macrostickies in relation to these figures as mm²/g adhesive or mm²/cm² application respectively.

6 Report

The following should be recorded in the test report:

- Number and type of adhesive applications, amount used in the test if different
- A_{total} in mm^2/kg per single tested adhesive application and overall result for the printed product
- A_{MS} and S_{2000} per single tested adhesive application and overall result for the printed product
- Deviations from the conditions of this test method

7 References

7.1 Cited Standards and methods

Reference was made to the following standards in this method:

- ZELLCHEMING Technical leaflet RECO 1, 1/2006 “Terminology of Stickies”
- ZM V/1.4/86: Gleichzeitige Bestimmung des Gehaltes an Splittern und Faserfraktionen . http://www.zellcheming.de/download/merkblaetter/merkblatt_5_1_4_86.zip
- ISO 1762 – Paper, board and pulps – Determination of residue (ash) on ignition at 525 °C
- TAPPI T 275 sp-07: Screening of Pulp (Somerville-Type Equipment)
- TAPPI T 274 sp-08: Laboratory screening of pulp (Master Screen-type instrument)
- INGEDE Method 4: Analysis of macrostickies in deinked pulp
- INGEDE Method 11: Assessment of Print Product Recyclability - Deinkability Test
- ERPC: Assessment of Printed Product Recyclability – Scorecard for the Removability of Adhesive Applications www.paperforrecycling.eu
- ISO 5269/2: Pulp – Preparation of laboratory sheets for physical testing – Part 2: Rapid-Köthen method
- ISO 287 (2009): Paper and Board – Determination of moisture content – Oven drying method

7.2 Sources

This INGEDE Method was developed and tested within the scope of INGEDE project 66 99 PMV “Evaluation of recyclability of print products with particular consideration of adhesive pulp components” in 2001. In the course of the INGEDE project 129 09 “Preparation of an adhesive application database and development of a recyclability scoring system” the INGEDE Method 12 was revised in 2010.

Assessment of the Recyclability of Printed Paper Products

Testing of the fragmentation behaviour of adhesive applications

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INGEDE Method 1

December 2014

Test sheet preparation of pulps and filtrates from deinking processes

This document was originally developed and launched by INGEDE, its members and its research partners. In the frame of the EcoPaperLoop Project INGEDE Method 1 was translated into several languages. However, in case of any discrepancies the only valid version is the one in English language.

INGEDE Method 1 **September 2014** 6 Pages	**Test sheet preparation of pulps and filtrates from deinking processes**	INGEDE International Association of the Deinking Industry

Introduction

Pulp made of paper for recycling contains typically printing inks influencing its optical properties. Cleaning and flotation remove small sized impurities and printing inks whereby the removal efficiency depends also on applied printing process. The determination of residual ink content uses reflectance measurements of light in the near infrared region. The reflectivity of light gives indication for the content of fine and filler materials which influence the light scattering coefficient and for ink content that alters the light absorption coefficient. The calculation for scattering coefficients requires paper specimens with an opacity less than 95 % (ISO 9416) that is mostly fulfilled with machine papers. Pulp samples taken along the deinking process or pulp samples from deinkability tests (INGEDE Method 11) have to be treated. This INGEDE Method describes the preparation of filter pads where fine and ink losses during preparation are negligible. Filter pads are opaque that hinders the calculation of light scattering coefficient s. Assuming a constant light scattering coefficient is not a recommended approach due to the fact that light scattering varies from pulp to pulp for example when the ash content changes. INGEDE Method 1 describes therefore the preparation of handsheets with the use of recirculated water. The method can be used for industrial as well as for laboratory pulp samples.

1 Scope

This INGEDE method is used to prepare test sheets and filter pads from pulps of the deinking process and laboratory samples.

2 Principle

For testing purpose filter pads are prepared from industrial or laboratory pulp samples using a Büchner funnel and defined filter paper. Handsheets are prepared with the Rapid-Köthen method from industrial pulps under defined conditions. The filtrate samples are drained over a membrane filter and compared with a reference membrane filter made with tap water.

Optical measurements are conducted according to INGEDE Method 2.

3 Equipment and auxiliaries

3.1 Equipment

- Distribution device (volume: 10 l)
- Büchner funnel with appropriate vacuum device that allows a pressure difference ≥ 60 kPa
- Filter paper: Munktell type 1289
- Analytical balance up to 3000 g having an accuracy of at least ± 0,1 g
- Standard sheet former (model: Rapid-Köthen) with dryer (vacuum 95 kPa, 94 ºC), according to ISO 5269-2

- Paper cover sheets and carrier boards according to ISO 5269-2

Filtrate darkening:

- Cellulose nitrate membrane filter: Sartorius type 11306-050N, ø 50 mm, pores ø 0,45 µm
- Vacuum filtration unit with 39 mm bottom inside diameter of the funnel
- Water jet pump or vacuum pump
- Desiccator

3.2 Chemicals

- Cationic Polyacrylamide (CPAM) – high molecular weight, low cationic charge – a polymer for example used for sludge dewatering. Use the CPAM as solution of 1 g/l concentration (powder diluted in tap water).
- Alum

4 Samples

4.1 Pulp samples

A sample should be analysed in the laboratory after sampling a representative quantity of material at the relevant recovered paper processing stage or taking a sample from laboratory deinking test. The consistency of the material should be measured according to ISO 4119.

After the consistency of the material has been measured, the sample is diluted and homogenised to a consistency of 8 g/l in a distribution device. After the consistency has been measured again, a sample can be taken for preparing the test sheet. No pH adjustment is required.

Pulp suspensions up to a consistency of 10 % can be used immediately for sheet preparation without further preparation. However, deinked pulp with higher consistency must be disintegrated before sheet forming. Disintegration takes place in accordance with ISO 5263-2, whereby the disintegration process is restricted to five minutes. At a consistency of 2 % periods of mechanical stress should be held short in order to avoid changes in size distribution of unwanted particles, e. g. ink and stickies.

4.2 Filtrate samples

The preparation of filter pads for measuring the optical properties generates filtrates which are used to prepare the membrane filter samples afterwards. The preparation of the two filter pads produces two filtrate samples from each pulp sample.

5 Procedure

5.1 Filter pads

At least two filter pads are prepared of the pulp samples respectively.

The filter pad is formed using a Büchner funnel which has been covered by a moistened filter paper. The prepared filter pads have a basis weight of 225 g/m². A filter paper diameter of 150 mm and a maximum Büchner funnel diameter of 160 mm are recommendable. For this case 4,0 g oven-dry pulp material is used and the suspension is topped up with tap water to a volume of 1 litre.

Other filter diameters may be used referring to table 1. The diameter of the Büchner funnel corresponds to the filter diameter and should not exceed the maximum value in table 1. Usually, Büchner funnels are purchased by their nominal diameter that is identical with the filter diameter.

If a differing size of Büchner funnel and filter paper are used the sample volume has to be adapted according to table 1. The consistency of the pulp sample remains 0,4 %.

Table 1: Pulp volume for Büchner funnel filtration

Diameter max Büchner funnel in mm	Diameter filter paper (Munktell 1289) in mm	Oven dry material in g	Sample volume at 0,4 % consistency in ml
120	110	2,15	538
135	125	2,75	688
160	150	4,00	1000
195	185	6,10	1525

After filtering and carefully removing the filter paper, the wet filter pad is laid between two new sheets of filter paper before drying. The drying time in the Rapid-Köthen dryer is 10 minutes. The dried filter paper should not be removed from the filter pad until immediately prior to measuring the optical properties.

Experiences have shown that the support of a thin wire made of nylon helps to avoid marks. For this purpose use a nylon wire with a mesh width of about 140 µm and a mesh diagonal of about 190 µm and place it under the filter paper. This option is allowed when preparing the filter pads, but not if the filtrate is analysed for filtrate darkening measurements. For optical assessment of filtrate quality according to chapter 5.5 collect the filtrate obtained from filter pads prepared with one filter paper.

5.2 Laboratory handsheet formation – General procedure

An appropriate volume of material should be taken from the distribution device for each handsheet. After standard laboratory handsheet formation, dry the sheet in the Rapid-Köthen dryer between carrier board and a cover sheet. The drying time should be 7 minutes. The carrier board and the cover sheet should not be removed from the handsheet until immediately prior to measuring the optical characteristics.

5.3 Handsheets for determination of the dirt particle area A

At least two handsheets for the determination of the dirt particle area (A) are prepared with fresh water in order to reach better contrast for the optical analyses. Grammage m_A should amount to 42,6 g/m^2 ± 1,6 g/m^2, related to oven-dry substance.

5.4 Handsheets for the determination of Kubelka Munk parameters

Handsheets for the determination of Kubelka Munk parameters specific light absorption coefficient (k) and specific light scattering coefficient (s) are prepared with recirculated water. Their opacity should not exceed 95 % in the near infrared area.

A homogeneous suspension quantity corresponding to 1,35 g of oven-dry substance is being taken from the distribution container to prepare a laboratory sheet in compliance with ISO 5269-2. After dewatering it is removed from the wire section and either disposed of or used as laboratory sheet for piling to determine reflectance factor R_∞. The filtrate obtained in the process (white water) is being retained and used to dilute the next sheet. To increase the concentration of the white water, this procedure is repeated for four times without changing oven-dry substance. The 5th sheet is removed from the wire section and dried between carrier board and cover sheet in the Rapid-Köthen drier for a minimum of seven minutes. Determine the grammage of the sheet.

The suspension quantity required for sheet formation is modified for the first time so as to obtain a laboratory sheet of a grammage m_A of 42,6 g/m^2 ± 1,6 g/m^2, related to oven-dry substance.

Note: The above grammage corresponds to a laboratory sheet weight of 1,35 ± 0,05 g after RK-drying.

The adapted suspension quantity is then used to prepare two more laboratory sheets (sheets 6 and 7) with the concentrated filtrate, which are also dried between carrier board and cover sheet in the Rapid-Köthen drier for a minimum of seven minutes. To facilitate the following optical measurement, it is recommended to mark top side and wire side.

Prior to optical assessment, the two laboratory sheets have to be conditioned in compliance with ISO 187. The sample grammage after conditioning in a standard reference atmosphere ought to be 45 g/m^2. The value is rounded to 0,1 g/m^2.

5.5 Filtrate samples

The complete filtrate obtained by dewatering the pulp for one filter pad is homogenised. 100 ml filtrate is completely drained using a cellulose nitrate membrane filter in a vacuum filtration unit. Any fibrous material found on the membrane filter may indicate that some pulp bypassed the filter paper when preparing the filter pad. In such a case the membrane filter and filtrate have to be discarded. Prepare a new filter pad and filtrate as described in chapter 5.1.

The filtrate of two filter pads (chapter 5.1) is filtered respectively. Generally, the filtration is done without any retention aids. The result of this filtration must be a discoloured, clear liquid.

Exception:

In case of still having a colourised filtrate after membrane filtration, repeat the procedure with a new sample (100 ml). Add retention aid solution (start with 5 ml) before membrane filtration,

possibly alum or cationic polyacrylamide (CPAM) with high molecular weight and low cationic charge. State in the report whether the membrane filtrate was coloured and a retention aid was used, if yes, how much.

The membrane filters are removed from the filtration unit and dried in a desiccator.

Reference membrane filters are made in the same way, but using exclusively 100 ml of tap water without pulp. Prepare a membrane filter for each test series or on a daily basis at least.

6 Report

- The type of handsheets prepared
- Büchner funnel diameter
- Photography of handsheets and filter pads, membrane filtrate and membrane filter
- Filtrate sample preparation with or without retention aid, dosage
- All deviations from the method

7 References

7.1 Cited Standards and methods

- INGEDE Method 2: Measurement of optical characteristics of pulps and filtrates from deinking processes.
- ISO 187: Paper, board and pulps – Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples (1990).
- ISO 4119: Pulps – Determination of stock concentration (1995).
- ISO 5263-2: Pulps – Laboratory wet disintegration - Part 2: Disintegration of mechanical pulps at 20 °C (2004)
- ISO 5269/2: Pulp – Preparation of laboratory sheets for physical testing, Part 2: Rapid-Köthen method.
- ISO 9416: Paper – Determination of light scattering and absorption coefficients (using Kubelka-Munk theory) (2009)

7.2 Sources

This method has been published for the first time in 1997. A major revision was done according to the definitions made in INGEDE Project 85 02 CTP/PMV/PTS – European Deinkability Test Method. In 2006, also parts of the INGEDE Methods 3 and 10 were transferred to this method. In 2014 a filter paper was defined based on the results of INGEDE Project 140 13.

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INGEDE Method 2

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Measurement of optical characteristics of pulps and filtrates from deinking processes

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INGEDE Method 2	Measurement of optical characteristics of pulps and filtrates from deinking processes	INGEDE International Association of the Deinking Industry
24 November 2014 11 Pages		

Introduction

Optical properties are key parameters for the quality of deinked pulp as well as for determining the efficiency of deinking operations. Parameters and details of the measurement procedures defined and described in this method are reflectance factors, light absorption and scattering coefficients, ERIC, colour values and dirt specks.

The method contains the determination of the Ink Elimination IE based either on the light absorption coefficients or on ERIC of undeinked and deinked pulps.

Particularly when dealing with extremely fine dispersed printing ink particles (e. g. water based printing ink) in the deinked pulp, the filtrate analysis method allows assessing possible pollution levels which may occur in the water systems of deinking plants.

1 Scope

This INGEDE method describes procedures for measuring and calculating various optical characteristics of pulps and filtrates from deinking processes by means of filter pads and handsheets. The method is applicable for industrial as well as for laboratory samples.

2 Terms and definitions

IE: Ink Elimination, calculated as the ratio of the difference of the absorption coefficient *k* of the undeinked and deinked samples to the difference of the absorption coefficient *k* of undeinked and unprinted samples.

ERIC: Effective Residual Ink Concentration, calculated as the ratio of the absorption coefficient *k* of a pulp or paper sample divided by the absorption value of black printing ink and multiplied by 10^6. For black printing ink, a constant *k* value of 10 000 m²/kg may be used. For further details please refer to TAPPI T 567 om-09 or ISO 22754.

3 Principle

Industrial or laboratory samples of pulp and filtrates in deinking processes are transformed to filter pads and handsheets by means of INGEDE Method 1. This INGEDE Method 2 describes and defines the parameters and the settings of the measurement devices to obtain results for optical characterisation of the samples. The calculation of the Ink Elimination is also part of this method and allows an assessment of the deinking process.

4 Equipment and auxiliaries

4.1 Equipment calibration for reflectance measurements

Any measuring equipment set-up which meets the ISO 2470-1 and ISO 5631-2 (colour) requirements may be used for measuring.

- **Zero point initialisation**

A black standard which meets the requirements which are specified in ISO 2469 is used to check the zero point.

- **Upper limit initialisation**

A white standard which meets the criteria described in ISO 2469 is used to set the upper limit.

4.2 Dirt particle measurement (A)

For the determination of dirt particle area A, a scanner based image analysis system is needed for optical analysis. The scanner is to be calibrated to ensure reproducibility of the measurements.

Technical requirements of flatbed scanner:

- Scanning area ≥ ISO A4
- Optical scan resolution ≥ 2000 dpi,
- Colour depth 48 bit,
- Optical density $D_{MAX} \geq 4{,}0$

Requirements on measuring accuracy of flatbed scanner after warm-up period (see scanner manual) and under scanning conditions (see chapter 5.3)

- Reproducibility of mean grey value (8 Bit) ±1 (A ISO A4 sample has to be scanned 10 times without any movement of the sample. All mean grey value of total sample area should be within 2 grey values.)
- Deviation of colour value (RGB-8 Bit) ≤ 5 (After calibration a scanned image of IT8-Target shouldn't have more deviation to associated reference file than ± 5 values in every colour channel - R,G,B.)

Suitable scanners: DOMAS Scanner*Advanced*, Techpap proposed scanner

NOTE:

"Scanner*Advanced*" is a name given by PTS to a commercial scanner that was accredited by PTS. This scanner device is delivered with the DOMAS 3.0 version.

The image analysis software is to be parameterised according to the specifications as described in chapter 5.9.

Suitable software packages are: DOMAS 3.0 and above image analysis software, SIMPALAB Image Analysis Software.

5 Analysis

5.1 General

5.1.1 Sample preparation

The sample preparation is described in INGEDE Method 1. According to Table 1 use either a filter pad or a handsheet to determine the optical parameter and state in the report, which specimen was measured. The samples have to be air-conditioned in accordance with ISO 187.

5.1.2 Sample illumination for reflectance measurements – edge filter

For deinkability testing according to INGEDE Method 11, the samples are illuminated with C/2° conditions while using the edge filter of 420 nm (UV filter)[1]. This applies to all reflectance measurements. Other tests are conducted according to the stated standards.

5.1.3 Measuring points and number of measurements for reflectance measurements

Both sides of the test sheets should be measured (filter pads and laboratory handsheets). Best care and attention is given to avoid measurements too near to edges, kinks or on visible non-uniformities of the test sheets.

Respectively two samples should be measured with four measurements on each side of filter pads and laboratory handsheets. Just one measurement is made on the top side of membrane filter samples.

NOTE: When measuring laboratory handsheets, these ought to be stacked in a way to guarantee an opaque pile of sheets.

5.2 Overview of measurements

The luminosity Y, R_{457} and the reflectance factors R_∞ and R_0, the CIELab colour coordinates (L*, a*, b* values) are measured from the samples. The absorption coefficient *k* and scattering coefficient *s* and ERIC are determined based on reflectance factors, commonly provided by the measuring device. The Ink Elimination IE is calculated from the absorption coefficients or ERIC of the undeinked, deinked and unprinted samples. The dirt specks particle area A is analysed with the help of scanner based image analysis system. For more details see the following chapters.

[1] It has been shown that the results with light source C/2° with an edge filter of 420 nm and D65/10° with an edge filter of 420 nm are nearly identical. For this reason and because the method measured originally C/2° it was decided that the measurement is performed with C/2°.

Table 1: Overview of sample types, parameters to be measured and corresponding chapter in INGEDE Method 1

Sample	Chapter in INGEDE Method 1	Parameters
Filter pad	5.1.	Y, R_{457}, ERIC, IE_{700}, IE_{ERIC}, L*, a*, b*
Handsheet without recirculated white water	5.3	Dirt particle area A
Handsheet with re-circulated water	5.4	ERIC, *s*, *k*, IE_{700}, IE_{ERIC}
Filtrate	5.5	Y, ΔY

5.3 Reflectance factors

The reflectance R_∞ is measured with a device according to ISO 2469 at a wavelength of 700 nm and 950 nm. R_∞ is the reflectance factor of a layer handsheets or pad that is thick enough to be opaque.

R_∞ at a wavelength of 457 nm (brightness) is measured with a device in accordance with the standard ISO 2470-1 ISO brightness.

The reflectance of a single sheet R_0 is measured at 700 and 950 nm with the stated conditions above. In accordance to ISO 9416, the single sheet must meet the requirements, that the opacity must not exceed 95 %. R_0 is the reflectance factor of a single sheet of paper with a black cavity as backing.

5.4 Y, L*, a*, b* and opacity

The luminosity Y is determined in accordance with DIN 6174. The CIELab colour coordinates L*, a* and b* are determined according to ISO 5631-2. From handsheets, the opacity following ISO 2471 is determined.

5.5 Determination of the light absorption coefficient k and the light scattering coefficient s

The light absorption coefficient *k* in m^2/kg and the light scattering coefficient *s* in m^2/kg are obtained by means of the measured reflectance factors R_0 and R_∞ as well as basis weight according to Kubelka-Munk in compliance with ISO 9416. In addition to ISO 9416 where *k* and *s* are obtained by means of the tristimulus filter used to determine reflectance factor, the reflectance factor in the case of Ink Elimination has to be determined at a wavelength of either 700 nm or 950 nm.

The use of the method ISO 9416 is restricted to samples with opacity less than 95 %, otherwise inaccuracy in calculation of *s* will occur, for more details see ISO 9416. Therefore it is not possi-

ble to determine *s* from filter pads or thick handsheets. Handsheets with recirculated water meet possibly the requirements of ISO 9416.

Equation 1: Light absorption coefficient in m^2/kg

$$k = s \cdot \left(\frac{(1 - R_\infty)^2}{2 R_\infty} \right)$$

R_∞ is expressed as decimal.

Equation 2: Light scattering coefficient in m^2/kg

$$s = \left(\frac{1000}{w} \right) \cdot \left(\frac{R_\infty}{1 - R_\infty^2} \right) \cdot \ln \frac{R_\infty (1 - R_0 \cdot R_\infty)}{R_\infty - R_0}$$

w= grammage (g/m^2)

R_∞ and R_0 are expressed as decimal.

5.6 Ink Elimination IE

Generally speaking, Ink Elimination is obtained using the light absorption coefficient *k* of the undeinked, deinked and unprinted samples. For known scattering coefficient *s*, Equation 1 gives the determination of *k*. Ink Elimination (IE) is calculated as follows:

Equation 3: Ink Elimination in %

$$IE = \frac{K_{UP} - K_{DP}}{K_{UP} - K_{unpr}} \cdot 100$$

Where:

UP= undeinked pulp

K_{UP}= light absorption coefficient k of undeinked sample

DP= deinked pulp

K_{DP}= light absorption coefficient k of deinked sample

unpr= unprinted sample

K_{unpr}= light absorption coefficient k of unprinted sample

It is assumed that the difference in *s* of filter pads before and after the flotation is as magnitude as the losses of pulp components during hand sheet preparation. Assuming *s*=const, Equation 3 can be used simplified by neglecting the light scattering coefficient *s*, calculating IE only with R_∞ from filter pads (Equation 4). Alternatively, the scattering coefficient *s* of the deinked pulp in the

investigated plant or of the investigated sample has to be determined for the specific sampling point in the process and its value used as approximation.

Equation 4: Ink Elimination in %

$$IE = \frac{\left(\frac{\left(1-R_{\infty,UP}\right)^2}{R_{\infty,UP}}\right) - \left(\frac{\left(1-R_{\infty,DP}\right)^2}{R_{\infty,DP}}\right)}{\left(\frac{\left(1-R_{\infty,UP}\right)^2}{R_{\infty,UP}}\right) - \left(\frac{\left(1-R_{\infty,unpr}\right)^2}{R_{\infty,unpr}}\right)} \cdot 100$$

Where:

$R_{\infty,UP}$ = reflectance factor R_∞ of undeinked sample

$R_{\infty,DP}$ = reflectance factor R_∞ of deinked sample

$R_{\infty,unpr}$ = reflectance factor R_∞ of unprinted sample

R_∞ is obtained either at a wavelength of 700 nm (for IE_{700}) or at 950 nm (for IE_{ERIC}).

- **IE_{700}**

The R_∞-values measured at 700 nm on filter pads of deinked pulp (DP) and undeinked pulp (UP) are not used in %, but as absolute values, e. g. 0,69. If no unprinted samples are available, the value for the term $(1–R_{\infty,unpr})^2/ R_{\infty,unpr}$ may be set to 0.

Equation 5: IE_{700} in %

$$IE_{700} = \frac{\left(\frac{\left(1-R_{\infty,UP}\right)^2}{R_{\infty,UP}}\right) - \left(\frac{\left(1-R_{\infty,DP}\right)^2}{R_{\infty,DP}}\right)}{\left(\frac{\left(1-R_{\infty,UP}\right)^2}{R_{\infty,UP}}\right) - \left(\frac{\left(1-R_{\infty,unpr}\right)^2}{R_{\infty,unpr}}\right)} \cdot 100$$

- **IE_{ERIC}**

The ERIC values (chapter 5.7) are measured at 950 nm of DP and UP. If no unprinted samples are available, the value for $ERIC_{unpr}$ may be set to 0.

If the deinkability of different paper grades is to be compared, the measurement of the corresponding unprinted papers is recommended.

Equation 6: IE_{ERIC} in %

$$IE_{ERIC} = \frac{ERIC_{UP} - ERIC_{DP}}{ERIC_{UP} - ERIC_{unpr}} \cdot 100$$

5.7 ERIC

The calculation for ERIC follows ISO 22754 and TAPPI T567 om-09:

Equation 7: ERIC

$$ERIC = (k_{Sheet} / k_{ink}) \cdot 10^6$$

R_0 and R_∞ are determined at a wavelength of 950 nm of specimen, which meet the requirements to ISO 9416. If a lightweight sample is not available, make certain that the scattering coefficient *s* is truthfully representative of the sample being tested. For this case, ERIC value and the value for *s* has to be stated together in the report.

5.8 Filtrate Darkening

The filtrate darkening ΔY is the difference in luminosity Y between membrane filter pads made from filter pad filtrate and tap water as reference. The preparation of membrane filter pads is described in INGEDE Method 1.

The luminosity Y of the filtrate membrane filter pad ($Y_{filtrate}$) and of the reference membrane filter pad ($Y_{reference}$) are determined at identical conditions according chapter 5.4. $Y_{filtrate}$ is the average value of the two membrane filter pads- see INGEDE Method 1. By subtracting $Y_{filtrate}$ from $Y_{reference}$, ($\Delta Y = Y_{reference} - Y_{filtrate}$), all factors affecting filtrate quality and not attributable to the pulp are eliminated.

5.9 Procedure for dirt particle measurement (A)

A scanner based image analysis system is to be used for the determination of the dirt specks area “A”.

The top and the bottom side of at least two laboratory handsheets per specimen are to be assessed by the image analysis system. The arithmetic mean of min. 4 measured values is to be calculated. This mean value is to be taken as the dirt specks area “A”.

Scanning conditions:

The sheets should be free of crinkles and waves to lie flat on the scanner. The sheets are to be scanned individually. As background an opaque batch of woodfree copy paper (min. 5 sheets with a luminosity of Y= 84 ± 2 measured with illumination D65/10° and 420 nm edge filter) should be used. Every handsheet should be scanned one time from top and from the bottom with 8-bit grey modus, 600 dpi and reflective light.

If the scanner is idle for more than 15 minutes a blank scan has to be made in advance of any new measurement.

Image analysis software parameterisation: The threshold values and the size classification are to be defined as described in the appendix.

In case of using DOMAS image analysis system the following parameters are recommended:

- The threshold of measurement is determined by file “ingede2.sw“
- The size classification is determined by file “ingede2.kls”
- Select: “Circular specimen with border” if specimen is circular
- Select threshold method “file” and select “ingede2.sw”
- Select size classification “circle equivalent diameter” and select “ingede2.kls”
- Select: image source “scan series” and select “specks_1.scn”
- Set “No of specimens” to “4”
- Select: “Average series of results”

In case of using SIMPALAB software (Techpap SAS):
Select the family "Ingede2.cfg", to take pre-installed parameters for the measurement of optical characteristics into account. The threshold of measurement, sizes for classification (50–100 µm, 100–150 µm, …) and other parameters are already determined in the file “ingede2.cfg” and set automatically.

6 Report

When measuring laboratory handsheets and filter pads, where top and bottom are measured separately, the mean of the two values should always be reported. If the top and bottom values differ considerably, the individual values should also be reported.

The following should be noted in the test report:

- Type of test specimens the optical measurements refer to (laboratory handsheets or filter pads),
- Type of light and the inspection angle for which the values were calculated,
- The light absorption coefficient *k* in m^2/kg, the light scattering coefficient *s* in m^2/kg, ERIC or R_∞ at 700 nm of the undeinked and deinked pulp samples and the Ink Elimination derived in %,
- For filtrate samples, the test report should list the mean of both optical measurements ($Y_{filtrate}$ and $Y_{reference}$).

7 References

7.1 Cited Standards and methods

- IFRA Newsshade 2003, IFRA Special Report 1.11.2.
- INGEDE Method 1: Test sheet preparation of pulps and filtrates from deinking processes
- ISO 187: Paper, board and pulps: Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples (1990)
- ISO 2469: Paper, board and pulps – Measurement of diffuse radiance factor (diffuse reflectance factor) (2014)
- ISO 2470-1: Paper, board and pulps – Measurement of diffuse blue reflectance factor – Part 1: Indoor daylight conditions (ISO brightness) (2009)
- ISO 2471: Paper and board – Determination of opacity (paper backing) – Diffuse reflectance method (2008)
- ISO 4119: Pulps: Determination of stock concentration (1995)
- ISO 5269-2: Pulps – Preparation of laboratory sheets for physical testing – Part 2: Rapid-Köthen method (2004)
- ISO 5631-2: Paper and board - Determination of colour by diffuse reflectance - Part 2: Outdoor daylight conditions (D65/10 degrees) (2014)
- ISO 9416: Determination of light scattering and absorption coefficients (using Kubelka-Munk theory) (2009)
- ISO 22754: Pulp and paper - Determination of the effective residual ink concentration (ERIC number) by infrared reflectance measurement (2008)
- TAPPI T 567 om-09: Determination of effective residual ink concentration by infrared reflectance measurement (2009)

7.2 Sources of the material used

DOMAS

- Files:
  "ingede2.sw", and "ingede2.kls": www.INGEDE.org
- Software:
  "DOMAS Calibration Tester", PTS Heidenau and Munich www.ptspaper.de
- Scanner:
  DOMAS Scanner*Advanced*, PTS Heidenau and Munich
- Image analysis software:
  DOMAS 3.0, PTS Heidenau and Munich

SIMPALAB

- Files:
  "ingede2.cfg"
- Software:
  SIMPALAB_[]_3.00.[1x], Techpap SAS Grenoble
- Scanner:
  A list of compatible scanners is available from Techpap SAS Grenoble
  (www.techpap.com, sales@techpap.com)

Contact:
INGEDE e. V. (International Association of the Deinking Industry)
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74321 Bietigheim-Bissingen, Germany
Tel. +49 7142 7742-81
Fax +49 7142 7742-80
E-Mail office@ingede.org
www.ingede.org

Annex

Threshold value determination

Use this formula: Threshold = mean – k_factor

Linear interpolation between two pairs of values gives the threshold value demanded.

mean (8-bit grey value)	k_factor
167,42	35,81
202,01	30,43
221,37	30,91
239,17	35,38
248,16	33,75

For use within DOMAS software the threshold of measurement is determined by the file “ingede2.sw“(see software attachment).

For use within Techpap SIMPALAB Software the threshold of measurement is determined by file “ingede2.cfg”.

Size classification

Definition of the size classes of an equivalent diameter of a circle:

from (µm)	to (µm)
> 50	≤ 100
> 100	≤ 150
> 150	≤ 200
> 200	≤ 250
> 250	≤ 500
> 500	≤ 50 000

For use within DOMAS software the size classification is determined by file “ingede2.kls“ (see software attachment).

For use within Techpap SIMPALAB Software the size classification is determined by the file “ingede2.cfg”.

INGEDE Method 4

April 2013

Analysis of macrostickies in pulps

This document was originally developed and launched by INGEDE, its members and its research partners. In the frame of the EcoPaperLoop Project INGEDE Method 4 was translated into several languages. However, in case of any discrepancies the only valid version is the one in English language.

INGEDE Method 4 **April 2013** 13 Pages	**Analysis of macrostickies in pulps**	INGEDE International Association of the Deinking Industry

Introduction

Stickies in pulps originate from tacky components in paper for recycling. They cause problems during paper production and converting as well as quality defects.

This method is widely accepted to measure the macrosticky content of pulps.

1 Scope

This INGEDE Method is used to analyse macrostickies in pulps.

2 Terms and definitions

Macrostickies:

Tacky components originating from paper for recycling which can be analysed from the residues of a laboratory screening (see also the corresponding leaflet of ZELLCHEMING).

3 Principle

The method describes a laboratory screening procedure for pulps of a paper recycling process. The reject of this screening procedure is prepared in such a way that the macrostickies can be determined by means of an image analysis system.

4 Equipment and auxiliaries

4.1 Equipment

4.1.1 Disintegration

Any device which fulfils the requirements of ISO 5263-1 may be used for disintegration of samples.

4.1.2 Screening

Macrostickies can be separated from recycled pulp suspensions using various laboratory screening devices. Possible screening devices are the Haindl classifier (ZM V/1.4/86), the Somerville tester (TAPPI T 275 sp-07) or the Pulmac Master Screen (TAPPI 274 sp-08).

4.1.3 Slotted plate

For pulps in deinking, the use of a slot width of 100 µm is recommended. Other slot widths have to be reported as deviation from the method.

NOTE:

Screening investigations with slotted plates of nominally the same slot width showed significant differences in the screening result (see INFOR Project 118). The maximum slot width correlates with the macrosticky area. Therefore it is recommended to measure all slots widths on the slotted plate. A ZELLCHEMING test method for the quality requirements of slotted plates in laboratory screening devices is available.

4.1.4 Reject dewatering and drying

Any device which fulfils the requirements of ISO 5269-2 may be used for dewatering and drying the dewatered screening rejects e. g. the Rapid-Köthen unit. Additionally, an oven which fulfils the specifications of ISO 287 is required.

4.1.5 Image analysis

An image analysing system comprising a flatbed scanner and PC with a suitable control and analysis program is used for the measurements. The scanner is to be calibrated to ensure reproducibility of the measurements.

Technical requirements of the flatbed scanner:

- Scanning area ≥ ISO A4
- Optical scan resolution ≥ 2000 dpi
- Colour depth 48 bit
- Optical density DMAX ≥ 4,0

Requirements on measuring accuracy of flatbed scanner after warm-up period (see scanner manual) and under scanning conditions (see chapter 5.6).

- Reproducibility of mean grey value (8 Bit) is ±1. That means that an ISO A4 sample has to be scanned 10 times without any movement of the sample; all mean grey values of total sample area should be within 2 grey values.
- Deviation of colour value (RGB 8 Bit) ≤ 5. That means that after calibration a scanned image of IT8-Target should not deviate more from associated reference file than ± 5 values in every colour channel R, G, B.

Suitable scanner: DOMAS ScannerAdvanced or Techpap SIMPALAB proposed Scanner

“ScannerAdvanced” is a name given by PTS to a commercial scanner that
was accredited by PTS. This scanner device is delivered with the DOMAS 3.0 version.

The software should be able to detect white particles on a black background. Suitable software package are DOMAS 3.0 and above image analysis software as well as Techpap SIMPALAB software.

4.2 Test material

The following testing material may be used:

- Black water-based ink, e. g. Pelikan No. 4001
- One sided, silicone-coated release paper (60 g/m²)
- Filter paper: medium to large pores, medium filtration speed, machine finished, good wet strength, white, e.g. Munktell Filtrak 1289, 240 mm diameter
- Special fused alumina powder: white, sharp-edged particles, grain size 220 according to FEPA Method.

(Sources of supply see chapter 7.4)

5 Procedure

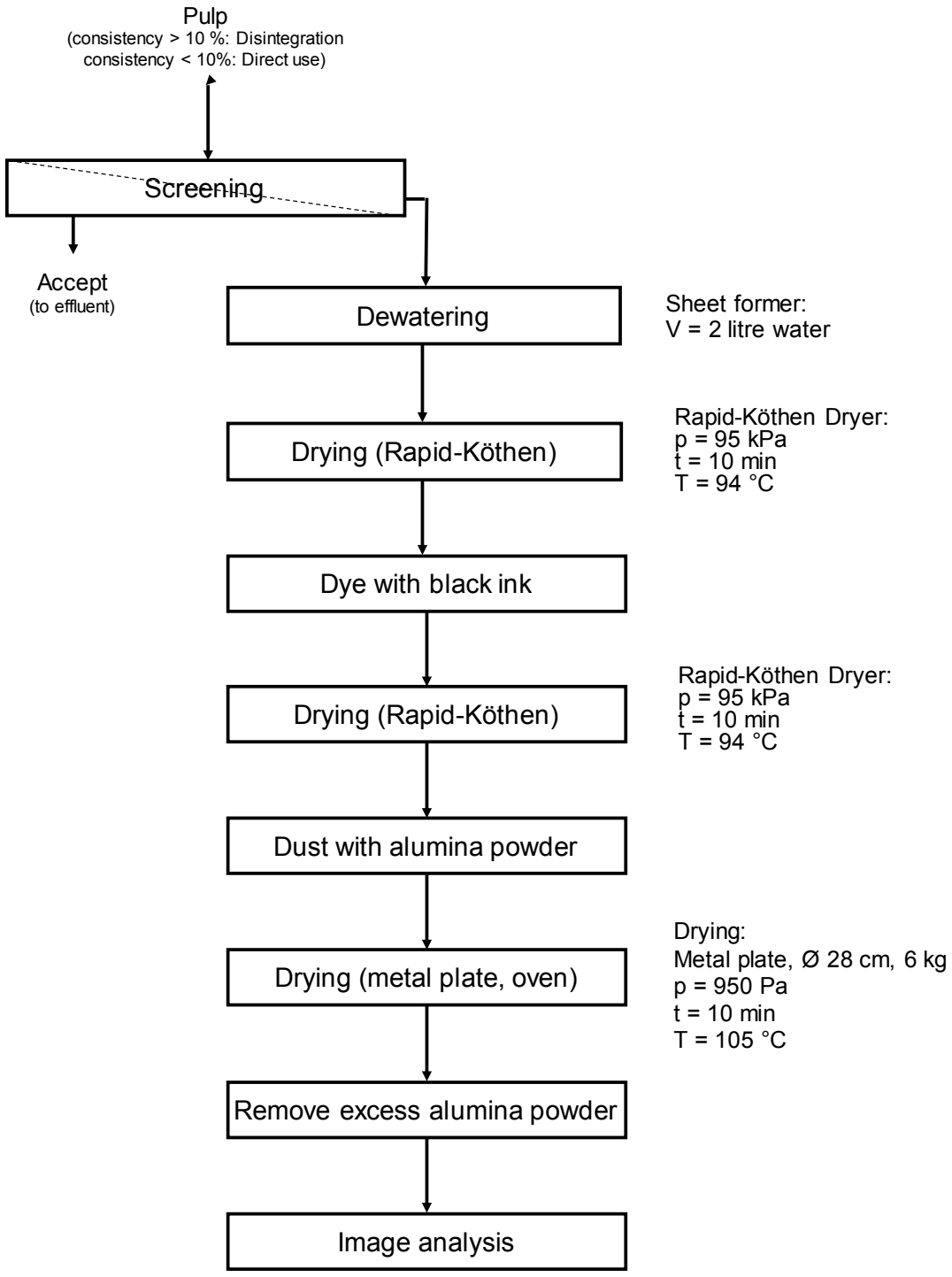

Figure 1: Test procedure INGEDE Method 4

5.1 Sampling and sample preparation

Pulp suspensions with a consistency up to 10 % can be used immediately for screening without further preparation. However, pulp with higher consistency must be disintegrated before screening. 50 g oven-dry pulp in 2000 ± 25 ml is disintegrated. Disintegration is fulfilled in a device in accordance with ISO 5263-1 whereby the disintegration process is restricted to five minutes. Longer periods of mechanical stress should be avoided in order to prevent changes of the sticky size distribution in the sample.

5.2 Screening

5.2.1 General

For a statistically sound statement about the macro sticky content, the screening of three individual portions, each containing 50 g of oven-dry material from one sample, is recommended. Some pulps may cause difficulties during screening due to high long fibre content or high level of contamination. In this case the pulp amount can be split into portions (e.g. 2 x 25 g) and/or the screening time can be elongated. In case of Pulmac Master Screen the reduction is preferred vs. the change of the screening programme. A reduction of the sample's quantity is also necessary if the load of stickies is so high that the stickies overlap heavily after contrasting.

Any deviation has to be reported.

When using a plastic screen plate, mechanical stress can lead to material fatigue and destruction of the slotted plate. For this reason the use of a metal plate is recommended.

5.2.2 Screening conditions overview

The following table gives an overview about screening equipment and conditions.

Table 1: Screening conditions

Equipment	Reference	Water flow	Stroke	Duration (Pulp input + further screening)
Haindl classifier	ZM V/1.4/86	10 l/ min	480 double strokes per minute	5 min + 5 min
Somerville tester	T 275 sp-07	8,6 l/ min	700 rpm	2 min + 18 min
Pulmac Master Screen	T 274 sp-08	Depends on programme setting (Modus B)		

5.2.3 Haindl classifier

The screening with the Haindl classifier is performed according to ZM V/1.4/86, without using the McNett unit. In order to guarantee problem-free screening of 50 g of oven-dry pulp, unlike ZM V/1.4/86 the screening conditions should be set up as follows. The stroke frequency of the membrane should be increased to 480 double strokes/ minute (maximum stroke rate). Because of the resulting turbulence increase in the screening chamber, the height of the cylindrical supply vessel wall should be increased from 130 mm to 370 mm. The container can be extended using an acrylic glass top. The washing water flow should be 10 litres per minute for the entire screening duration. After continuously adding pulp for 5 minutes, the pulp continues to be screened for 5 minutes until screening is complete.

5.2.4 Somerville tester

The screening in a Somerville tester is performed referring to T 275 sp-07. 50 g oven dry pulp is poured into the screen box within the first 2 minutes of 20 minutes overall screening duration.

5.2.5 Pulmac Master Screen

When using the Pulmac Master Screen, 50 g of oven-dry pulp is added to the supply chest. The screening which follows is automatic. Programme setting should be “Modus B”. Before screening, a wet filter paper which retains the reject when the screening is complete must be placed onto the sieve in the dewatering unit (autofilter).

5.3 Dewatering the reject

The reject is flushed from the slotted plate into a container using about one litre of water. Using a moistened white paper filter above the sheet forming wire the reject is dewatered in the sheet former (Rapid-Köthen model). It is advisable to operate the sheet former manually. When the reject sample and an additional litre of water are in the sheet former, the aeration is started before dewatering. After dewatering, the specimen which has been formed is placed onto a couching board with the bottom of the filter (reject-free side). If the load of stickies is so high that the stickies overlap after contrasting (chapter 5.5), the reject has to be portioned to several filter papers. It is also possible to separate overlapped stickies carefully on the filter or transfer the larger sticky fragments on an additional filter. Big, cubic sticky particles must be transferred on an additional filter (in a later step smaller and flat particles are better covered by the alumina powder).

When using the Pulmac Master Screen, the reject is dewatered in the unit automatically using the same type of filter paper. The dewatered specimen can be removed after the screening is complete. It is also laid onto a couching board with the bottom side of the filter.

5.4 Drying

The top side of the specimen is then covered with the coated side of the silicone-coated sheet of release paper. Then the sample is dried for 10 minutes in the sheet dryer (Rapid-Köthen model) at 94 C and a pressure of 95 kPa.

5.5 Sticky examination

After drying, the stickies are examined by utilising their adhesive properties in order to provide the contrast to the specimen's background which is required for image analysis. Before removing the silicone coated release paper the specimen is cooled down for a short time. Heavy sticky particles could adhere to the silicon paper and must be transferred back to the filter paper.

The dried specimen is then drawn through a submersion bath containing black water-based ink, so that the entire surface is covered. The dyed specimen is then laid with its bottom side on a piece of blotting paper (bleached sheet of cellulose or tissue), so that any excess ink is absorbed. Then the specimen is dried for another 10 minutes, the top side covered with the previously used silicone-coated release paper.

In order to avoid discoloration of the drying equipment, the specimen should be placed between two couching boards during drying.

Subsequently after a short cooling down, the specimen is completely covered with a thick, even layer of white special fused alumina powder, the top and bottom sides are covered with couching board and it is then dried for 10 minutes in an oven at 105 °C. The specimen is loaded with a pressure of 950 Pa (6 kg metal plate, Ø 28 cm) to fix the powder on the tacky areas. The metal plate should be stored in the oven permanently to keep the high temperature. After the procedure is complete, the specimen should be removed from the oven. Excess, loose powder has to be removed with a soft cosmetic brush, without applying pressure, whilst holding the specimen in a vertical position.

After the stickies have been contrasted inspect the stickies visually. It is important that the stickies do not overlap. The visual inspection also serves to check whether all white hydrophobic impurities such as pieces of plastic film have been removed. In order to do this, the components to be eliminated should either be removed using tweezers or marked using a black permanent marker so that they are not detected during the subsequent image analysis.

5.6 Image analysis

The prepared specimen is then analysed using a scanner-based image analysis system. When selecting the measuring area, the preparation area should be used in order to analyse as many of the stickies which were retained during screening as possible. The largest possible measuring area should be selected.

The top side of the recommended 3 specimens per sample are to be assessed by the image analysis system. The arithmetic mean of the 3 measured values is to be calculated. Scanning conditions: The sheets should be free of crinkles and waves to lie flat on the scanner. An opaque batch of black carton should be used as background. Every specimen should be scanned one time from top side with 8-bit grey modus, 600 dpi and reflective light.

If the scanner is idle for more than 15 minutes, a blank scan has to be made before any new measurement.

Parameterisation of image analysis software: The threshold value and the size classification are defined in the following. Other threshold setting is regarded as deviation from this method and must be reported. When setting the class limits, the size of the slots in the slotted plate which

was used for screening is set as lower limit (regular 100 µm). Smaller stickies cannot be expected because of the sticky surface increase which is associated with the drying process. The final class may not have an upper limit, so that all stickies are recorded.

The amount of pulp used (typically 50 g oven-dry) has to be known as input value for the image analysis software to calculate the macrosticky area as described in chapter 5.7.

In case of using DOMAS image analysis system the following parameters are recommended:

- Set “Slot width” to “100” (µm)
- Select “Circular sample with border”
- Select “Light contrasted stickies”
- Select Threshold method “fixed threshold” and set parameter to “95”
- Select “Size classification” with “circle equivalent diameter” and select “ingede4.kls”
- Set “Pulp mass depended” and “…g”
- Select “Image source” “scan series” select “stickies_1.scn”
- Set “No. of samples” to “3”
- Select “Average series of results”

NOTE:

If the load of stickies is very high, it is recommended to reduce the amount of stickies on the filter paper instead of changing the threshold. In that case follow the same procedures as for over-lapping (chapters 5.3 and 5.5).

In case of using Techpap SIMPALAB software:

- Open Family (from Menu Parameter)
- Select the family "ingede4.cfg" from the list

All the settings are pre-installed for the measurement of stickies. The threshold of measurement, sizes for classification (100–200 µm, ...) and other parameters are already determined in the file "ingede4.cfg", and set automatically.

5.7 Calculation of the macrosticky content

The results of the image analysis should be given in mm² of stickies per m² of specimen. This value should be then converted into mm² of sticky area per kg of pulp (see Equation 1). The specimen area which was actually measured by the image analysis system in relation to the covered filter paper surface (or in maximum the inner diameter of the sheet former) and the amount of material used during screening (recommended 50 g of oven-dry pulp) have to be taken into consideration.

Equation 1: Macrosticky area in mm^2/kg

$$Macrosticky\ area = \frac{Sticky\,area\ in\ \frac{mm^2}{m^2} \cdot Specimen\ area\ in\ m^2}{Amount\,of\,material\ in\ kg}$$

The use of 50 g of oven-dry pulp and dewatering using the Rapid-Köthen unit results in a conversion factor of 0,634 for converting the area-based sticky area into a weight-based sticky area. Then the mathematical mean of the individual results should be calculated for the three specimens which were made from each pulp sample.

It is advisable to calculate the coefficient of variation and to repeat the measurements if the coefficient of variation is higher than 10 %.

The measurements can be shown separately for the determined size classes and also as the total sticky area for all size classes.

6 Report

The following should be noted in the test report:

- Designation of the sample
- Type of screening unit used
- Type of slotted plate used
- Type of image analysis system used
- Threshold setting if other than defined
- Average macrosticky content in mm²/kg of the recommended three individual samples and coefficient of variation
- Any deviation from this method.

7 References

7.1 Cited standards and methods

- ZELLCHEMING Technical Leaflet RECO 1, 1/2006 “Terminology of Stickies”; www.zellcheming.com/service/ , follow “leaflet” and “RECO”
- ZM V/1.4/86: Simultaneous determination of shives and fibre fraction content (in German); www.zellcheming.com/service, follow “leaflet” and “TEST”
- ZELLCHEMING Technical Leaflet RECO 1, “Anforderungen an die Güte von Schlitzplatten für Labor-Sortieraggregate” (engl.: “Quality Requirements of Slotted Plates for Laboratory Screening Devices”); www.zellcheming.de
- TAPPI T 275 sp-07: Screening of Pulp (Somerville-Type Equipment)
- TAPPI T 274 sp-08: Laboratory screening of pulp (Master Screen-type instrument)
- ISO 5263-1 (2004): Pulps – Laboratory wet disintegration – Part 1: Disintegration of chemical pulps
- ISO 5269-2 (2005): Pulp – Preparation of laboratory sheets for physical testing – Part 2: Rapid-Köthen method
- ISO 287 (2009): Paper and Board – Determination of moisture content – Oven drying method
- FEPA: www.fepa-abrasives.org

7.2 Literature and other related documents

- Ackermann, C.; Putz, H.-J.; Göttsching, L.: INGEDE Method for the Analysis of Macro Stickies in DIP. Das Papier 51 (1997), no. 6, 271-282 (in German)
- Ackermann, C.; Putz, H.-J.; Göttsching, L.: Improved Macro Sticky Analysis for DIP based on Screening. Progress in Paper Recycling 7 (1998), no. 2, 22-32
- German INFOR Project 118: Improvement of Reproducibility of standardized Macrosticky Methods (Final Report: PMV/PTS September 2009)
- H.-J. Putz, E. Hanecker: Untersuchung relevanter Einflüsse auf Makro-Stickyergebnisse. Wochenblatt für Papierfabrikation, 139 (2011), no.2, 116-123

7.3 Sources

The INGEDE Method is based on the INGEDE Project 38 94 PTS/PMV “Developing methods for performing quantitative analyses of micro and macro stickies”.

PTS (www.ptspaper.de), PMV (www.pmv.tu-darmstadt.de)

7.4 Sources of supply

Silicone paper:

- 60 g/m^2, for example from Gieselmann Stanztechnik GmbH, Germany, www.gieselmann-stanztechnik.de

Special fused alumina powder:

- Elektrokorund Alodur SWSK 220 from Treibacher Schleifmittel
- Obtained from PMV (Papierfabrikation und Mechanische Verfahrenstechnik), TU Darmstadt, Alexanderstraße 8, 64283 Darmstadt, Germany

Filter paper:

- Type 1289, Munktell & Filtrak GmbH, Niederschlag 1, 09471 Bärenstein, Germany, www.munktell.com

Ink:

- Pelikan No. 4001 or Parker Quink

DOMAS

- File:

  “ingede4.kls”: www.ingede.org
- Software

  “DOMAS Calibration Tester”, PTS Heidenau and Munich
- Scanner:

  DOMAS ScannerAdvanced, PTS Heidenau and Munich
- Image analysis software:

  DOMAS 3.0, PTS Heidenau and Munich

SIMPALAB

- File:

  “ingede4.cfg"
- Software:

  Simpalab, current version 3.02.00 Techpap SAS Grenoble
- Scanners:

A list of compatible scanners is available from Techpap SAS Grenoble (www.techpap.com, sales@techpap.com).

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Annex A

Size classification

Definition of the size classes of an equivalent diameter of a circle:

from (µm)	**to (µm)**
>100	≤ 200
>200	≤ 300
>300	≤ 400
>400	≤ 500
>500	≤ 600
>600	≤ 1 000
>1 000	≤ 1 500
>1 500	≤ 2 000
>2 000	≤ 3 000
>3 000	≤ 5 000
>5 000	≤ 10 000
>10 000	≤ 20 000
>20 000	≤ 50 000
>50 000	≤ 200 000

For use within DOMAS software the size classification is determined by file “ingede4.kls“(see software attachment).

For use within Techpap SIMPALAB Software the size classification is determined by file “ingede4”.