DEVELOPMENT OF A EUROPEAN DEINKABILITY TEST METHOD AND RESULTS OF SELECTED TYPES OF PRINTED PRODUCTS

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ABSTRACT
The International Association of the Deinking Industry (INGEDE) has established a database containing a high number of deinkability test results for the most relevant types of printed products. The INGEDE finances research work in order to improve the existing deinkability test method for printed products (INGEDE Method No. 11) and to harmonize it with methods being in use in other laboratories leading to comparable results with acceptable reproducibility. Details of the procedure and the parameters are explained in this paper.

INTRODUCTION
Usually paper is recyclable as it is produced on paper machines. During converting of paper into paper products materials can be applied on paper which can affect their recyclability, e. g. wax, or adhesive applications. Regarding the printing ink it is used the term deinkability. This is meant for the ability of a printed product to be deinked with the established flotation process in stock preparation lines of graphic paper producers. Good deinkability requires first the possibility to detach the ink of the fibers and fillers using deinking chemistry and shear forces in the pulping process. The ink detachment sets the stage for the success of the flotation process, which separates the ink as a hydrophobic component and leads to a significant brightness gain. This can be quantified by INGEDE Method No. 5 "Evaluation of Printing Ink Detachment by Hyperwashing with the Haindl-McNett Classifier" [1]. The deinkability test method characterizes the deinked pulp quality and compares it to the undeinked pulp. Parameters are required, describing the behaviour of the printed products to be tested during their laboratory processing [2].

The demand on a deinkability test method for printed products has its routes in developments of printing technology resulting in problems in deinking lines. The mechanism and the standard recipe for the deinking chemistry do not work satisfactory if the ink particles are of wrong size or even water soluble. This is the reason for further parameters like dirt specks or filtrate darkening. The idea is to avoid problems in the process of recycling paper production and product quality by control of the feed. If the feed should not only be observed but also be influenced in order to save recovered paper quality, the printed products have to be examined. This is done in laboratory scale.

The INGEDE takes care for the quality of recovered paper which is the most important raw material for its members. This is of special importance because of the fact that in Germany as well as in Europe recycling targets exist for paper. A voluntary agreement was presented by the CEPI (Confederation of the European Paper Industry) in 2000 by the "European Declaration on Recovered Paper" which covers all paper products and set a recycling target of 56 % in 2005 for all paper products. The members of INGEDE give a budget for research work and out of that a considerable investment has been made in developing the INGEDE Method 11 and in building up a database. This database contains five parameters evaluating the deinkability of printed products and for them orientation values have been discussed with all members of the paper chain. After these first experiences it is currently considered as necessary to harmonize the recyclability test methods throughout Europe leading to comparable results with acceptable reproducibility in the different laboratories, which will test deinkability [3]. The project for the deinkability test method has made progress in harmonization, simplification and better reproducibility. It is an objective procedure for the determination of criteria to evaluate the recyclability in terms of deinkability and decide between poor or good recyclable graphic paper products. The project which has started in 2002 is performed by the leading European paper institutes working in the area of recycling: CTP (Centre Technique du Papier, Grenoble, France), PTS (Papiertechnische Stiftung, München, Germany) and PMV (Fachgebiet Papierfabrikation und Mechanische Verfahrenstechnik (former IfP), Darmstadt, Germany).

TEST METHOD
INGEDE Method No. 11, published in the internet [4], describes a procedure to evaluate the deinkability of print products sub-sequent to flotation. The individual treatment stages simulate a mill process with the flotation deinking of printed recovered paper – including a preceding accelerated ageing stage of the sample material. The test is carried out under defined conditions in the laboratory. This method is basis for the actual harmonization project and is shown including the simplifications in Figure 1. The most important modifications:

- detailed description for the preparation of the deinking chemistry and its application before pulping
- specimen preparation (the preparation of handsheets with recirculated water is not necessary anymore)
- evaluation of the flotation process by calculation of the parameter Ink Elimination (IE) with

R_∞-values of filter pads of deinked pulp (DP) and undeinked pulp (UP) as shown in Formula 1

- characterization of DP using the optical parameters Y, a* and b* of filter pads and dirt specks of handsheets prepared with fresh water
- filtrate darkening (membrane filters are prepared according to INGEDE Method No. 3 “Optical Evaluation of Deinking Filtrates” with the filtrate of DP instead of UP) [5].

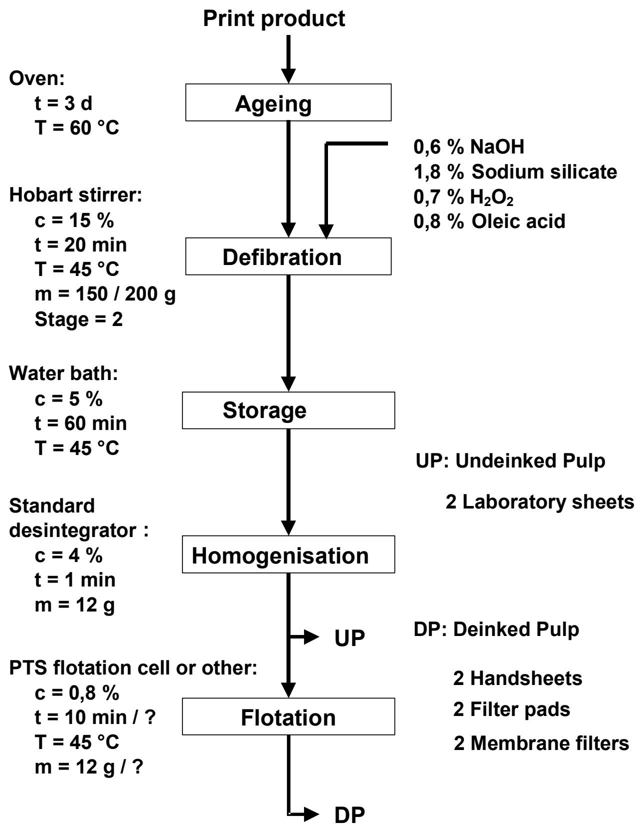

Figure 1: Flow chart test procedure assessment of deinkability, draft for new INGEDE Method No. 11.

$$IE_0 = \frac{\left(\frac{(1-R_{\infty,UP})^2}{R_{\infty,UP}}\right) - \left(\frac{(1-R_{\infty,DP})^2}{R_{\infty,DP}}\right)}{\left(\frac{(1-R_{\infty,UP})^2}{R_{\infty,UP}}\right)}$$

Formula 1: Calculation of the Ink Elimination with the R_∞-values of reflectance measurements on filter pads in the near infra red area.

DEINKABILITY EVALUATION CRITERIA – ORIENTATION VALUES

The members of the paper chain already agreed on 5 criteria for the evaluation of the deinkability of printed products. To classify a printed product as “recyclable”, all five parameters must fulfill minimum requirements that differ to some extend both according to the category of printing process used and according to the type of paper [6]. Figure 2 shows the numeric values fixed as orientation values, based on the data base of the INGEDE. The parameters are named and explained in Figure 3. The additional parameter b* for the colour locus is not planned to get a 6th orientation value but should be under observation, as the project delegates of the companies of different European countries pointed out.

Print Product	Y -	IE %	a* -	Δ Y -	A mm²/m²
Newspapers	≥ 53	≥ 57	≥ - 2.5 to ≤ + 1.0	≤ 10	≤ 500 mm²/m² free of large visible impurities
Offset Magazines & Flyers, uncoated	≥ 55	≥ 57		≤ 10	
Offset Magazines & Flyers, coated	≥ 67	≥ 64		≤ 10	
Rotogravure Magazines, uncoated	≥ 64	≥ 69		≤ 10	
Rotogravure Magazines, coated	≥ 67	≥ 74		≤ 10	
Rotogravure Catalogues	≥ 61	≥ 71		≤ 10	

Figure 2: Orientation values for several printed products.

Objectives	*Evaluated Parameter*	*Testing Standard*
High reflection	Luminosity Y of deinked sample	INGEDE Method 2: Evaluation of optical characteristics of deinked pulp
High ink removal	Ink Elimination IE	INGEDE Method 10: Evaluation of ink removal during deinking of recovered paper
High optical cleanliness	Dirt particle area A of deinked pulp	Scanner based image analysis
No colour shade	Value a* of deinked pulp	DIN 6174: Colourimetric evaluation of colour differences of surface colours to the CIELAB formula
No discolouration of white water	Filtrate darkening ΔY	INGEDE Methode 3: Optical evaluation of deinking filtrates

Figure 3: Parameters and test methods for the evaluation of deinkability.

The IE-calculation in Formula 1 has three assumptions:

- The measurements at 700 or 950 nm are influenced mainly by black ink particles only. At 700 nm also cyan ink particles are included and the Elrepho equipment is sufficient. For both wavelengths it has to be in mind, that other colours are excluded.
- The ink particle size distribution has big influence on the reflectance measurements in the near infra red region. The assumption is that the distribution is rather the same before and after flotation.
- The new IE-formula does not include the scattering coefficient S [7] anymore, because filter pads are used for the determination of the optical properties. It is assumed that the differences in S before and after the flotation process compete in significance against the losses of components in the handsheet formation process. Because errors

for both procedures are of the same order, the simplest way for the determination won.

If the effective residual ink content (ERIC) is determined, these values can be used instead of the brackets in Formula 1. This will be actualized as well in INGEDE Method No. 10 "Quantitative Evaluation of the Ink Elimination during Deinking" [8].

RESULTS

The deinkability test results of selected types of print products show the difference in significance of the deinkability parameters for the discussions between the members of the graphic paper chain. The comparison of results for the changing Ink Elimination (IE) calculation gives information about the tendencies of IE-values for different types of printed papers.

Deinkability Test Results in Comparison to the Orientation Values

In 2004 the existing data base on deinkability of print products was extended. The deinkability data base covers now the following number of tests:

- 29 newspapers
- 21 offset magazines, uncoated
- 32 offset magazines, coated
- 13 rotogravure magazines, uncoated
- 11 rotogravure magazines, coated
- 14 rotogravure catalogues, coated.

Additional test series were performed on offset newspapers in cooperation with two publishing houses and digital office printing (laser, ink jet). The results shown in Figures 4-8 include 27 offset newspapers. The first two figures illustrate the parameters of less importance for offset newspapers. No printed product crosses the orientation value/area lines in case of Dirt Area A and Colour Value a*. The remaining dirt area after flotation is the critical parameter for electrophotography: Measured values are in the range of 100 mm²/m² and 17 000 mm²/m².

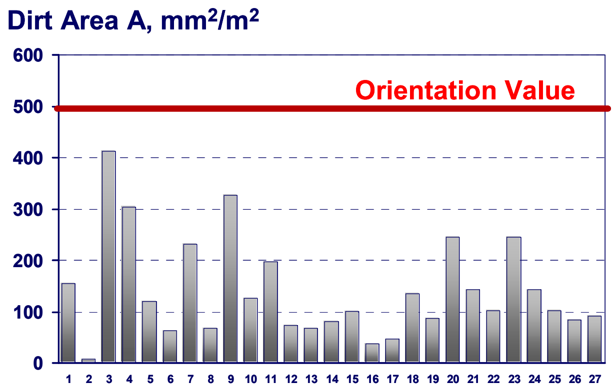

Figure 4: Deinkability test for printed products: Dirt Area after flotation, determined on handsheets with recirculated water.

The Filtrate Darkening is of significance as two offset newspapers do not fulfill the criterion. But it is still not the region of results for flexo newspapers. For waterborne ink systems luminosity, IE and filtrate darkening are the critical parameters. Despite the fact that improvements were obtained for waterborne flexo newspapers most orientation values are still not yet reached.

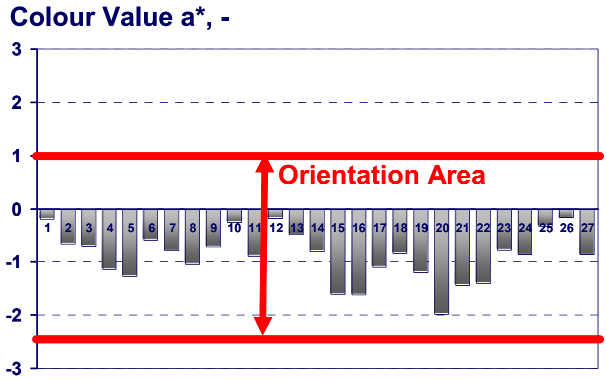

Figure 5: Deinkability test for printed products: Colour Value a* after flotation, determined on filter pads.

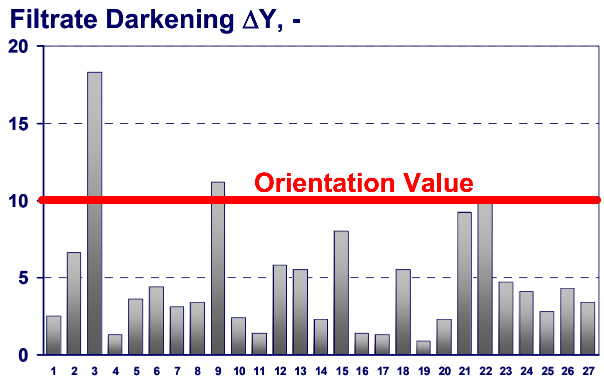

Figure 6: Deinkability test for printed products: Filtrate Darkening ΔY before flotation, determined on membrane filters after filtration of the filter pads' filtrates and tap water.

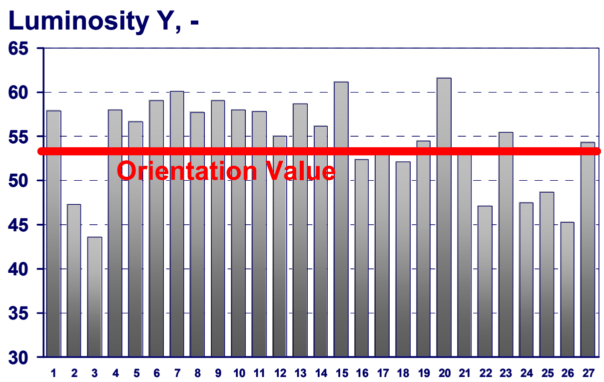

Figure 7: Deinkability test for printed products: Luminosity Y after flotation, determined on filter pads.

For offset newspapers the most critical parameter is luminosity (Figure 7). The orientation values are more difficult to be obtained for popular (full colour) newspapers than for conventional newspapers. Depending on the results it has to be discussed if separate orientation values for full colour newspapers should be established.

Most of the offset newspapers are above the requested value of 57 % Ink Elimination. It is interesting to compare Figures 8 and 6 and to find the same printed products failing the orientation values. This can be interpreted as a bad condition for the flotation process because of the ink particle size.

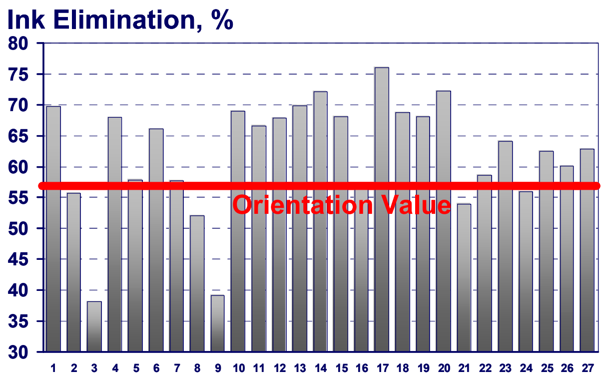

Figure 8: Deinkability test for printed products: Ink Elimination IE balancing the flotation process, calculated with K values determined on handsheets with recirculated water and K_0 = 0.7 m²/kg.

This paper concentrates on the results of the offset newspapers as an example. It may be added in few words that conventional (toluene) based rotogravure print products do not create large problems regarding deinkability. Sometimes insufficient ink removal and discolouration as well as filtrate darkening can be observed. Due to the unfortunate relation of ink to paper mass it becomes more critical for catalogues.

Comparison of Possibilities for the Calculation of the Deinkability Parameter Ink Elimination

The following results are related to lab scale trials with several printed products according to a modified version of INGEDE Method No. 11, using the Voith Delta 25™ laboratory flotation cell. The printed products tested and exemplified in Figure 9 were:

- Magazines M (rotogravure R, offset O)
  - Improved newsprint IN (MR-IN), SC (MR-SCB), (MO-SC)
  - LWC (MR-LWC), (MO-LWC)
  - HWC (MR-HWC), illustration printing paper (MO-HWC)
- News N (waterborne flexo F, offset O)
  - Virgin fibres VF (NF-VF), (NO-VF)
  - Deinked pulp DIP black ink B (NO-DIP-B),
  - coloured ink (NO-DIP-C).

The calculation of the Ink Elimination is simplified because of different aspects. The old IE calculation included an absorption coefficient, which should correspond to unprinted paper, but resulted in values above 100 % for some printed products, which do not fit to that absorption coefficient K_0. The numerical value K_0 = 0.7 m²/kg is the average of various wood-containing paper grades. In order to be able to use the same formula for all printed products, it was decided to neglect the value for the unprinted paper (Figure 4). For the printed products, which have unprinted paper of high ink content, this leads to lower IE-values.

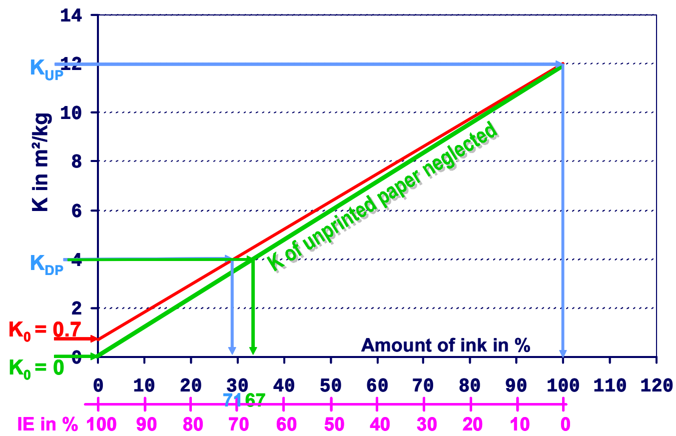

Figure 10: Effect of the different IE-calculation on results

The specimen preparation of handsheets with recirculated water was criticized because of effort and representativeness compared to the pulp. There has been no doubt about the specific light absorption coefficient K being the best parameter to observe the ink content [9, 10]. For the calculation of K, the opacity of the specimens should be according to TAPPI below 97.0 at 950 nm [11] or according to ISO even below 95 % [12].These thin specimens are required, because the scattering coefficient needs reflectance measurements of the single sheet (R_0) and of the stack of sheets (R_∞) and is a factor in the formula for K. The effects of neglecting the difference in S before and after flotation and of different specimen preparation are visible comparing the first two columns in Figure 9.

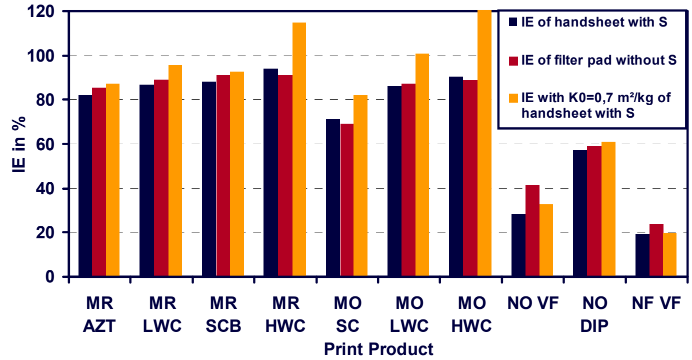

Figure 9: The deinkability parameter Ink Elimination IE following up the improvements of INGEDE Methods No. 10 and 11.

CONCLUSIONS

The INGEDE Method No. 11 persists in big parts. The defined parameters on deinkability enables to predict the deinkability in commercial scale correctly. Nearly the same parameters can be determined with less effort and possibilities for misunderstandings. The deinkability test method for printed products should be feasible for less well equipped laboratories as well. This is reached by simplifications and better descriptions. The reproducibility has been checked carefully within the project and is improved by analyzing sources for variations step by step, starting with the pulping device. A comparison of several flotation cells in laboratory scale helps to compare results, because the method is opened to other cells than only to the PTS cell.

For other types of printed products, e.g. office papers, orientation values have to be found.

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