Assessment of INGEDE Method in Different Laboratories and Protocol Modifications

Alvaro Buitrago*, Paul D. Fleming III*, John Cameron*, and Jan Pekarovic*

Keywords: INGEDE, deinking, recycled pulp

Abstract

Two samples of LWC heat-set offset printed substrates were tested according to INGEDE Method 11. The INGEDE Method was modified to provide continuity between the TAPPI testing standards and the INGEDE Method and enhance both the ease of testing and reliability of the test results. The main modifications include the procedure of test sheet preparation of pulp (TAPPI Standard T 272 applied at WMU), and the Ink Elimination values (ERIC measurements according TAPPI Standard T 567 applied at WMU). The results obtained at WMU were statistically analyzed and found to be consistent with the data obtained from INGEDE Laboratories and the data published in the literature. All individual optical properties considered in this experiment reached the criteria of deinkability.

Introduction

Deinked Pulp (DIP) has become a principal raw material source for many European and global papermaking operations. The European Recovered Paper Council (ERPC, 2007) reported that 60.1 million tonnes of paper were recycled in 2007. Much of which was deinked. Many newsprint and tissue grades commonly contain 100% deinked pulp and in many other grades, such as lightweight coated for offset printing and writing papers for office and home use. DIP makes up a substantial proportion of the furnish (Moore, 2005).

---

* Center for Recycling and Deinking, Paper Engineering, Chemical Engineering and Imaging, Western Michigan University, Kalamazoo

In addition, more or less half of the 340 million tons of paper and board produced worldwide are produced using recovered pulp. Most of them are used mainly in the production of brown papers, but in the last 15 years there has been increasing use of recovered fiber to produce, using deinking, white grades such newsprint, tissue, magazine papers, etc. (Carre, B. & Ayala, C., 2005)

INGEDE Method 11 (INGEDE, 2009) was developed to form a basis for comparing deinkability of prints. In March 2009, ERPC adopted the INGEDE scoring method as an assessment scheme (Fischer, 2010). This method simulates pulping and flotation at the laboratory scale. The revised version of INGEDE Method 11 to include pH adjustment has made it more suitable for a wider range of printed products.

Experimental Specifics

Two samples of LWC heat-set offset printed substrates were tested according to INGEDE Method 11 (INGEDE, 2009). The INGEDE Method was modified to provide connectivity between TAPPI testing standards and the INGEDE Method. This enabled us to enhance both the ease of testing and reliability of the test results. The main modifications include the procedure of test sheet preparation of pulp defined by TAPPI T 272 (1997), and the Ink Elimination values defined by ERIC (Effective Residual Ink Concentration) defined by TAPPI measurements according to TAPPI Standard T 567 (2009). The results obtained at Western Michigan University (WMU) were statistically analyzed and found to be consistent with the data obtained from the INGEDE Laboratory and other data published in the literature (Faul, 2008). All individual optical properties considered in this experiment reached the criteria of deinkability.

The evaluation of recyclability of paper and board products is very important for improving the recovered paper (Moore, 2005). The paper recyclability has two main components, the removal of printing inks (Moore, 2005) and the removal of adhesives (Abubakr, 2009a, b). Concerning the removal of the printing ink used, the term used is deinkability. This is just the simple ability of a printed product to be deinked. The definition is easy to give, but the hard part is how to accomplish and measure deinking. Guidelines and test methods, for evaluating of deinkability of finished products are necessary for developing recycling-friendly paper and board products. The research reported on here involves an investigation into the use of INGEDE method 11 (2009), to evaluate deinkability of printed products subsequent to flotation. The protocol consists of pulping followed by air flotation and handsheet production. A total of five optical characteristics are determined on deinked pulp, which contribute to the final test results. Three of those, luminosity, a* color value, and equivalent black area refer to the quality of deinked pulp, the other two, ink elimination and filtrate darkening, are process characteristics. Test results are converted to a score system. For a complete evaluation of the deinkability, the five individual scores are added. The experiments used two samples, LWC heat set (V1) and LWC heat set (V2) from the INGEDE institute (Faul, 2008) and were tested by the authors at WMU, in an attempt to adapt the INGEDE method to the equipment and materials available at the University.

Experimental

We summarize our research here. A detailed description of the research and all of the experimental results are given elsewhere (Buitrago, 2010).

For aging the print products, 200 g of oven-dry fiber (ODF) were placed in a warming cabinet at 60° C for 72 hours. All test samples were cut into pieces of about 2 cm square. A KitchenAid Professional 5 Plus was used to replace the Hobart N50 mixer recommended by INGEDE Method 11. The KitchenAid is the same capacity and is similar in footprint and mechanism to the Hobart. A Hobart N50 has three speeds and the KitchenAid has 10 speeds. Speed setting 3 on the KitchenAid is comparable to speed setting 2 on the Hobart. The KitchenAid was used during the pulping stage with consistency of 15%., 20 min of repulping and treated water at 45°C. The INGEDE standard formulation was used, i.e. 0.6% caustic soda, 1.8% sodium silicate, 0.7% hydrogen peroxide and 0.8% oleic acid. The sample was mixed with the deinking chemicals and de-ionized water, which was previously treated with calcium chloride dihydrate to obtain the desired hardness water, 128 mg Ca^{2+}/l. In the storage stage, the stock was diluted with treated water at 45°C to get a consistency of 5% and stored for 60 min. After that, the stock was diluted with warm water to obtain a consistency of 4% and a TAPPI disintegrator was used for one minute to disintegrate fiber bundles. Before the flotation stage, the consistency was adjusted with warm water at 45°C to reach a consistency of 0.8% in a Voith flotation cell. The flotation time was 12 min. Additional warm water was used to ensure overflow. The rotor speed was 1470 rpm, with an airflow of 6-7 l/min.

Ink elimination was calculated as ERIC (TAPPI, 2009), with R_∞ and R_0 values of handsheets of deinked pulp and undeinked pulp measurements in the infrared region of the spectrum, 945-955 nm. The ERIC value for unprinted paper was taken as zero. The spectrophotometer used was an Ocean Optics 2000. Verity IA software version 4.0.0 was used to evaluate the Equivalent Black Area. The optical properties of deinked pulp, luminosity Y, L*, a*, b*, were measured using the Technidyne (2008) Brightimeter Micro S-5, corresponding to a C 2° illuminant (CIE, 1971).

The filtrate darkening measurement followed the INGEDE Method 1 (2007). The key steps of the INGEDE method 11 (2009) were followed.

After the storage stage, a sample of stock was taken to make 6 handsheets in a sheet machine to evaluate reflectance with TAPPI T 567 (1997), 2 handsheets in a sheet machine to evaluate equivalent Black Area and 2 filter pads in a Buchner Funnel to evaluate filtrate darkening. The optical properties were evaluated using hand sheets, and the filter pads were used in the evaluation of optical properties (INGEDE, 2009).

Results

The numerical values for the components in the scoring for this study and published data (Faul, 2008) are summarized in Table 1. Figure 1 summarizes the deinkability scores of samples tested by both groups. All of the tested samples meet the criteria for deinkability. A deinking score of offset coated magazine was included to make a double data check. In the LWC Heat set (V2) sample, the ink elimination score contributed in less proportion to the total score compared with the other samples.

The IE value is reported here as IE_{ERIC} (Buitrago, 2010), while the IE value is reported for INGEDE laboratory as IE_{700} (Faul. 2008).

Table 1. INGEDE and WMU summary and comparison of optical measurements.
NR = not reported.

Print Product	Group	Statistic	Y	ΔY	A	a*	IE	Brightness	Yield
LWCV1	WMU	Mean	69.25	3.30	199.2	–0.813	56.38	66.23	70.8
		StDev	0.635	0.27	38.9	0.352	3.37	1.99	2.22
	INGEDE	Mean	69.7	1.94	NR	–1.4	54.2	57.6	71.2
		StDev	–NR	–NR	NR	–NR	–NR	–NR	–NR
LWCV2	WMU	Mean	68.77	2.41	493.2	–0.162	49.09	61.95	63.5
		StDev	1.22	0.57	40.30	0.099	4.17	4.89	2.38
	INGEDE	Mean	72.8	0.81	NR	–1.2	62.8	59.00	64.0
		StDev	–NR	–NR	–NR	–NR	–NA	–NA	–NA

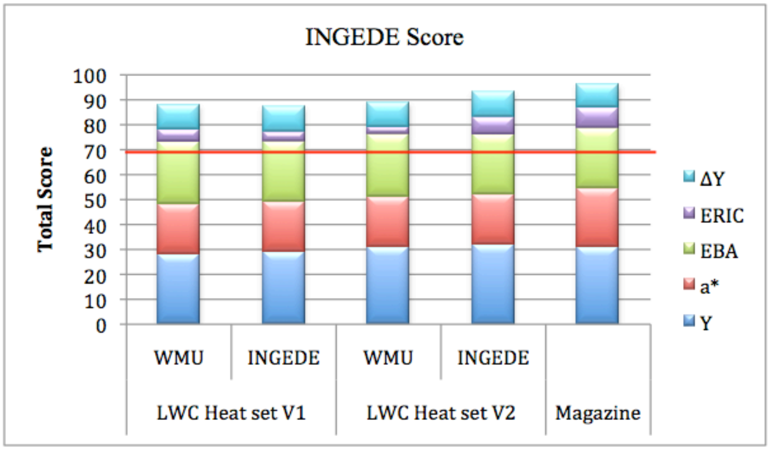

Figure 1. Comparison of WMU measured scores with published data (Faul, 2008).

In our study, sample LWC heat set (V2), showed lower values of the Ink elimination and higher values in the equivalent Black Area property. However, these individual differences were not evidenced in the property score contribution, except for the ink elimination. These indicate that the difference in the individual properties were not significant except for ink elimination.

Table 2 shows the Deinking score for the samples tested. The Deinking score was calculated using spreadsheet software provided by the INGEDE institute. The data were taken from an INGEDE project (Faul, A., 2008). The dirt particle area A of the samples included in INGEDE laboratory results was taken from this database. The INGEDE laboratory didn't include the corresponding value of this optical property for the samples tested.

Table 2. Deinkability scores from present study and published data (Faul, 2008).

PROPERTY	LWCV1		LWCV2		MAGAZINE INGEDE
	WMU	INGEDE	WMU	INGEDE	
Y	28	29	31	32	31
a*	20	20	20	20	23
A	25	24	25	24	24
IE	5	4	3	7	8
ΔY	10	10	10	10	9
Total score	88	87	89	93	95

The optical properties with a desired high value such luminosity and ink elimination show values higher than its threshold value and lower than its target value. This is illustrated in Figures 2 and 3. This behavior is similar in both WMU and INGEDE laboratory results.

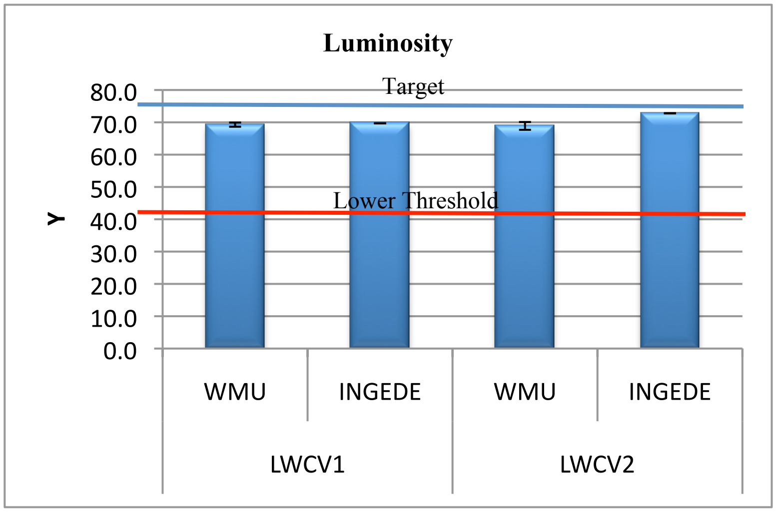

Figure 2. Luminosity values from the different laboratories.
The error bars represent standard deviation.

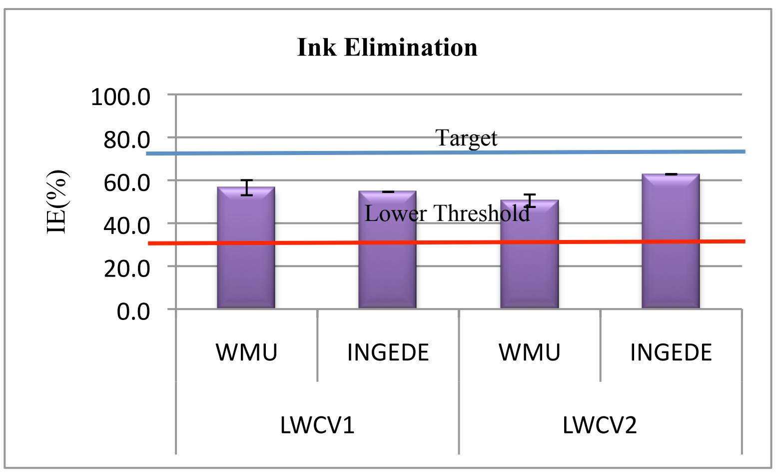

Figure 3. Ink elimination results from the different laboratories.
The error bars represent standard deviation.

On the other hand, optical properties with a desired low value such as filtrate darkening shows values lower than its upper threshold and target values. The maximum score is assigned. This is illustrated in figure 4.

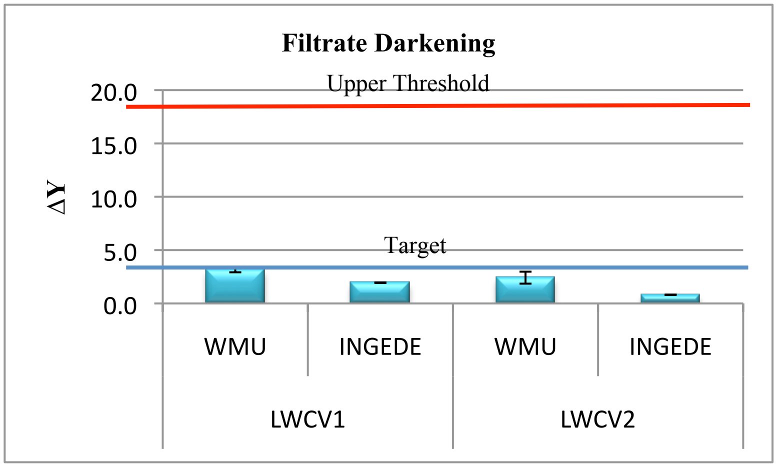

Figure 4. Filtrate darkening. The error bars represent standard deviation.

Figure 5 shows the color value a* data. The results are located in the target corridor and consequently take the maximum deinking score. This is true for both WMU and INGEDE laboratories.

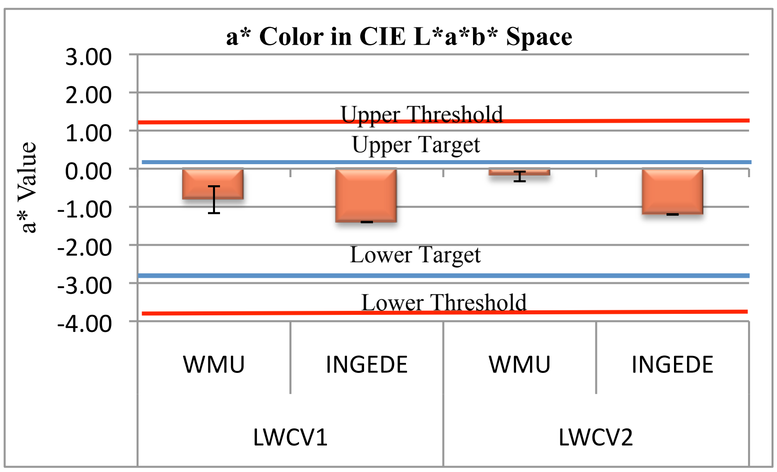

Figure 5. a color values from both laboratories.*
The error bars represent standard deviation.

The brightness and yield of flotation are illustrated in the Figures 6 and 7.

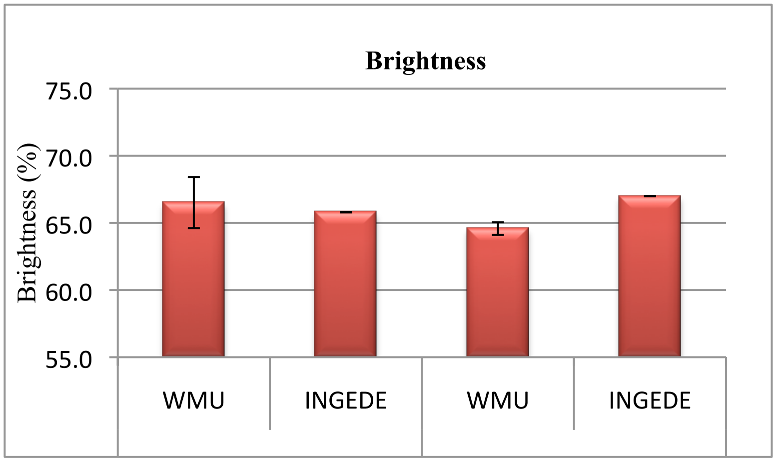

Figure 6. Brightness of handsheets from the two laboratories.
The error bars represent standard deviation.

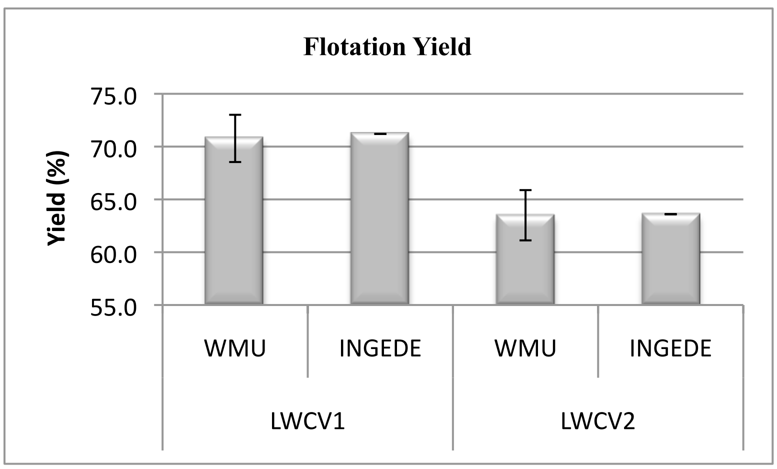

Figure 7. Yield of the flotation cell for the different groups.
The error bars represent standard deviation.

Figure 8 illustrates the total INGEDE scores of the samples analyzed and offset coated magazines. The Score of samples are located in the intermediate region of the Magazine Scores.

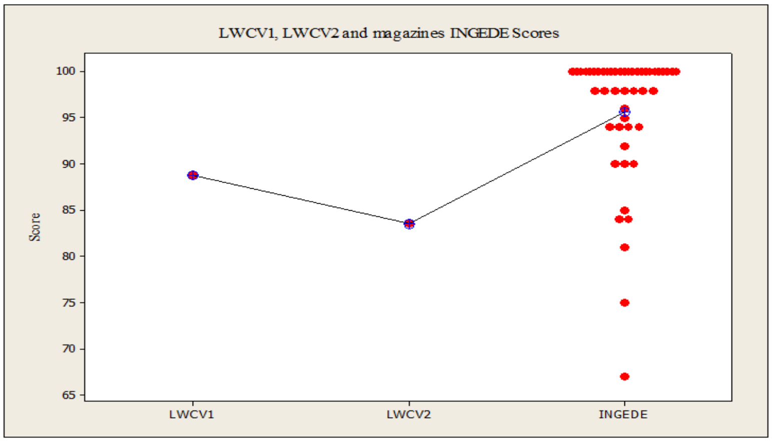

Figure 8. Total INGEDE Scores.

Finally, Figure 9 shows the individual INGEDE scores of the samples analyzed and offset coated magazines. The EBA score of the samples tested shows an important difference with the EBA score of the all magazines. This suggests that a more detailed study of this particular method must be considered in future research.

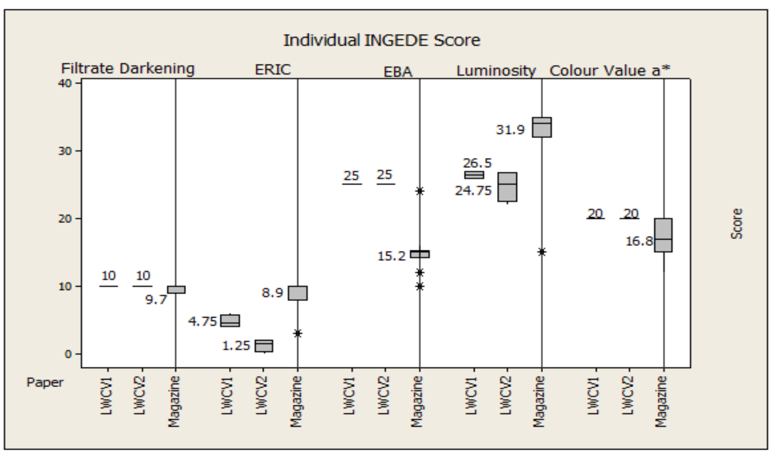

Figure 9. Individual INGEDE Score.

Conclusions

The INGEDE protocol was followed very closely. This research was focused in samples of heatset offset paper. The properties defined by the method were able to predict the deinkability on a laboratory scale. The WMU results were consistent with the data obtained from INGEDE, laboratory and data obtained from related literature. All individual optical properties considered in this experiment reached the criteria of deinkability.

The deinkability test methods should be capable of being done by other laboratories with different supplies. This requires harmonizing the test methods considered in the European protocol, as well as modifications to adapt them to a particular laboratory conditions. The methods and observations given here will be used in our laboratory for investigation of additional ink/paper combinations.

References

Abubakr, S.

1999a, with Houtman, C., Bormett and Sutherland, N. R., "Recycling Evaluation of New Generation Environmentally Benign Pressure Sensitive Adhesives," Proceedings of The Adhesive and Sealant Council, Inc. 1999 Spring Convention and EXPO April 11–14.

1999b, with Peng, J., "Innovative Approach to Solving "Stickies" Problem and Developing Environmentally Benign Pressure Sensitive Adhesives Through Partnerships," Proceedings of the 1999 TAPPI international environmental conference; APRIL 18-21; Nashville, Atlanta, GA: TAPPI Press, Vol. 2; 763–765.

Buitrago, A.

2010, "INGEDE Protocol," Masters Thesis, Western Michigan University, April.

Carre, B.

2002, with Magnin, L., "Digital Printing: a treat to the deinking industry?" *Paper presented at the 10th PTS-CTP Deinking Symposium*, Munich, Germany, April.

CIE

1971, "Colorimetry (Official Recommendations of the International Commission on Illumination)," CIE Pub. No. 15 (E-13.1), Bureau Central de la Paris, 1971.

ERPC

2007, Monitoring Report, downloaded from http://www.intergraf.eu/Content/ContentFolders/PressReleases/2008-09_ERPC_AnnualReport_2007.pdf, 3/3/2011.

Fischer, A.

2010, "Advances in Deinking and Deinkability of Inkjet Inks," Proceedings of IS&T NIP 26, Austin, September 19–23, p107.

Moore, G.

2005, "Where next for deinking?" Pulp and Paper International, July.

Faul, A.

2008, "European Deinkability Survey of Printed Products," Proceedings of PTS-CTP Deinking Symposium, Munchen, Germany.

INGEDE
2009, “INGEDE Method 11P, Assessment of Print Product Recyclebility – Deinkability Test,” Downloadable from http://www.ingede.com/ingindxe/methods/meth-e.html, accessed 2/2/11.
2007, “INGEDE Method 1, Test Sheet Preparation of Pulps and Filtrates fro Deinking Processes,” Downloadable from http://www.ingede.com/ingindxe/methods/meth-e.html, accessed 2/2/11.

TAPPI
1997. “Forming Handsheets for Reflectance Testing of Pulp (Sheet Machine Procedure),” T 272 sp-97.
2009, “Determination of Effective Residual Ink Concentration (ERIC),” T-567, Draft 2.

Technidyne
2008, “Brightmeter Micro S-5”, accessed from http://www.technidyne.com/Brightimeter-Micro-S5.html, accessed 3/4/2011.