Introduction

A red colouration of deinked pulp is detrimental to its use for the production of higher quality paper grades. Red colouration is therefore one aspect of the recyclability of printed products and thus for the sustainability of the graphic paper loop.

Red colouration of process water and pulp filtrates is caused by water soluble dyes, red pigments or parts of red pigments under certain conditions. Some pigments are only insufficiently alkali resistant and can be soluble under the deinking process conditions. Once in solution, a dyeing substance is not removable in the deinking process and cannot be separated by the circulation water treatment. It can even be attached again to the fibres during the sheet forming process unintentionally supported by retention aids. The sources of dyes and pigments are mainly printing inks and to a lesser extent additives for shading and mass dyeing.

One possibility to prevent red colouration from affecting the pulp and the circulation water is to control the raw material whether it has the potential to transport soluble colourants into the process. The method described was developed from a practical point of view, uses simple laboratory equipment and is one approach to inspect the incoming material. The visual assessment of a filtrate of a pulp sample slushed under alkaline conditions gives a good indication whether the product to be investigated shows a tendency for red discolouration.

1 Scope

This INGEDE method is used to analyse paper products on whether they show the tendency to cause any red colouration in deinked pulps. This test is primarily used as an entry inspection.

2 Principle

The samples are pulped under alkaline condition at defined pH. The samples are dewatered over a paper filter, then the filtrate is used to evaluate the red colouration visually.
3 Equipment and auxiliaries

3.1 Equipment

- Analytical balance with a scale up to 3000 g
- Graduated beaker (500 ml / 1000 ml / 2000 ml)
- Standard disintegrator or low consistency laboratory pulper
- Büchner funnel with vacuum equipment
- Clear glass beaker to collect and visually assess the filtrate. If a series of samples is evaluated, all beakers should have the same quality, diameter and volume.
- Cleaning equipment (scrubber, etc.)

3.2 Test material and chemicals

- pH measurement device or pH value stripes (range at least from pH 7.5 to pH 12) (e. g. Fa. Merck)
- Soda lye at a concentration of 5 % NaOH or 1,28 mol/l NaOH
- Filter paper 589/2 or 598 (Schleicher & Schüßl)
4 Procedure

4.1 Overview

- Print product
  - $m_{\text{oven\ dry}} = 80\ g$
  - $c = 4\ %$
  - $t = 5\ \text{min}$
  - $T = 45\ ^\circ\text{C}$

- Disintegration
  - $H_2O (45^\circ\text{C})$
  - 5 ml soda lye

- Büchner funnel
  - Filter paper no 589/2, 598
  - Filter pad of 2546 g/m²

- Filtration

- Filtrate

- Assessment

Figure 1: Flow chart of the test procedure

4.2 Sampling and sample preparation

80 g bone dry paper of one individual printed product without any impurities as clamps, plastic and etc. are torn into 4 cm x 4 cm pieces.

4.3 Disintegration

The sample is pulped at a concentration of 4 %, using tap water with a temperature of 45 °C and adding 5 ml soda lye. The consumption of 5 ml soda lye is an empirical value. After disintegration the pH must be higher than 9. If the pH is below 9, the pulping procedure has to be repeated with a new sample and a higher dosage of soda lye. It is useful to test a smaller sample amount first if there is any indication for an acidic paper. The standard pulping time is 5 minutes. If the sample is not free of fibre bundles, pulp for another 5 minutes.
4.4 Filtrate preparation

The disintegrated sample is filtered with a Büchner funnel. A filter pad is formed of theoretical 2546 g/m², derived from 80 g bone dry pulp and 200 mm diameter of the Büchner funnel. In order to ensure that the basis weight of the filter pad corresponds approximately to that number, follow Table 1 and adapt the pulp volume to the diameter of your filter and Büchner funnel.

The filtrate is poured into a beaker.

Table 1: Different bottom bore diameter and the required pulp volume

<table>
<thead>
<tr>
<th>Bottom diameter (Büchner funnel) [mm]</th>
<th>Pulp volume (consistency 4 %) [g]</th>
<th>Bone dry pulp [g]</th>
</tr>
</thead>
<tbody>
<tr>
<td>110</td>
<td>605</td>
<td>24</td>
</tr>
<tr>
<td>125</td>
<td>780</td>
<td>31</td>
</tr>
<tr>
<td>126</td>
<td>795</td>
<td>32</td>
</tr>
<tr>
<td>130</td>
<td>845</td>
<td>34</td>
</tr>
<tr>
<td>132</td>
<td>870</td>
<td>35</td>
</tr>
<tr>
<td>150</td>
<td>1125</td>
<td>45</td>
</tr>
<tr>
<td>160</td>
<td>1280</td>
<td>51</td>
</tr>
<tr>
<td>185</td>
<td>1710</td>
<td>68</td>
</tr>
<tr>
<td>200</td>
<td>2000</td>
<td>80</td>
</tr>
</tbody>
</table>
4.5 Filtrate evaluation

The filtrate colour is visually assessed. The examples in Figure 2 should give a support to categorise the colouration into six classes. The certitude to estimate the degree of colouration might become better after several tests. Helpful would be also a comparison between two different samples filtrates or a series of tests.

<table>
<thead>
<tr>
<th>Colouration</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Photo</td>
<td><img src="image1" alt="Photo" /></td>
<td><img src="image2" alt="Photo" /></td>
<td><img src="image3" alt="Photo" /></td>
<td><img src="image4" alt="Photo" /></td>
<td><img src="image5" alt="Photo" /></td>
<td><img src="image6" alt="Photo" /></td>
</tr>
<tr>
<td>Description</td>
<td>Milky white (cloudy due to fillers)</td>
<td>Milky orange</td>
<td>Clear red</td>
<td>Milky red</td>
<td>Intense red</td>
<td>Very intense red</td>
</tr>
</tbody>
</table>

**Figure 2: Description of the classes**

5 Report

The report should contain:

Printed product
- Title
- Publishing date
- Printing location (if possible)
- Printing method (e.g. flexo, offset, rotogravure, ink jet, etc.)

Method
- Disintegration time
- Volume of soda lye
- pH after pulping
- Result of the evaluation.

Any changes of the method have to be reported.
6 References

6.1 Cited standards and methods
- ISO 5263-1 (2004): Pulps – Laboratory wet disintegration

6.2 Literature and other related documents
- Müller-Mederer, C., Putz, H.-J.; Basic Research on Red Discolouration of Deinked Pulp and Effluent INGEDE Project 59 97; January 2000 (in German)
- Müller-Mederer, C., Putz, H.-J., Göttsching; L.; Possibilities to Reduce Red Discolouration of Deinked Pulp; Wochenblatt für Papierfabrikation Nr. 10, 2002
- Ivana Plazonic; Investigations on red discolouration in deinked pulps and filtrates; COST-STSM-E48-04408; March 2009

6.3 Sources
This INGEDE method was developed by Utzenstorf Papier mill as part of the entry inspection of recovered paper.

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