A New Burnout Test for Wood Containing Papers

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1. Introduction

The Burnout- Test was developed in 1967 by W. Irby [1]. It is an easy and fast method to visualize the coating layer evenness on coated paper by charring the paper fibers without affecting the coating layer. For the test the paper specimen is submerged into a solution composed of ammonium chloride, ethanol and water and then air-dried for several hours. Then the specimen is charred at a temperature of about 200°C for several minutes. As a result the fibers are blackened without any discoloration of the coating layer. The coating layer unevenness can be directly observed due to the good contrast between white coating on the blackened fibers.
Later R. Dobson examined the mechanisms of the test [2]. He showed that carbonization is responsible for fiber blackening. Carbonization depends on the concentration of the charring catalyst as well as the charring temperature. O´Neil M. and Jordan B. optimized the parameters of the test [3]. They also showed, that the Burnout- Test can be used only for wood-free coated paper which is a major restriction. Jordan and O´Neil state that inadequate blackening of wood fibers is caused by insufficient charring of the lignin. Applying the burnout test to a TMP handsheet, figure 1a, demonstrates that the fibers are only partially blackened. One can additionally observe light grey regions which probably descend from insufficiently blackened fines.
C. Voura modified the Burnout- Test [4] to analyze filler distribution of uncoated wood containing papers (SC-A papers). The modified test employs a different impregnating agent, ammonium chloride was substituted by a commercial flame inhibitor, *Eucam Pappe/Papier*[1]. This procedure enables blackening wood fibers.
Founding on the work of Voura [4] we have developed a Burnout-Test for wood containing paper. In order to ensure constant chemical composition of the charring catalyst, which can not be ensured from a commercial flame retardant, we have developed a new testing procedure based on charring agents commonly employed in flame retardants.

2. Flame Retardants

Charring agents like *Eucam Pappe/ Papier*[1] are based on nitrogenous and phosphoric compounds like di-ammonium phosphate $(NH_4)_2HPO_4$, mono-ammonium phosphate $(NH_4)H_2PO_4$, ammonium chloride $(NH_4)Cl$, and phosphoric acid. At high temperature inorganic salts release acids which attack predominantly cellulose and hemicellulose [5]. Thermogravimetric analysis had furthermore shown a significant reduction of the charring temperature due to those inorganic salts [6]. An increasing amount of carbon at the fiber surface further enhances the effect of carbonation [7]. Secondary inorganic salts are used for chemical activation of the carbonates by increasing the internal surface [8]. During carbonization an isolating carbon layer is produced. This layer on the fibers protects the surface against high temperature and seals it from oxygen. In this way, inflammation is blocked.

3. Testing Procedure

3.1. Preparation of the Charring Agent

The impregnating agent includes inorganic acids to accomplish fiber charring and sorbit (sugar alcohol) which helps build the isolating carbon layer. The preparation of these components is described in the following:

1) Preparation of a 0.5 molar $(NH_4)H_2PO_4$ solution.
2) Preparation of a 0.5 molar $(NH_4)_2HPO_4$ solution.
3) Mix solution 1) and 2) to equal volumes.

[1] www.endotherm-brandschutz.com

4) Add $(NH_4)Cl$ in the amount of 0.53 g/100ml.
5) Add borax in the amount of 1g/100ml and sorbit in the amount of 0.5g/100ml.
6) Adjust pH- value to 6.5 by adding (NH_4) H_2PO_4 solution.
7) Stir for about 15 minutes before using as an impregnating agent.

Before using, all components have to be dissolved completely. Keeping the impregnation agent without repeated stirring can lead to phase separation which makes it useless.

3.2. Testing Procedure

The paper is conditioned for 24 hours in standard conditions (25°C and 50% humidity). Specimen with size 6 x 6 cm² are cut from the paper. The testing procedure is carried out in three steps: impregnation, pre-drying and carbonization.

1) Impregnation: The specimen is fully covered by the impregnation agent. It is impregnated under vacuum for 30 minutes. In time intervals of 10 minutes the sample is turned over.
2) Pre-Drying: After Impregnation, the sample is pressed lightly between blotting paper to remove the liquid from the surface. After that the sample is dried for 30 minutes at a temperature of 110°C.
3) Carbonization: After Pre-Drying the paper sample is clamped to hang freely in the oven. For carbonization a temperature of 225°C and a charring time of 30 min are required.

4. Evaluation of the new Burnout Test

4.1. Evaluating Methods

Paper blackening evaluates the blackening due to the Burnout- Test and thus measures charring intensity. The charred sample is imaged by a flat bed scanner at 1200 dpi. With each sample, reference papers for absolute white (0%) and absolute black (100%) are scanned. The blackening level is given as a relative grey value between 0% (white reference) and 100% (black reference).
Structure analysis. The size distribution of the coating layer structures visible after the Burnout Test is measured using a 2D Fast Fourier Transform (FFT) of the scanned images of the paper in combination with band pass filtering. This measurement gives the intensity of a structure (variance) over the size of the structure (wavelength λ), compare figures 2b and 2c.

4.2. Results

In this section results with the new burnout- method will be shown and compared with the standard burnout test [3]. All tests were performed with a commercial 62g/m² LWC paper containing 40% groundwood, 50% DIP and 10% bleached softwood kraft pulp.

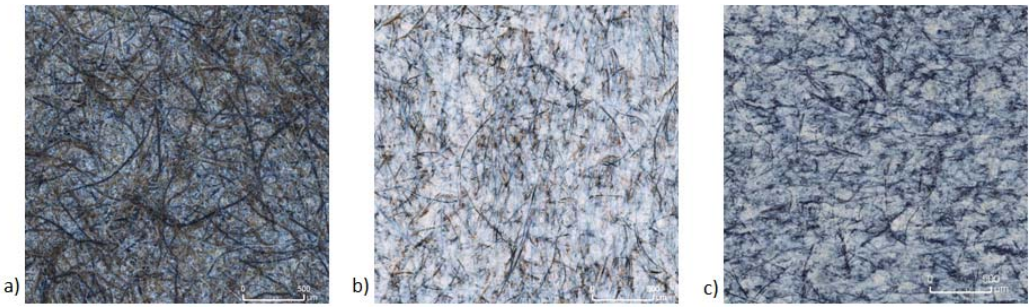

Figure 1. a) The standard Burnout- Test [3] applied to a TMP handsheet. The fibers are not sufficiently blackened. b) Standard Burnout test [3], wood fibers carbonized incompletely. c) Result of the new Burnout Test, all fibers are entirely and evenly blackened.

Figure 1b show an incomplete carbonization of a wood containing LWC paper after the standard burnout test [3]. Figure 1c presents the result of the new testing method where fibers are entirely blackened. The grey regions are blackened fibers under the white coating layer. Variations of coating coverage are clearly observable, there are no distractive brightness variations of the base paper. The test has also been performed on pure TMP handsheets, confirming that full blackening of all fibers is achieved.

4.3. Evaluation

First optimal charring time is evaluated. Therefore carbonized samples with increased charring time are analysed. In figure 2a, results of brightness analysis are shown. At a charring time of 20 minutes the fibers are entirely blackened, up to 90 minutes blackening is not further increased. If the charring time exceeds 90 minutes, the coating layer is starting to be discoloured and the blackening level further increases. Small confidence intervals indicate a good repeatability of the charring behaviour.
The main purpose of the Burnout Test is visualisation of the coating layer unevenness. Thus it is very important, that the testing procedure reveals only the coating structure. Figure 2b shows the structure size distribution of the same paper with different charring time, the FFT here has been normalized for equal contrast. Figure 2b shows, that the new Burnout Test captures the structure in a very stable way, charring time does not affect the structure visible after carbonisation of the base paper and no new structures are introduced by increased charring.
Finally repeatability of the test has been examined by conducting 5 consecutive tests with samples from the same LWC paper. For each test a new impregnation solution was prepared and the specimen were impregnated, pre-dried and charred. Then the samples were scanned at 1200dpi and the structure size distribution of the coating layer structure was measured, figure 2c. The structure size distribution of coating structure is nearly equivalent for all samples, thus the Burnout Test provides high repeatability.

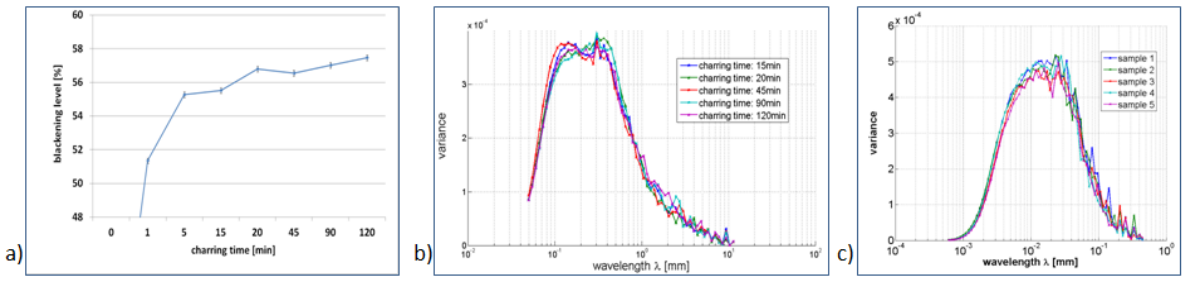

Figure 2. a) Result of blackening analysis, the selected charring time is 30 minutes, charring stabilizes afterwards. b) Structure size distribution of samples with different charring time, the difference between the samples is minimal. c) The structure size distribution of the analysis show a high repeatability, the curves are very similar.

5. Conclusions

The development of a new burnout test enables the blackening of wood containing base papers without discolouration of the coating layer. Thus it is an easy and fast method to visualize coating layer coverage and coating layer unevenness of wood containing papers. The test results are highly repeatable and the structure of the coating layer is captured in a very stable way.

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