Serial Sectioning of Coated Paper as a Novel Method to analyze Binder Penetration

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ABSTRACT
Information regarding the distribution of the coating binder and the penetration of the binder into the base paper is important to understand mechanical and printing properties like e.g. picking resistance. This work focuses on monitoring binder penetration using serial sectioning combined with fluorescence microscopy to create cross-section images for 3D coating analysis. Coating trials with three different coating binders varying in their AA-GWR value (Abo Akademi Gravimetric Water Retention Value) were used to analyze binder penetration depth into the base paper.

INTRODUCTION
X-ray Microtomography, Confocal Laser Scanning Microscopy ([1], [2]), Scanning Electron Microscopy [3], Atomic Force Microscopy ([4], [5]) and - especially for binder migration analysis - Confocal Raman Microscopy ([6], [7], [8]) have been used so far in the 3D-analysis of coating layers on paper. A combination of serial sectioning and light microscopy [9] is a further method to analyze three-dimensional features of coating layers. The µSTRUCSCOP device (Figure 1) is a second generation prototype equipped with a fluorescent and a visual light source [10].
It performs fully automatic serial sectioning and microscopy of coated and uncoated paper samples. Sample sizes of 2x6mm at resolution down to (0.47 x 0.47 x 1) µm^3 can be analyzed. Online stitching and aligning (2D) is performed automatically.

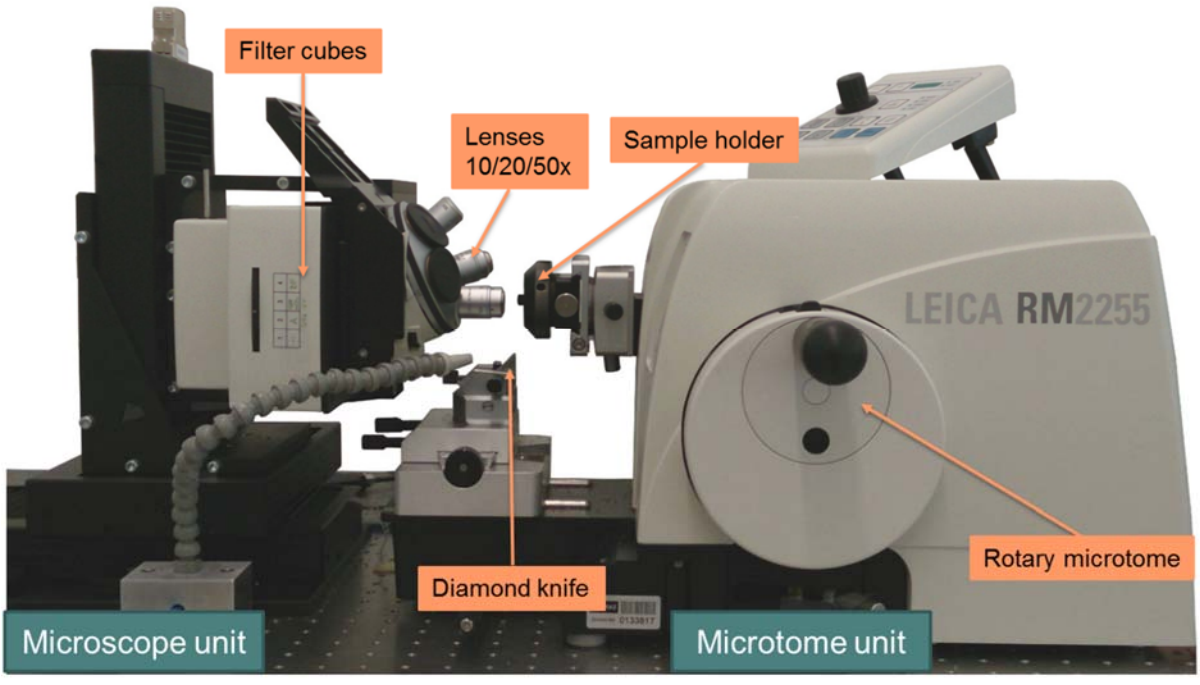

Figure 1: µSTRUCSOP

Via fluorescence microscopy the binder penetration is determined. A molecule gets excited into a higher state of energy and on the way back to the ground state it emits light (= fluorescent light) [11]. Fluorescence microscopy leads to colored images and simplifies the segmentation (transformation from a RGB to a binary image).
The first step is the establishment of a method to stain the binder. Different types of fluorescence (primary fluorescence, secondary fluorescence) are available to label the binder [12]. The next steps include the checking of compatibility with the resin for embedding and the “proof of concept” by analyzing the penetration depth into the base paper of three different binders.

MATERIALS AND METHODS

Coating Color Preparation and Coating Process

Ground calcium carbonate (90% < 2µm) was mixed with binder and distilled water. Three different binders (see Table 1) were used varying in their AA-GWR value (Abo Akademi Gravimetric Water Retention Value). The target solid content of the coating color was 60%. The pH was adjusted to 9. The coating process was performed with a manual rod coater. As base paper, laboratory handsheets with a grammage of 80g/m^2 made out of short fiber sulfate pulp with a low Kappa number (free of fluorescence) were used.

Table 1: The three different binders and their ABO WRV

Type	AA – GWR value [g/m^2]
Binder A	95
Binder B	400
Binder C	970

Sample Preparation

Paper samples are cut into pieces of 8x15mm and infiltrated with TechnoVit 7100 (Sanova). This solution has been stained with Sudan Black (Roth) (0.3 mass%) and stirred for 45 minutes. Samples are infiltrated in a plastic vacuum exsiccator, and a vacuum pump is switched on and off for 4 hours in intervals of 15 minutes. Infiltrated paper samples are embedded in gelatin capsules with stained TechnoVit 7100. Hardening time was 12 hours. Samples were then polished.

Microtomy, Image Creation and Analysis

Embedded samples were precut with a glass knife and then cut with a 4mm diamond knife (Diatome). The slice thickness was set to 4µm and 100 cuts were performed. For fluorescence microscopy a GFP-filter was used. Images with a dimension of 0.5x5.4mm were taken and stitched and aligned automatically. Images were transformed to binary images using ImageJ and penetration depth into the base paper was calculated (adapted method described in [9]).

RESULTS AND DISCUSSION

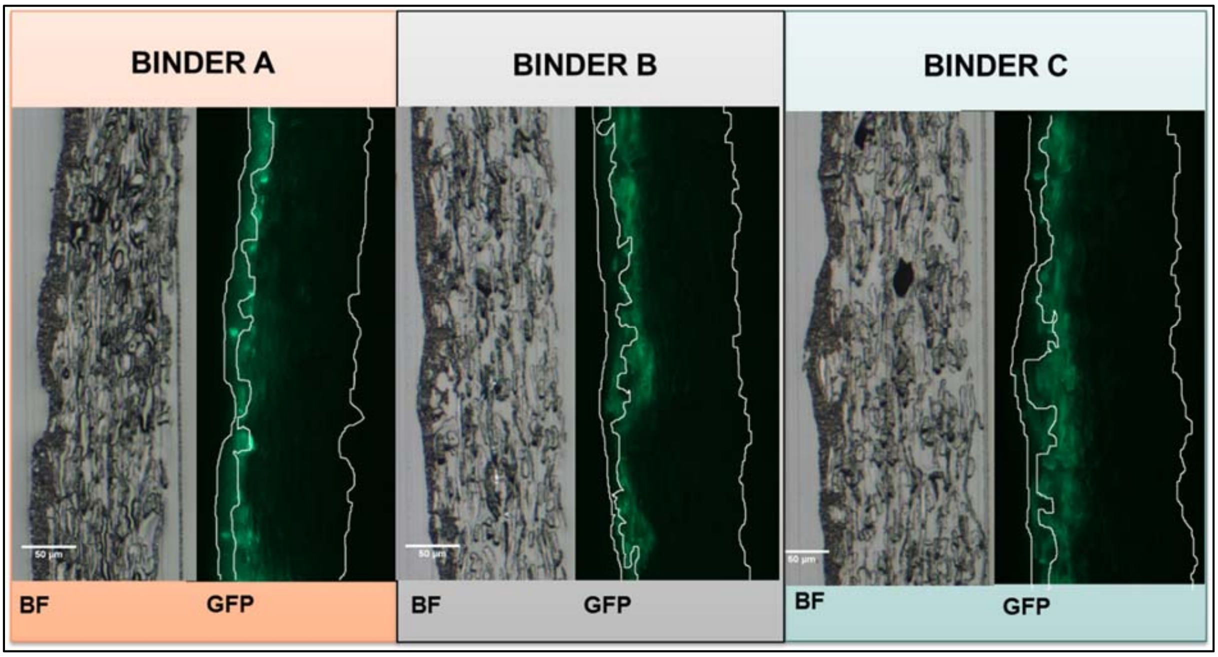

Figure 2: Cross-sections of coated papers (VIS – standard light microscope image, GFP – UV light microscope image) using binders with different AA-GWR

Figure 2 shows the visual light image (BF) and the UV light image (GFP) of each binder. The coating layer can be seen on the left side of the BF image. To visualize the binder penetration depth into the base paper the contour of the BF image was superposed with the GFP image (white line).

Figure 2 shows that Binder A has the lowest and Binder C has the highest penetration depth into the base paper. Due to the fact that a lower water retention value leads to less binder penetrating into the base paper the water retention value is consistent with the microtome images analyzed. The coating layer with Binder C is not fluorescent anymore because all of the binder penetrated into the base paper. This is an extreme case of binder penetration and this kind of binder cannot be used in conventional coating application.

Table 2: 3D data analysis of binder penetration depth

Sample	Penetration depth into the base paper[µm]
Binder A	16.9
Binder B	21.0
Binder C	31.1

In Table 2 the results of the 3D data analysis of an image stack are presented. Binder A shows a penetration value of 16.9µm compared to Binder C with 31.1µm. The binder follows the water which cannot be retained in the coating color into the porous system of the base paper. The lower the retention of water of the coating color is the deeper the binder penetrates into the base paper (see Binder C in Table 2).
Figure 3 shows the distribution curves of binder penetration into the base paper. Binder A and B show a narrow distribution and in comparison Binder C has a broad distribution.

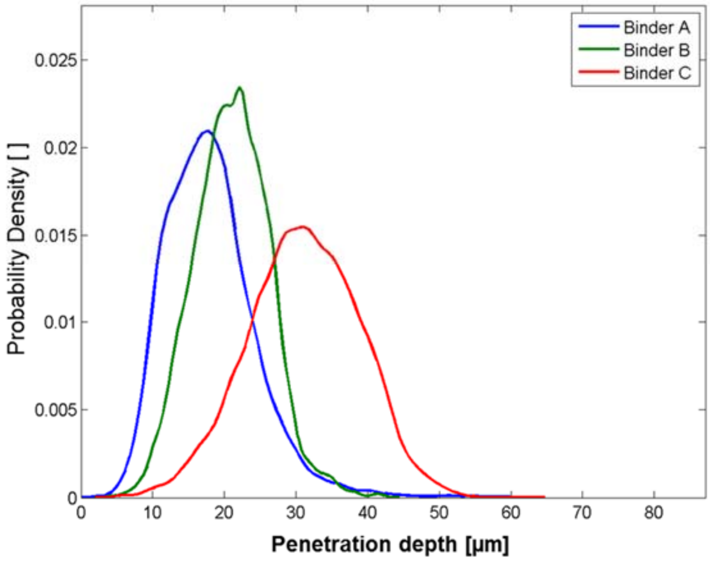

Figure 3: Distribution curves of binder penetration into the base paper

CONCLUSION AND OUTLOOK

In conclusion serial sectioning of fluorescent coating layers is an appropriate method to get information about the binder penetration into the base paper. Cross-section images and 3D data analysis of an image stack can be correlated with water retention values of the coating colors. An important step regarding the establishment of this new binder penetration analysis method is the development of the staining process of each binder. So far just natural binders were analyzed. In the future staining the most used conventional binder latex will be an important issue. Influences of coating application and drying method need to be investigated too.

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