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#### SIZING EVALUATION OF UNCOATED FINE PAPERS

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#### **SUMMARY**

This paper is intended to give some contribution to sizing measurements techniques and their role in modern paper sizing systems.

**Keywords:** Bristow Absorption Test (B.A.T.), Cobb, Contact angle, PDA, Sizing, UWF.

#### INTRODUCTION

Sizing of UWF papers, produced in neutral alkaline medium, has been traditionally performed using AKD as internal sizing agent. The AKD has some disadvantages such as cost, non-complete cure at the reel and loss of paper machine efficiency, due to sticky by-products resulting from the reaction of hydrolysis.

For these reasons gPS (grupo PortucelSoporcel) studied a different approach in what concerns sizing strategy of its papers:

- i) Using ASA in the wet end.
- ii) Complementing the sizing with a surface sizing agent (SSA).

For the previous sizing conditions the Cobb 60 test method proved to be a very good method to evaluate and control the sizing degree, due to its low variability.

However, in what concerns new sizing conditions, and namely regarding the adequacy of the method to measure and control the dosages of the sizing agents, new challenges arose. In fact, by maintaining the traditional Cobb 60 method to control the final paper sizing at the same level, different final functional properties, mainly related to the printing quality characteristics, were obtained. Therefore, a comparative study of the various possible approaches available to directly or indirectly evaluate internal and surface sizing was carried out in order to define the best method to be used in routine tests and the most appropriate one to decouple the effects of internal and surface sizing agent dosages.

#### **METHODOLOGY**

The study was conducted in three stages:

- i) Five distinct mill samples with different internal sizing dosages, with and without surface sizing, were tested;
- ii) Repeatability tests were performed on a selected paper sample;
- iii) An industrial test was performed, where internal and surface sizing agent dosages were changed.

Different sizing evaluation methods were used, as detailed in Table 1:

Table 1 – Brief description of the laboratory methods and parameters used in this paper.

| Abbreviation | Test  | Standard        | Definition  |
|--------------|---|-----------------|---|
| Cobb60       | Cobb 60   | ISO 535:1991(E) | Determines the water absorptiveness of sized paper, with 45 seconds contact with water.   |
| Cobb30       | Cobb 30   | ISO 535:1991(E) | Determines the water absorptiveness of sized paper, with 20 seconds contact with water.   |
| HST          | Hercules Sizing Test  | T 530 om-07     | Determines the aqueous resistance of paper, with a final reflectance value of 85%.  |
| StCA         | Static Contact Angle  | T 458 cm-04     | Determines the resistance of paper surface to wetting by water.   |
| SurfEner     | Surface Energy  | -               | Determined by the contact angle using water and methylene iodide, through the OWRK method. [1]  |
| % Polar      | Percentage of the polar<br>component in the<br>Surface Energy | -               | Determined by the contact angle using water and methylene iodide, through the OWRK method. [1]  |
| WetVel       | Speed of Water<br>Absorption                                  | -               | Determined by the contact angle with water, between 3 and 5 s.  |
| TapVel       | Speed of Water<br>Absorption                                  | T 458 cm-04     | Determined by the contact angle with water, between 5 and $60 \text{ s}$ .  |
| MaxVel       | Maximum Speed of Water Absorption                             | -               | Determined by the contact angle with water, determined by the highest slope of the adsorption curve.  |
| FinVel       | Final Speed of Water<br>Absorption                            | -               | Determined by the contact angle with water, determined by the slope of the adsorption curve at the end.   |
| Vel10        | Speed of Water<br>Absorption                                  | -               | Determined by the contact angle with water, between 0 and 10 s.   |
| KA           | Absorption Coefficient  | -               | Determined by the Bristow Absorption Test (B.A.T.)  |
| PDA          | Penetration Dynamic<br>Analyser                               | -               | Several parameters are determined based on ultrasonic measurements.   |
| BendPer      | Bendtsen Air<br>Permeability                                  | ISO 5636-3:1992 | Determine the rate of flow of air through a unit area of a sheet of paper, under a unit pressure difference, using the Bendtsen apparatus.  |
| BendRoug     | Bendtsen Roughness  | ISO 8791-2:1990 | Determines the rate at which air will pass between a flat circular land and a sheet of paper, under a fixed operating pressure, using the Bendtsen apparatus.   |
| SFC          | Static Coefficient of Friction                                | ISO 15359:1999  | Determines the static coefficient of friction, using the horizontal plane method.   |
| DFC          | Kinetic Coefficient of Friction                               | ISO 15359:1999  | Determines the kinetic coefficient of friction, using the horizontal plane method.  |
| TA           | Toner Adhesion  | EN 12283:1996   | Determines the toner adhesion through the IGT test.   |
| AG           | Area Gamut  | -               | Determines the area of the irregular hexagon created by plotting a*, b* CIE colour coordinates for 6 colours: Cyan, Magenta, Yellow, Red, Green, and Blue.  |
| OD           | Optical Density   | -               | Is a measure of the colour intensity (for the printing optical density, the paper density is discarded)   |
| PT           | Print Through   | -               | Determines the ink penetration in the ZD direction of the paper, through optical measurements, by the difference between a non printed paper and the reverse of a printed area.                             |
| СВ           | Colour Bleed  | -               | Determines the ink interpenetration in contiguous areas, through image analysis techniques. It is based on the width of a black line on a yellow background or the width of a yellow on a black background. |

#### Stage 1

This stage was performed aiming to make a first screen of the sizing tests, using the coefficient of variation (CV) as a screening factor. The repeatability of the method was calculated using Equation 1:

$$Sr = \sqrt{\frac{\sum_{w=1}^{p} \left[ (n_w - 1) \times S_w^2 \right]}{\sum_{w=1}^{p} (n_w - 1)}}$$
 (1)

Where  $S_W^2$  stands for the standard deviation of each sample,  $n_w$  for the number of tests on each sample and p for the number of samples.

Five off-machine samples of gPS, 80 g/m<sup>2</sup> office papers (Table 2), were chosen to perform 30 sizing tests on each one and to calculate the coefficient of variation (Figure 1).

Table 2 – Samples characterization.

| Tubic =    | 10 111-1-111               |  |  |
|------------|----------------------------|--|--|
| Eucalyptus | Long Fibre                 | ASA <sup>(a)</sup>                             | SSA  |
| (%)        | (%)                        |  |  |
| 90         | 10                         | 100  | No   |
| 90         | 10                         | 144  | No   |
| 90         | 10                         | 213  | No   |
| 90         | 10                         | 100  | Yes  |
| 90         | 10                         | 120  | Yes  |
|            | Eucalyptus (%) 90 90 90 90 | Eucalyptus (%) (%)  90 10  90 10  90 10  90 10 | Eucalyptus         Long Fibre         ASA <sup>(a)</sup> (%)         (%)         (%)           90         10         100           90         10         144           90         10         213           90         10         100 |

(a) - 100% corresponds to the normal ASA dosage.

The air permeability and roughness were also measured, to further evaluate the dependence of sizing with these two paper characteristics.

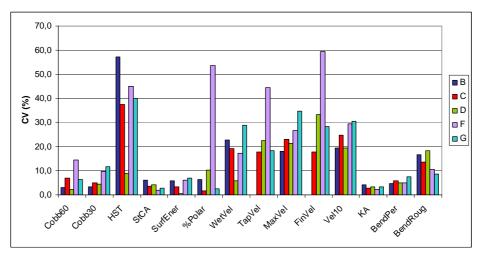


Figure 1 – Coefficient of variation for the different parameters that were measured.

The repeatability of the tests, using Equation (1) and the results from the 5 samples that were analysed, was then calculated. The results are plotted in Table 3.

Table 3 – Estimated repeatability of the sizing and structural tests.

| Test                      | Repeatability of the Test |
|---------------------------|---------------------------|
|                           | (%)                       |
| B.A.T.                    | 2,9                       |
| Contact angle             | 3,5                       |
| Surface energy            | 4,3                       |
| Bendtsen Air Permeability | 5,4                       |
| COBB 60                   | 6,4                       |
| COBB 30                   | 6,6                       |
| Bendtsen Roughness        | 13                        |
| % of polar component      | 15                        |
| Adsorption speed at 10 s  | 24                        |
| Maximum adsorption speed  | 25                        |
| Tappi adsorption speed    | 26                        |
| HST                       | 37                        |

Based on these results it was decided to continue the study by using the COBB 60 and the contact angle tests. At this stage of the work it was also decided to introduce another dynamic adsorption test, the PDA. The results of this test are specified in terms of several distinct parameters: W, which relates to both surface roughness and hidrophobicity, and gives the initial resistance to surface complete wetting; Max, which gives the time (s) for the surface to be completed wetted; A30 and A60, corresponding to the amount of adsorbed water at 30 and 60 s, respectively (similar to Cobb concept); and Imin, the intensity of the ultrasonic waves at 60 seconds, which is an indication of the level of residual sizing after 60 s.

#### Stage 2

Next, it was decided to use boxes of office paper, chosen randomly from different finishing lines, with different grades of 80 g/m2, produced on 3 different paper machines of the group. The corresponding results of the Cobb and of the contact angle measurements are listed in Table 4.

Table 4 – Stage two test results of the Cobb 60 and the contact angle measurements.

| 1       |                       | PMA  |                       | PMB  | PMC                   |       |  |
|---------|-----------------------|------|-----------------------|------|-----------------------|-------|--|
|         | Cobb 60 Contact angle |      | Cobb 60 Contact angle |      | Cobb 60 Contact angle |       |  |
| Average | 33.4                  | 76.3 | 29.3                  | 93.2 | 28.1                  | 101.7 |  |
| CV (%)  | 2.1                   | 2.6  | 3.3                   | 2.1  | 7.4                   | 2.6   |  |

Relevant differences among the samples were found, representing the industrial MD and CD process variations. In order to overcome these differences and, at the same time, to study these variations, it was decided to perform an industrial test.

#### Stage 3 - Industrial test

The industrial test was carried out in one PM of the group, under the following surface and internal sizing agent dosages indicated in Table 5:

Table 5 – Industrial test conditions.

| I unic c | maasti ai test conditions. |                    |  |  |
|----------|----------------------------|--------------------|--|--|
| Samples  | ASA <sup>(a)</sup>         | SSA <sup>(a)</sup> |  |  |
| Samples  | (%)                        | (%)                |  |  |
| 1        | 109                        | 100                |  |  |
| 2.1      | 109                        | 148                |  |  |
| 2.2      | 109                        | 186                |  |  |
| 3        | 129                        | 100                |  |  |
| 4        | 157                        | 100                |  |  |
| 5        | 157                        | 0                  |  |  |

(a) - The normal dosage of the respective sizing agent is assumed to be 100%

The reams were produced from the same paper machine CD position in order to take out the CD profile influence. These reams were then studied taking into account the sizing characteristics and the inkjet printing performance. The results are summarized in Table 6.

Table 6 – Industrial test results.

|                                     |        | Table 0 - | muusii iai te | est results. |           |          |           |  |
|-------------------------------------|--------|-----------|---------------|--------------|-----------|----------|-----------|--|
|                                     |        |           | Samples       |              |           |          |           |  |
|                                     |        | 1         | 2.1           | 2.2          | 3         | 4        | 5         |  |
| Cobb60 S1/S2                        | g/m2   | 33,5/32,6 | 27,3/27,3     | 24,4/21,5    | 25,3/27,9 | 23,9/26  | 25,9/28,5 |  |
| StCA                                | o      | 92,8      | 92,7          | 94,7         | 93,4      | 104,6    | 99,8      |  |
| TapVel                              | °/s    | 0,13      | 0,13          | 0,13         | 0,11      | 0,09     | 0,10      |  |
| FinVel                              | °/s    | 0,06      | 0,06          | 0,05         | 0,04      | 0,04     | 0,04      |  |
| MaxVel                              | °/s    | 0,43      | 0,35          | 0,34         | 0,29      | 0,24     | 0,35      |  |
| Vel10                               | °/s    | 0,28      | 0,25          | 0,26         | 0,21      | 0,19     | 0,26      |  |
| PDA - W                             | -      | 1,91      | 3,26          | 4,61         | 2,79      | 3,56     | 1,73      |  |
| PDA - Max                           | S      | 0,68      | 0,94          | 1,16         | 0,86      | 0,99     | 0,65      |  |
| PDA - A30                           | -      | 21,4      | 19,5          | 18,2         | 19,0      | 18,0     | 19,1      |  |
| PDA - A60                           | -      | 27,4      | 25,7          | 23,9         | 24,6      | 23,4     | 24,2      |  |
| PDA - Imin (t<60s)                  | -      | 10,8      | 18,8          | 27,1         | 23,2      | 28,6     | 24,3      |  |
|                                     |        |           |               |              |           |          |           |  |
| BendRoug S1/S2                      | ml/min | 108/131   | 102/129       | 108/136      | 103/135   | 102/131  | 97,4/132  |  |
| BendPer                             | ml/min | 1200      | 1190          | 1210         | 1220      | 1200     | 1220      |  |
| TA S1/S2                            | #      | 1,05/1,1  | 1,05/1,05     | 1,1/1,05     | 1,1/1,05  | 1,1/1,05 | 1,05/1,1  |  |
| $SFC^{\scriptscriptstyle (a)}MD/CD$ | %      | 116/120   | 116/122       | 118/124      | 118/115   | 120/122  | 122/124   |  |
| $DFC^{(a)}MD/CD$                    | %      | 100/100   | 98/102        | 98/100       | 100/98    | 102/100  | 100/100   |  |

<sup>(</sup>a) – The kinetic coefficient of friction of sample 1 is assumed to be 100%.

The results of Table 6 show that the sizing agents dosage, in that range of dosages, has no influence on the coefficients of friction neither on toner adhesion.

The following correlation coefficients between the PDA parameters and Cobb 60 were found:

Table 7 – Correlation coefficients between Cobb 60 and PDA parameters.

| Parameter | W     | Max   | A30  | A60  | Imin (t<60s) |
|-----------|-------|-------|------|------|--------------|
| Cobb 60   | -0,78 | -0,79 | 0,97 | 0,91 | -0,93        |

These results show that the A30 and/or A60 parameters could substitute the Cobb measurements, as expected.

The assessment of the inkjet printing quality was performed by testing all the samples in several inkjet printers and then by measuring the main quality parameters. These printers and the used printing mode are listed in Table 8.

Table 8 – Inkjet printers and printing modes used.

| Printer | Manufacturer | Model            | Printing mode      |
|---------|--------------|------------------|--------------------|
| 1       | HP           | Deskjet F370     | Best/plain Paper   |
| 2       | HP           | Photosmart B8850 | Best/plain Paper   |
| 3       | Lexmark      | X8350            | Best/plain Paper   |
| 4       | Lexmark      | X6675            | Normal/Plain Paper |
| 5       | Lexmark      | X7675            | Normal/Plain Paper |
| 6       | Epson        | BX600FW          | Normal/Plain Paper |
| 7       | Epson        | PX800FW          | Normal/Plain Paper |
| 8       | Epson        | SX405            | Normal/Plain Paper |
| 9       | Canon        | IP4600           | Normal/Plain Paper |
| 10      | Canon        | MP240            | Normal/Plain Paper |
| 11      | Canon        | MP980            | Normal/Plain Paper |
| 12      | Canon        | MX860            | Normal/Plain Paper |
| 13      | Canon        | MX7600           | Normal/Plain Paper |

The results obtained are presented using the average and the coefficient of variation for the 6 samples tested with the same inkjet printer. The influence of the amount of the sizing agent added is visible by analysing the coefficient of variation obtained (CV), which is listed in table 9.

Table 9 – Coefficient of variation of the inkjet quality parameters

| Table .             | / - Cucin                | ciciit oi varia | ation of the mix | jet quanty pai | ameters. |      |      |  |
|---------------------|--------------------------|-----------------|------------------|----------------|----------|------|------|--|
|                     | Coefficient of Variation |                 |                  |                |          |      |      |  |
|                     |                          |                 |                  | (%)            |          |      |      |  |
| Printer             | AG                       | OD              |                  |                |          | СВ   | РТ   |  |
|                     | _                        | Cyan            | Magenta          | Yellow         | Black    | _    |      |  |
| HP Photosmart B8850 | 1,8                      | 0,6             | 1,1              | 0,7            | 1,1      | 3,7  | 5,6  |  |
| Lexmark X8350       | 1,6                      | 1,4             | 1,1              | 1,3            | 0,8      | 7,0  | 21,4 |  |
| HP DeskJet F370     | 1,2                      | 1,3             | 1,3              | 1,5            | 3,7      | 11,3 | 33,5 |  |

The results of the inkjet printing quality, just for three different printers, as an example, are presented in Figures 2 and 3.

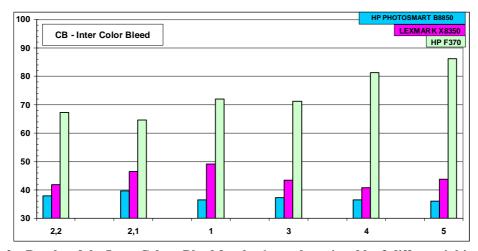


Figure 2 – Results of the Inter-Colour Bleed for the 6 samples printed by 3 different inkjet printers.

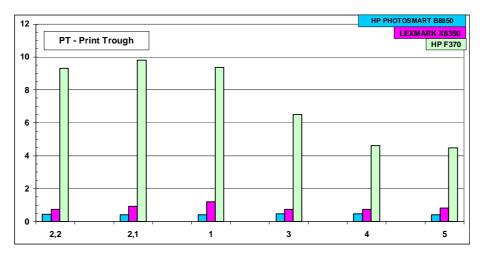


Figure 3 – Results of the Print Trough for the 6 samples printed by 3 different inkjet printers.

The only parameters with the same variability induced by the sizing agents were the Inter Colour Bleed and Print Through for the black ink.

It was only possible to see the effects on the sizing agents variation by using, in the HP F370 printer, higher amounts of black ink to evaluate the Print Through. An inverse relation to the internal sizing agent dosage was detected. The changes on the surface sizing agent did not show a significant impact on the Print Through parameter.

Parallel to this previous work, CD paper samples were taken, from the different test conditions, to study the paper machines CD sizing profiles. The results are summarized in Table 10.

Table 10 - Cobb 60 figures and CD deviation.

|         | Samples |      |      |      |      |      |  |
|---------|---------|------|------|------|------|------|--|
|         | 1       | 2.1  | 2.2  | 3    | 4    | 5    |  |
| Average | 37.1    | 29.3 | 27.3 | 29.7 | 27.4 | 30.6 |  |
| 2 Sigma | 3.7     | 2.1  | 2.1  | 2.1  | 1.2  | 1.7  |  |

A reduction on the CD variation with the increment of the internal sizing agent dosage was observed. The CD Cobb profiles flatten with the increased internal sizing agent dosage.

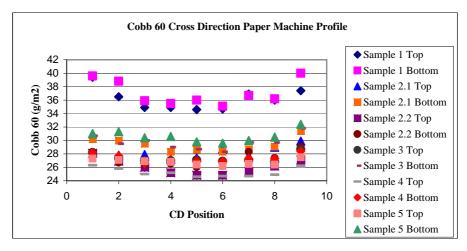


Figure 4 – Paper machine Cobb 60 CD profiles.

#### **CONCLUSIONS**

The best test to evaluate the sizing degree is the B.A.T. test not only in terms of variability and accuracy, but also because it shows real numbers and simulates the time scale used in real printing technologies. The problem with this test is that it is time consuming, and therefore not adequate to be used in a routine basis at paper quality production control.

This study reveals that the best method still is the COBB 60 test, or alternatively the COBB 30 test, which show a very low variability.

It can also be concluded that the PDA test may substitute the COBB test, with advantages regarding the automation and data transfer.

The static contact angle with water seams to be the test which better differentiates the sizing agent dosage on the wet-end and on the surface, being however little sensitive to the latter. This issue seams to be very important for a future control strategy and will be deeply investigated in future works.

It was found that there is no relevant influence of the dosages of the sizing agents on the coefficients of friction and on the toner adhesion, at the levels used in this work.

It was confirmed that, as expected, the surface sizing agent plays an important role in the control of the inkjet inter colour bleed.

This study also shows that the wet-end sizing dosage is the main parameter in controlling the show-through, and has positive influence on the optical density of the inkjet inks.

From this study it is possible to verify that the colour gamut does not significantly vary with the sizing agents dosages.

It can be concluded that the COBB CD variation is dependent on the internal sizing – the lower variability and the lower Cobb value are obtained with the highest ASA dosages in the wet end and the normal dosage of surface sizing agent. One possible explanation for this fact can be the ZD penetration of the surface starch. With low wet end sizing dosages, the starch ZD penetration controlling factor is the air permeability CD profile, or the pore size CD profile. With the increment of the internal sizing, the pore surface tension starts to be a controlling parameter, being the starch penetration more uniform all across the CD profile.

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