Automatic Stöckigt Sizing Test Using Hue Value Variation of a Droplet

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ABSTRACT

The Stöckigt sizing test of the most-commonly used sizing tests is easily influenced by the individual testers' bias in recognizing red coloration. Therefore the test had to be modified to improve its reliability and reproducibility by automated recognition of a coloration procedure during testing. In order to achieve this, all measured variables occurring during the Stöckigt sizing test was first be analyzed and then reflected in the new automatic system. Secondly, the most important principle applied was to transform the RGB values of the droplet image to hue (H), saturation (S) and value (V) respectively. This is because RGB cannot be used as a color standard, owing to RGB's peculiarity of being seriously affected by the observer's point of view. Therefore, the droplet color had to be separated into three distinct factors, namely the HSV values, in order to allow linear analysis of the droplet color. When the average values of the vectors calculated during color variation from yellow to brown were plotted against time, it was possible to determine the vector value of hue, the most sensitive factor among HSV, at the specific time by differentiation of a function when it exceeds the critical point. Then, the specific time consumed up to the critical point was regarded as the Stöckigt sizing degree. The conventional method took more time to recognize an ending point of coloration than the automatic method, and in addition the error ranges of the conventional sizing degrees on the specific addition points of AKD were wider than those of the automatic method.

INTRODUCTION

Most paper is treated with sizing agents to provide water resistance. In general, the most frequently used methods to determine sizing degrees are the Hercules sizing test (HST), the Stöckigt sizing test and the Cobb sizing. In the HST, the remission of monochromatic light at the bottom surface of the sample is photo-electrically measured until the intensity of remitted light has decreased to a preset value, usually 80% of the initial intensity, according to the penetration of a colored test liquid (refer to TAPPI Standard Method T 530). The fluid (e.g., Naphtol Green B) used for HST is a disadvantage for this method, as a test specimen containing $CaCO_3$ will be affected by the formic acid in the HST fluid (Neimo, 1994).

The Cobb test is probably one of the most widely used tests in papermaking. One surface of the test sample with a circular area of 100 cm^2 is exposed to water under a given hydrostatic head for a specified time. At the end of the test, the water is decanted, excess water is blotted off, and the amount of water absorbed is determined by the weight gain, reported as g/m^2 (refer to TAPPI Standard Method T 441). It is known that the principal drawbacks of the method are its insensitivity when applied to either hard-sized or very slightly sized paper or board, inapplicability to thin or absorbent papers, lack of correlation with penetration-type tests, and the requirement for constant attention during the test (Kumler, 1989; Neimo, 1994).

The Stöckigt test uses a test fluid of ammonium thiocyanate solution and the specimen is lightly treated on the top side with a ferric chloride solution. The time required for the thiocyanate and ferric chloride to make contact and react, forming a deep red ferric thiocyanate, is recorded. This method has been widely used in determining the sizing degree in many countries, and has been designated a TAPPI Useful Method 429 and KS M 7025. However, despite the simplicity of this test, the coloring points read by observers from the same specimen may be quite different, and furthermore the test does not distinguish differences at higher sizing levels (Gess, 1981; Kurrle, 1987). Considering all the variables that can influence the test, it is believed that the automatic recognition of color expression for the Stöckigt test will make the test more reliable and reproducible.

In order to achieve this, the automatic system was developed by a RGB recognition method. However, it is well-known that RGB cannot be used as a color standard owing to RGB peculiarity seriously affected by an observers' point of view (Pratt, *1991*). Therefore the recognition of the droplet color should be made by a different method such as three distinct factors such as Hue (H), Saturation (S) and Value (V) for analyzing the color linearly (Gonzanlez , 1992).

This study explored to develop a fundamental principle that was capable of recognizing a coloration point automatically during the Stöckigt sizing test and finally to make the Stöckigt test more standardized.

MATERIALS AND METHOD

Stock preparation and internal sizing

Bleached softwood kraft pulp beaten to 400 mL CSF was used to prepare handsheets of 80 g/m^2 sized by alkyl ketene dimmers (AKD) according to TAPPI Standard Methods T 200 and T 205.

Measurement of sizing degree

For the Stöckigt sizing test, around 0.06 mL of 7% $FeCl_3$ (II) solution was carefully dropped onto a paper specimen, which then was floated on the surface of a 6% NH_4SCN solution. The time taken for the red coloration to appear was noted as the Stöckigt sizing degree. For the automatic Stöckigt sizing test, the same order was applied, but all steps were automatically operated by a liquid dispenser, a stepping motor and a specimen shifter according to the computer's commands. The average time after five tests on each specimen was reported, with standard error, as the sizing degree for a single specimen. Six different testers carried out the measurement in order to reveal potential biases that may occur during the measurement.

RESULTS AND DISCUSSION

Variation of sizing degree by different testers

The experiments were carried out to make sure that the Stöckigt sizing test was dependent upon experimental conditions such as variations in dropping height and the droplet amount of $FeCl_3$ (II) created by the six different testers. Based on these experiments, it would be suggested that modifications to the optimum Stöckigt sizing test are desirable.

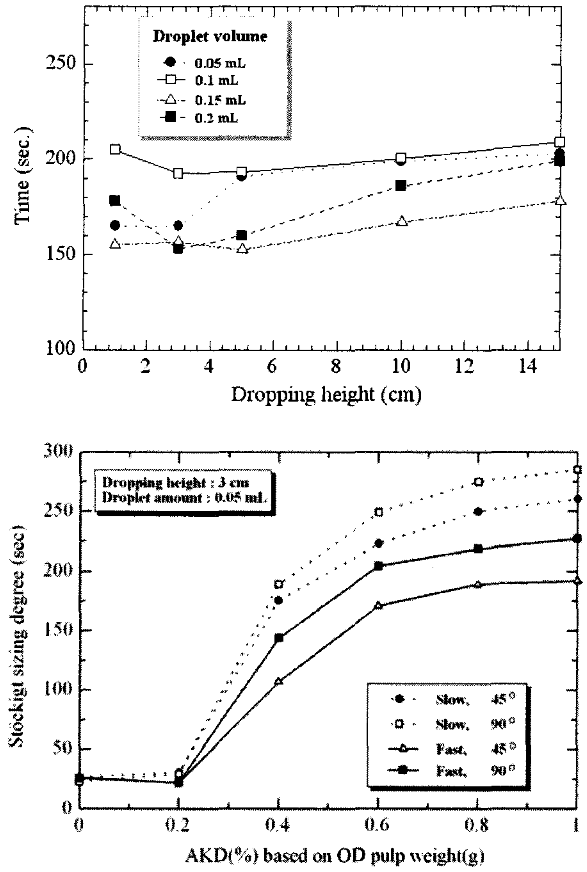
Fig. 1 shows the effects of a dropping height, a droplet amount and dropping speed of a $FeCl_3$ solution and a viewing angle on the Stöckigt sizing degree. When the measuring conditions were not constant, a tester created quite diverse sizing degrees for the same specimen. It made it necessary to regulate the testing method by specifying all testing conditions including the dropping amount, the dropping height, the dropping speed and the viewing angle etc.

Based upon the above results, it can be concluded that the Stöckigt sizing test is extensively dependent upon testers as well as testing conditions. Thus, for reliable and reproducible measurement of Stöckigt sizing degree, it is necessary to modify the test, considering all variables that can be created by different testers as well as testing conditions.

Determination of the starting point of red coloration

In order to determine the starting point of the appearance of red coloration, it is necessary to calculate a feature vector of H from a segmented region of the sequential images, as expressed in the following equations:

$$H = \{\eta_1, \eta_2, \eta_3, ..., \eta_i, ..., \eta_n\} \quad [1]$$



a) Effect of a dropping height and volume

(b) Effect of a viewing angle and dropping speed

Figure 1. Variation of Stöckigt sizing degree by (a) different dropping heights and droplet volumes and (b) a viewing angle and dropping speed.

$$\eta_i = \|h_i\| \quad [2]$$

where:
i = the time of a sequential image,
n = the total number of acquired images,
H = the feature vector set of the sequential images,
η_i = the feature vector at i,
h_i = the hue composing the image at i.

Equation 1 is the set of the feature vector. This expresses the variation of the liquid color from the sequential images during coloration. Each element of H represents the mean values of hues from every individual image. If the set is displayed in a graph, it becomes the change of the feature vector according to time. The feature vector of η_i contains the mean value of the hue of an image at i (Carron *et al*, 1994). The change in these vector values with time is displayed as shown on the left of Fig. 2. The time, t, on the graph is the coloration point determined by the automatic recognition system. In this study, the differentiation was used to determine the time at the coloration point, t. By differentiation of the function, $f(t)$, the graph on the right of Figure 2 was obtained. Here the coloration pint, t is the same as the value on the x-axis under the maximum of $f'(t)$. The differentiation of $f(t)$ is expressed as the following equation 3:

$$f'(t) = \frac{\partial f}{\partial t} \qquad [3]$$

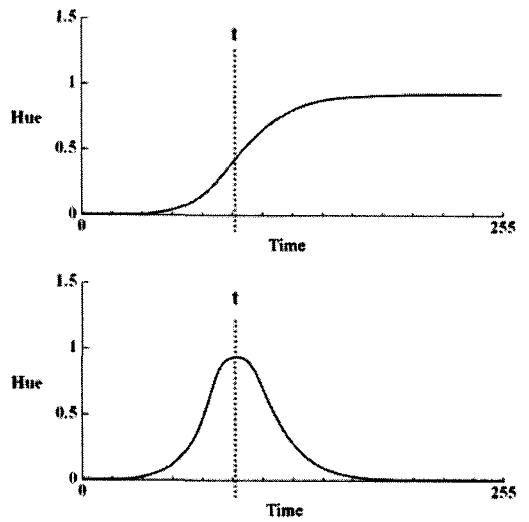


Figure 2. Variation of a feature vector according to time (top) and differential graph of the feature vector (bottom).

In practice, the mask in Fig. 3 was used to apply a discrete value. The regional differentiation was accounted for by applying the mask to the discrete values (refer to Fig. 3 (a)), and its related equation is as follows:

$$\nabla f \approx |z_1 - z_3| \qquad [4]$$

The differential value of the point of interest, z_2, for equation 6 is similar to the difference of two outliers, z_1 and z_3. The value of each mask is shown in Fig. 3 (b). These masks were sequentially applied to the function, f, which is called convolution (15).

z_1	z_2	z_3

(a)

1	0	-1

(b)

Figure 3. Mask used in differential calculation.

HSV variation of a droplet

Fig. 4 shows representative sensitivities of hue, saturation and value in recognizing color variation of a droplet on a paper specimen. Unlike hue variation, saturation and value did not respond well to the gradual change of a droplet's color with elapsed time. This means that saturation and value cannot be used as significant means for recognizing the color variation of the droplet. On the other hand, after 30 seconds, hue sharply decreased until about 130 seconds and then stabilized. This implies that among HSV, hue is the most decisive factor able to detect the specific coloration point.

Through the Hue variation, it was possible to designate a starting point of the stabilizing time with a constant hue value as the Stöckigt sizing degree. The starting point could be known as time taking the droplet color to become deep red through the reaction between ammonium thiocyanate and ferric chloride. Based on this, a measuring system was designed to stop automatically when hue reaches the critical or minimum value of hue reaches.

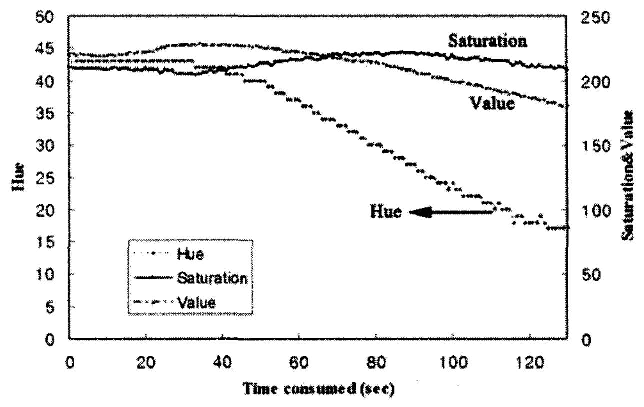


Figure 4. HSV variation of a droplet color along elapsed time during Stöckigt sizing test.

Comparison between automatic Stöckigt sizing test and conventional sizing test

Fig. 5 shows the difference of sizing degrees measured by the automatic Stöckigt sizing tester and the conventional sizing testing method. The point of time recognizing the complete red coloration by two methods were clearly different. As the sizing agent was added to a steadily increasing extent, the difference between two sizing degrees became greater and greater. The sizing degree by the conventional method was greater than that by the automatic sizing test, and in addition the error ranges of the conventional sizing degrees on the specific addition points of AKD were wider than those of the automatic method. The conventional testing method took more time to perceive the final ending point of red coloration of droplet differently from the automatic test. This implies that the automatic method is the much more reliable testing method than the conventional method which was seriously dependent upon testers' bias.

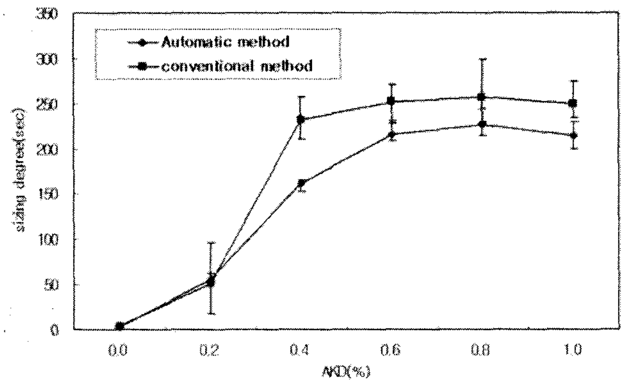


Figure 5. Comparison between the automatic Stöckigt sizing tester and the conventional sizing testing method.

CONCLUSIONS

The Stöckigt sizing test of the most-commonly used sizing tests is easily influenced by the individual testers' bias in recognizing red coloration. Therefore the test had to be modified to improve its reliability and reproducibility by automated recognition of a coloration procedure during testing. In order to achieve this, all measured variables occurring during the Stöckigt sizing test was first be analyzed and then reflected in the new automatic system. Secondly, the most important principle applied was to transform the RGB values of the droplet image to hue (H), saturation (S) and value (V) respectively. This is because RGB cannot be used as a color standard, owing to RGB's peculiarity of being seriously affected by the observer's point of view. Therefore, the droplet color had to be separated into three distinct factors, namely the HSV values, in order to allow linear analysis of the droplet color. When the average values of the vectors calculated during color variation from yellow to brown were plotted against time, it was possible to determine the vector value of hue at the specific time by differentiation of a function when it exceeds the critical point. Then, the specific time consumed up to the critical point was regarded as the Stöckigt sizing degree. Finally the automatic method was proved as the more reliable and reproducible testing method than the conventional test.

REFERENCES

(1) Gess, J. M. - The measurement of sizing in paper, *Tappi* 64(1): 35(1981).
(2) Kurrle, F. L. - Sizing test methods, *TAPPI 1987 Sizing Short Course Notes*, TAPPI PRESS, Atlanta: 9.
(3) Kumler, R.W. - Testing paper and board for sizing in *The Sizing of Paper* edited by W. F. Reynolds, TAPPI PRESS, Atlanta, 1989: 103.
(4) TAPPI Standard Method T 530 - Size test for paper by ink resistance (Hercules-type method), TAPPI PRESS, 1996.
(5) TAPPI Useful Method UM 429 - Sizing of Paper (Thiocyanate Flotation), TAPPI PRESS, 1991.
(6) TAPPI Standard Method T 441 - Water Absorptiveness of Sized (Non-bibulous) Paper and Paperboard (Cobb Test), TAPPI Press, 1996.
(7) Neimo, L. - Measurement of the hydrophobicity of paper, *Paper Science Centre Communication No. 61*, KCL, Finland, 1994: 45.
(8) Gonzalez, R. C., Woods, R. E. - Digital Image Processing, Addison-Wesley, 1992..
(9) Pratt, W. K. - Digital Image Processing, A Wiley-Interscience, 1991.
(10) Carron, T. and Lambert, P. - Color Edge Detector Using Jointly Hue, Saturation and Intensity, IEEE International Conference on Image Processing, 1994.
(11) KS M 7025 - Testing method for sizing degree of paper (Stöckigt method).

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