

# A Simple Method for Measuring the Immobilization Solids of Coating Colors Using an AA-GWR Water Retention Meter

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## ABSTRACT

The water retention of coating colors can be accurately measured by devices such as an AA-GWR water retention meter whose principle of measurement is based on pressure filtration of coatings under an externally applied air pressure over a certain period of time. It was hypothesized that such devices could be also used to determine the immobilization solids (IMS) of coating colors by determining a sudden drop in the rate of dewatering, that is, a sudden change in the drainage curves. To test this hypothesis, the immobilization solids of coating colors containing various thickeners and water retention additives at different levels were first accurately measured by a modified immobilization tester based on the well-known gloss drop method, and then their values were compared with those obtained by an AA-GWR water retention tester. They agreed very well and showed that the standard deviation is only 0.14% in the IMS points between both methods. This good agreement was not surprising because both test methods are based on the same end-point, that is, the immobilization solids point at which menisci begin to form at the coating surface. Theoretical considerations supporting this new method for measuring the immobilization solids of coating colors are presented and some recommendations for the test method are discussed. Also, the effect of various thickeners and water retention additives on the properties and printability of coated papers is discussed.

## INTRODUCTION

The immobilization solids (IMS) of coating colors are the critical transition points where the coating colors become immobilized. These points are very important from the viewpoints of coating runnability and structure. For this reason, the immobilization solids have been extensively studied in the literature [1-7]. In 1982, Watanabe and Lepoutre [3] showed that the

immobilization solids of coatings could be determined by a sudden drop in gloss. This sudden drop indicates that the water film at the coating surface is no longer continuous, and menisci begin to form at the coating surface. Using this gloss drop technique, Herbet, Gautam, and Whalen-Shaw [5] developed a test method for measuring the immobilization solids of coating colors in 1990. Since then, this method has been widely studied and has become the standard

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method for the measurement of the immobilization solids of coatings. Although this tester can be modified to measure both the water retention and immobilization solids of coatings and make the test easier, such modified devices are not yet commercially available.

Dewatering of coating colors and water penetration into the base sheet are well known phenomena in the paper industry. The most frequently observed effect of water absorption into the base paper is an increase in the recirculated coating solids content with time. However, under drastic dewatering, the rheological behavior of the coating color can be impaired, causing runnability problems such as scratches, streaks, and whiskers [8]. Although it would be ideal to be able to determine both the static and dynamic water retention and immobilization solids of coatings on paper substrates simultaneously, such laboratory testing devices are not yet widely available. However, a recent paper by Willenbacher et al [9] on a new laboratory test to characterize rheologically the immobilization and the dewatering of paper coating colors. is very promising.

From the above reasons, simple measurements of the water retention and immobilization solids of coatings on paper were thought to be well worth the effort. The water retention of coating colors has been accurately measured by devices such as an AA-GWR water retention meter [10, 11] whose principle of measurement is based on pressure filtration of coatings under an externally applied air pressure over a certain period of time. It was initially hypothesized that such devices could be also used to determine the immobilization solids of coating colors by determining a sudden drop in the rate of dewatering—that is, a sudden change in the coating solids with time—determined from a simple mass balance before and after each test. To test this hypothesis, the immobilization solids of coating colors containing various thickeners and water retention additives at different levels were first accurately measured by a modified immobilization tester based on the well-known gloss drop method, and then their values were compared with those obtained by an AA-GWR water retention tester.

This paper will first discuss some theoretical considerations for the measurement of the immobilization

solids of coatings using water retention testers such as an AA-GWR water retention meter, based on pressure filtration of coatings on paper substrates. Secondly, the paper will describe a modified immobilization tester based on a gloss drop method and its measurement of the immobilization solids of coatings containing hydrophobically modified alkali-swellable emulsion thickener (HASE), alkali-soluble emulsion thickeners (ASE), and carboxymethyl cellulose (CMC). Finally, measurements of the immobilization solids of the same coatings by an AA-GWR water retention meter will be discussed, and then the results obtained by these two methods will be compared. Also, some recommendations for the use of water retention testers to measure the IMS points will be presented, and the effect of various thickeners and water retention additives used on the properties and printability of coated papers will be discussed.

## THEORETICAL CONSIDERATIONS FOR DEWATERING AND IMMOBILIZATION SOLIDS

Water penetrates into paper by the following mechanisms [12-15]: (1) water transport through the pores by capillary action, (2) vapor phase transport through the porous structure, (3) surface diffusion in the pores, and (4) water diffusion through the fibers. However, when an external pressure is applied, the pressure penetration through the pores will be a dominant flow [16]. Now, let us take a look at the dewatering of coating colors applied onto paper under an externally applied air pressure. Coating colors on paper dewater by the same four mechanisms by which water penetrates paper, except that the aqueous phase in the wet coatings dewater through the packed pigment particles and into paper. As the wet coatings become immobilized, that is, as the dispersed particles are packed into the immobilized structures, a free planar aqueous surface disappears and menisci begin to form at the coating surface. As mentioned already, Watanabe and Lepoutre [3] showed that the gloss drops suddenly at this immobilization solids point due to the appearance of the menisci.

At immobilization, the formation of menisci at the coating surface will create upward capillary forces which counter-balance the applied pressure. The capillary pressure drop across the curved surface can be calculated by the following Young-Laplace equation:

$$\Delta P_c = 2 \gamma \cos \theta / r$$

where  $\Delta P_c$  is the capillary pressure drop,  $\gamma$  is the surface tension of the aqueous phase,  $\theta$  is the contact angle, and  $r$  is the pore radius.

By assuming that the average surface tension is about 45 dynes/cm, the contact angle is 0, and the average radius of pores is  $0.25 \mu\text{m}$ , the average capillary pressure can be estimated to be:

$$\begin{aligned} \Delta P_c &= 2 \times 45 \times 1 \text{ dynes/cm} / (0.25 \times 10^{-4} \text{ cm}) = \\ &3.6 \times 10^6 \text{ dynes/cm}^2 = 3.6 \text{ bar} = 360 \text{ kPa} = \\ &52.2 \text{ psi} \end{aligned}$$

The above-estimated back pressure created by the capillary force is high enough to stop the pressure filtration in the water retention test under an externally applied air pressure (e.g., 25 psig = 1.72 bars = 172 kPa). If a Teflon plug is used to pressure the coating [10], then the plug surface will be in contact with the coating surface so that the back pressure will be further increased by vacuum formation. In both cases, as soon as the wet coatings become immobilized and the menisci begin to form at the coating surface, the upward capillary pressure will counter-balance the

applied pressure, thus causing the pressure filtration to be stopped suddenly at the immobilization solids. Based on this sudden drop in the dewatering, the immobilization solids can be determined from the coating solids vs. time plots obtained by water retention testing devices based on pressure filtration.

Since both the gloss drop method and the dewatering drop method are based on the same end-point where the menisci form at the coating surface, the results obtained by these two methods are expected to agree very well. This is the theoretical basis of using the water retention testers such as an AA-GWR water retention meter for the determination of the immobilization solids.

## EXPERIMENTAL

Coating formulations composed of 35 parts No. 1 clay, 65 parts  $\text{CaCO}_3$ , and 12 parts styrene butadiene latex with 0.05-0.2 part synthetic thickener additives and 0.25-1.0 part CMC, respectively, were prepared as outlined in Table 1. Thickener A was prepared by Union Carbide, Inc. (Cary, North Carolina) and Thickeners B and C were prepared by J.W. Chemical Company (Pusan, Korea). The properties of these thickener/water retention additives are provided in Table 2. In this study, the terms *thickener and water retention additive* have been used interchangeably.

The coating colors were prepared by first dispersing the pigments in distilled water with a high shear

**Table 1. Coating Formulations and Properties**

Formulation (Parts) And Coating Color Properties	A	B	C	D	E	F	G	H
No. 1 Clay (Hydragloss 90)	35	35	35	35	35	35	35	35
$\text{CaCO}_3$ (Hydrocarb 90)	65	65	65	65	65	65	65	65
S/B Latex (Dow CP 638)	12	12	12	12	12	12	12	12
Thickener A (UCAR-A)	<b>0.05</b>	<b>0.1</b>	<b>0.2</b>	0	0	0	0	0
Thickener B (J-A)	0	0	0	<b>0.1</b>	0	0	0	0
Thickener C (J-B)	0	0	0	0	<b>0.1</b>	0	0	0
CMC (7L) @ 5%	0	0	0	0	0	<b>0.25</b>	<b>0.5</b>	<b>1</b>
%Solids	68.6	68.8	68.7	68.7	68.5	66.8	65.0	62.0
Viscosity (Brookfield DV-III), cps	1,100	1,690	3,570	1,720	1,820	1,480	1,990	2,660
pH (Corning pH Meter 340)	8.28	8.07	7.92	7.97	8.06	8.30	8.36	8.42

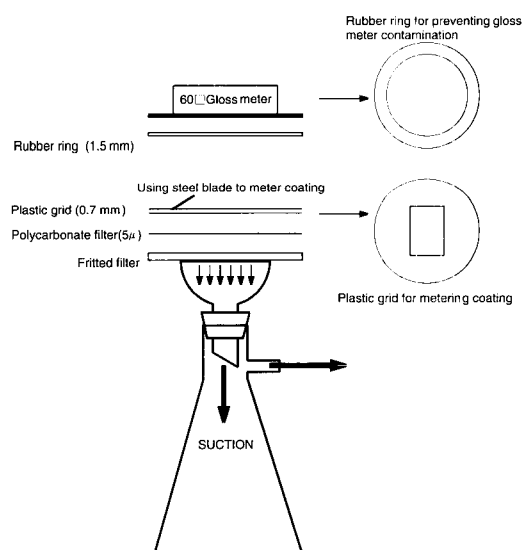
**Table 2. Monomer Compositions of Thickeners A, B, and C**

Composition (Wt. %) and Thickener Type	Ethyl Acrylate	Methacrylic Acid	Macromonomer (Hydrophobe: Stearyl Alcohol)	CTA (Chain Transfer Agent)	Molecular Weight
Thickener A (UCAR-A)	55	40	5	0	High
Thickener B(J-A)	75	25	0	0.5	Lower
Thickener C(J-B)	70	30	0	0	Higher

Cowles disperser. The binder was then added to the pigment under mild agitation and the pH adjusted to above 9.0 with NaOH before adding the associative (hydrophobically-modified alkali-swellable) and non-associative alkali-soluble emulsion thickeners. The solids contents of the coating colors were then adjusted to approximately 68.5 % solids with dilution water. The viscosities of the coating colors were measured using a Brookfield DV-III viscometer, No. 6 spindle, 100 rpm, 30 °C. The CMC was added to the coating colors as a 5% solution to prevent lumping. Since the viscosity and solids of the coating color can have a large influence on the IMS point and the rate of dewatering of the coating, care was taken to maintain similar viscosities and solids, except for those CMC-containing coating colors.

The immobilization solids (IMS) of the coatings were determined by measuring a sudden drop in gloss with time. In this method, the gloss is plotted against drainage time until a change in slope is observed. The solids of the coating at this point is called the IMS point. Preliminary tests were performed by applying the coating color onto a ceramic plate, as described by Herbet et al [5], but this method proved to be unreliable and time consuming. Our modified tester is shown in Figure 1. The test was performed using a fritted filter and 5 $\mu$ m polycarbonate filter. The 5 $\mu$ m polycarbonate filter was placed over the fritted filter which was placed in a vacuum flask. A 0.7mm thick plastic plate with a square cut-off was placed over the polycarbonate filter and the coating color was applied in the area of the grid with a stiff blade to form an even coating layer. A gloss meter equipped with a 1.5mm rubber plate was then placed over the plastic plate and the change in gloss with time recorded. A 60° Gardner

GlossGard-2 gloss meter was used for measuring the continuous change in gloss with time. The rubber plate was used to prevent the contamination of the gloss meter with the coating color and to provide a good seal between the coating layer and gloss meter. The IMS test measurements were performed at least three times for each sample and the mean and variation for each sample were reported. In performing this test, it was found that the conditioning of the fritted filter was extremely important to the reproducibility of the test data. Therefore, the following standard practice was adopted: The fritted filter was washed and dried in a 220 °F (105 °C) oven for 10 minutes, cooled with compressed air for 1 minute, and then dried under vacuum for 2 minutes prior to measuring the IMS point for each coating.

**Figure 1. A Modified Immobilization Solids Tester Based on a Gloss Drop Method.**

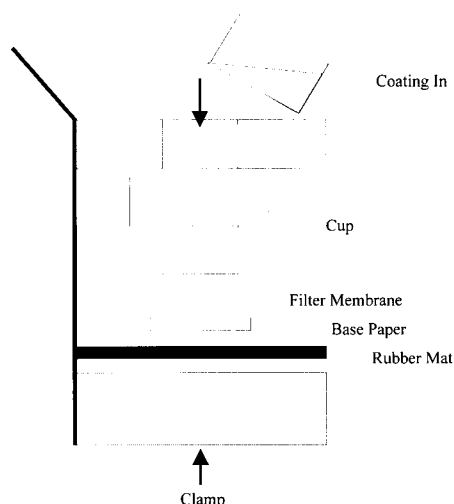


Figure 2. AA-GWR Water Retention Meter [11].

Table 3. Technical Data for AA-GWR Water Retention Meter.

Test Cell Area:	8.0 cm <sup>2</sup>
Sample Volume:	10 ml
Cell Pressure:	0-30 psi
Input Pressure:	100 psi
Electrical:	115 V
Timer:	LCD Digital Display
Weight:	18 lbs. Net
Dimensions:	10x10x7 (HxWxD) in inches

The water retention and immobilization solids of the coatings were measured using an AA-GWR water retention tester shown in Figure 2 (11). Table 3 shows the technical data for the tester. Tests were performed at 25 psig (1.72 bars = 172 kPa) at various times. The change in coating solids was calculated from the water loss through a polycarbonate filter into paper. The coating solids were then plotted against the drainage time to obtain a complete drainage curve. Changes in the slope of the drainage curves were then analyzed and the points where the slopes suddenly changed and became flat were determined as the immobilization solids. These points were compared with the IMS points determined by the gloss drop method.

## RESULTS AND DISCUSSION

### Experiment Results by a Modified Gloss Immobilization Solids Tester

The Modified Gloss IMS Tester produced the immobilization solids points with variations of 0.03 - 0.6% solids. Errors less than 8.0 % were found for the time to reach the immobilization solids point for each of the eight coatings tested. Experimental data obtained by the Modified Gloss IMS Tester are shown in Table 4 and a summary on the IMS points and times to immobilization is shown in Table 5.

Figures 3 and 4 show the influences of thickener /water retention additives on the IMS point and the time to reach the IMS point for each coating, respectively. From these two figures, it is evident that regardless of the type of thickener/water retention additives used, the immobilization solids of the coating decreased with increasing amount of thickener/water retention additives. The time to reach the immobilization solids also increased with increasing thickener /water retention

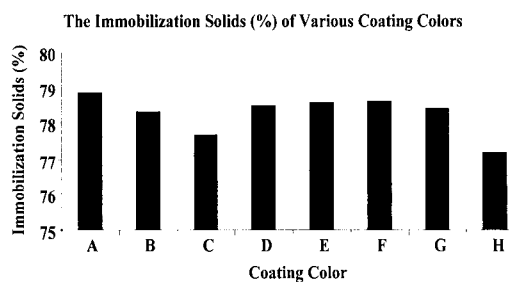


Figure 3. Comparison of Immobilization Solids.

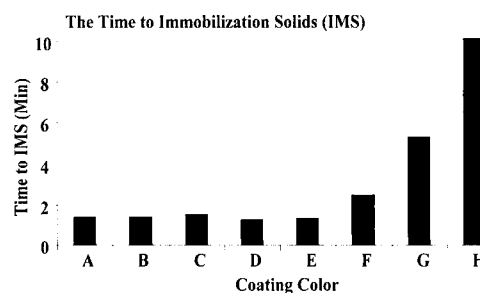


Figure 4. Comparison of the Times to Immobilization.

**Table 4. Experimental Data Obtained by the Modified Gloss IMS Tester.**

Coating Color	Initial Gloss(%)	Last Gloss (%)	Time to Rapid Drop Point (min: sec) Mean and Variation	Immobilization Solids (%) Mean and Variation	Test Conditions
A	1. 55.6	52.0	1:41	78.80	<ul style="list-style-type: none"> <li>• Fritted filter treatment for even absorption in each test: Maintain the same structure, moisture, and temperature</li> <li>1. Washing with fresh water each time</li> <li>2. Drying in 105°C (220°F) oven for 10 min.</li> <li>3. Cooling with compressed air for 1 min.</li> <li>4. Drying under vacuum for 2 min.</li> <li>• Room temperature: 21°C (70°F)</li> <li>• Microwave oven for immobilization solids measurement: Mark 2 Standard, Omnimark Co.</li> <li>• Gloss meter: Gardner Glossgard - 60°, Pacific Scientific Co.</li> </ul>
	2. 58.4	53.4	1:37 Mean: 1:39	78.97 Mean: 78.87	
	3. 57.1	51.8	1:40 Variation: 2.0%	78.86 Variation: 0.1%	
B	1. 57.0	55.2	1:35	78.23	
	2. 60.8	59.9	1:37 Mean: 1:40	78.55 Mean: 78.33	
	3. 58.4	57.1	1:48 Variation: 8.0%	78.21 Variation: 0.3%	
C	1. 55.9	54.5	2:02	77.96	
	2. 55.9	54.2	1:54 Mean: 1:56	77.93 Mean: 77.70	
	3. 55.0	53.7	1:40 Variation: 5.2%	77.22 Variation: 0.6%	
D	1. 59.0	54.9	1:30	78.83	
	2. 59.5	54.1	1:29 Mean: 1:30	78.40 Mean: 78.48	
	3. 58.7	53.7	1:31 Variation: 1.1%	78.20 Variation: 0.4%	
E	1. 58.6	52.5	1:35	78.40	
	2. 57.3	51.7	1:39 Mean: 1:36	78.66 Mean: 78.56	
	3. 57.2	51.8	1:35 Variation: 3.1%	78.62 Variation: 0.2%	
F	1. 59.2	55.3	2:45	78.56	
	2. 59.5	55.2	2:58 Mean: 2:49	78.65 Mean: 78.63	
	3. 59.5	56.1	2:44 Variation: 5.3%	78.69 Variation: 0.1%	
G	1. 60.4	56.1	5:25	78.47	
	2. 60.5	57.0	5:33 Mean: 5:29	78.22 Mean: 78.43	
	3. 60.9	55.4	5:28 Variation: 1.2%	78.60 Variation: 0.3%	
H	1. 55.6	51.2	10:44	77.17	
	2. 55.8	51.0	11:33 Mean: 10:56	77.22 Mean: 77.19	
	3. 56.4	53.1	10:30 Variation: 5.6%	78.20 Variation: 0.03%	
<b>Variation of Measurements</b>			<b>1.1 - 8.0%</b>	<b>0.03 - 0.6%</b>	

**Table 5. The IMS Points and Times to Immobilization Obtained by the Modified Gloss IMS Tester.**

Coating Color	IMS (%)	Time to IMS (Min:Sec)
A	78.87	1:39
B	78.83	1:40
C	77.70	1:56
D	78.48	1:30
E	78.56	1:36
F	78.63	2:49
G	78.43	5:29
H	77.19	10:56

additives, however, the amount of CMC added had a greater effect on the time to reach the IMS point than that of the associative and non-associative synthetic thickeners. This may have been partly due to lower

solids at higher levels of CMC, but also partly due to its greater water-holding capacity.

### Experimental Results by an AA-GWR Water Retention Tester

The water retention and immobilization solids of coating colors were measured by using the AA-GWR Water Retention Meter. The water retention of coating colors was measured by the following procedures: (1) The absorbing paper is weighed and placed on a rubber mat, (2) The filter and cup are set on top of the paper and then clamped into place, (3) The coating color is poured into the test cell and immediately pressurized for a given time period, and (4) Upon completion of the test period, the paper is re-weighed to determine the

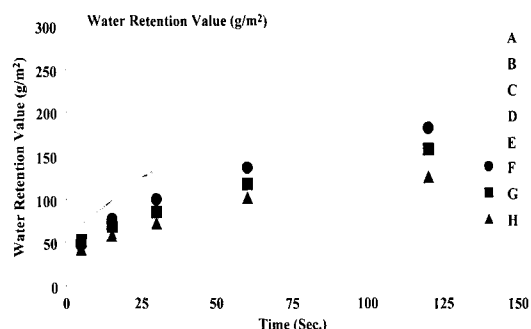


Figure 5. The Water Retention Values of Coatings

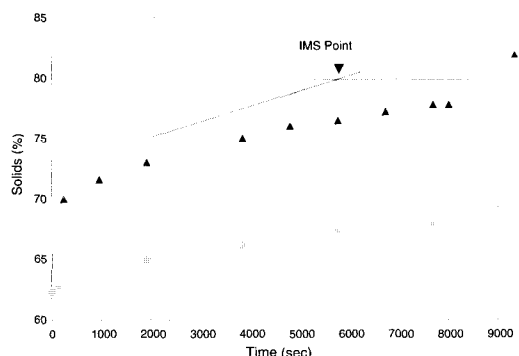


Figure 6. Drainage Rate Curves by AA-GWR Water Retention Meter.

amount of liquid dewatered from the coating. The amount of liquid dewatered is calculated in terms of g/m<sup>2</sup> and reported as the water retention value. The water retention values of coatings measured at a constant pressure of 25 psig are plotted against time in Figure 5. We can see from Figure 5 that the water retention values of coating colors decreased with increasing amount of thickener/water retention additives, regardless of their type. However, the coating color responded differently to different additives.

Figure 6 shows the drainage curves obtained by the AA-GWR Water Retention Meter. The % solids in Figure 6 were calculated according to the following equation:

$$\% \text{ Solids} = \frac{(\text{Weight of Coating}) \times (\text{Initial \% Solids})}{[\text{Weight of Coating} - (\text{Weight of Wet Paper} - \text{Weight of Dry Paper})]}$$

The immobilization solids points were determined from these drainage curves, as shown in Figure 6. In fact, the drainage curves become flat at and after the

immobilization solids because of the formation of menisci which would counter-balance the applied pressure and stop the pressure filtration, as discussed already in the section on the Theoretical Considerations for Dewatering and Immobilization Solids. For curve H, only its initial portion is shown in Figure 6, since Coating H had the highest water retention value among the samples, as shown in Figure 5, and the longest time to reach the IMS point. However, its endpoint was as sharp as that of the curve A from its entire curve.

Table 6 shows the comparison of the IMS points determined by the two methods. From the table, we can see that the IMS points determined by these two methods are in good agreement, thus confirming that devices such as AA-GWR Water Retention Meter could be used to determine both the water retention and immobilization solids of coating colors on paper. In fact, the standard deviation of the differences between the IMS points obtained by two methods is only 0.14%, as shown in Table 6.

Table 6. Comparison of IMS Points Obtained by the Gloss IMS Tester and AA-GWR Water Retention Meter.

Coating Color	IMS (%) by Gloss Drop Method	IMS (%) by AA-GWR Method
A	78.87	79.92
B	78.83	78.40
C	77.70	77.79
D	78.48	78.73
E	78.56	78.16
H	77.19	77.51

Standard Deviation of the Differences between the IMS Points Obtained by Two Methods = 0.14% Solids

### Recommendations for Measuring the Immobilization Solids Using an AA-GWR Water Retention Meter

Since time to reach the IMS point depends on how close to the IMS point an initial coating solids is, an amount of the coating sample added in the cell, and an applied air pressure, it is recommended to formulate the coating solids as high as possible, add smaller amounts of the coating sample, apply a higher air pressure or use any combinations of these variables so that test times can be shortened. Also, only three coating solids points below and two above the IMS point would be sufficient to construct a complete drainage curve from which the IMS point can be obtained, as shown in Figure 6. Furthermore, a test unit having multiple testing port chambers can be designed to shorten test times.

For quality control purposes, only a single dewatering value measured over about 1.2 to 1.5 times the known time to the IMS point for the control would be needed because its coating solids would not change very much after the IMS point, that is, the coating solids vs. drainage time curve becomes flat and independent of the drainage time after the IMS point, as shown in Figure 6. Thus, this simple method measuring the IMS point could be used for quality control.

### The Effect of Thickener/Water Retention Additives on Paper and Printing Properties

To determine the influence of different types of thickener/water retention additives on the print quality, mainly print-mottle, the coating colors were applied onto a commercial free sheet using a CLC (Cylindrical Laboratory Coater) with a stiff blade at 4300 fpm. All coating colors ran well with the exception of Coating C, which did not run well due to its high viscosity (3,570 cps). The runnability problems were associated with the presence of many spits and skips, which prevented us from obtaining the desired target coat weight of 15 g/m<sup>2</sup>. After permitting the samples to condition for at least 24 hours in a TAPPI standard controlled atmosphere, the samples were calendered at 60°C, 3 nips, and 900 pli. The gloss, PPS porosity, and PPS roughness of the samples were then measured according to TAPPI standards. The print mottle of the samples was both visually ranked and measured using a Tobias Mottle tester, after applying a Croda Ink stain.

Table 7 shows the paper and printing properties of each sample. From the results reported in Table 7, the following observations have been made: (1) the higher the coating color solids, the higher the sheet gloss, as expected [17], (2) it appears that Thickener A (an associative synthetic thickener) exhibited rougher sur-

**Table 7. Paper and Printing Properties.**

		A	B	C	D	E	F	G	H
Calendered Paper Properties	Roughness	1.51	1.51	-	1.31	1.26	1.35	1.42	1.57
	Porosity	1.95	1.84	-	1.73	1.73	1.57	1.49	1.44
	Paper Gloss: 75° (%)	75.5	73.0	-	76.5	76.5	72.0	69.0	65.0
Printing Properties	Print Gloss: 75° (%)	88	87	-	88	89	89	82	82
	Δ Gloss: 75° (%)	12.5	14.0	-	11.5	12.5	17.0	13.0	17.0
	Croda Stain Mottle Mottle / STD.	2 76/14	2 81/12	-	5 67/6	5 69/10	5 61/10	4 69/11	2 79/11
Test Conditions	Base Paper: 80 gms								
	Coater: CLC 6000, 4300 fpm (1300 mpm), 15 gms/side								
	Calendering Conditions: 60°C, 3 Nips, 900 pli (160.7 Kg/cm) by Soft-Nip Calender								
	Printability Tester: Japanese RI Tester								
	Parker Print-Surf Tester for Roughness and Porosity								
	Gloss Meter: Hunter 75°								
Visual Mottle Ranking for Croda-Stained Samples: 1 (Worst) and 5 (Best)									
Mottle Test for Croda-Stained Samples: Tobias MTI Mottle Tester									



face, higher porosity, lower sheet gloss, and worse print mottle than its counterparts, Thickeners B and C (non-associative synthetic thickeners), and (3) for the CMC-containing coating colors, lowering the coating color solids with increasing CMC was highly detrimental to the sheet gloss and print mottle. The second observation made above suggests that for high-solids paper coatings, non-associative synthetic thickener/water retention additives would be preferred to associative synthetic thickeners which would form wet coating networks.

## SUMMARY

A modified gloss drop immobilization solids tester was very reproducible for the measurement of the immobilization solids of coating colors. The immobilization solids of coating colors containing various thickener/water retention additives were first determined by this tester, and then these values were compared with those obtained by the AA-GWR Water Retention Meter. It was found that the results obtained by these two testers were in good agreement, thus confirming that devices such as an AA-GWR retention meter can be used to determine the immobilization solids of coatings.

In fact, the theoretical considerations for the dewatering and immobilization of coatings showed that the gloss drop method and the water retention test method are based on the same end-point: the immobilization solids point where menisci begin to form at the coating surface. This theory on the new test method and the experimental evidence demonstrated in this study strongly supported our hypothesis that the water retention and immobilization solids of coatings on paper can be measured by devices such as an AA-GWR water retention meter.

It has been recommended for the proposed immobilization solids test method to formulate coating solids as high as possible, adjust the amount of the coating sample added in the cell or apply a higher pressure so that test times can be shortened without sacrificing the accuracy of the data. With these considerations, this test method can potentially become a

routine test for both coating research and quality control.

Finally, some observations were made on the effect of various thickener/water retention additives used on paper and printing properties. For high-solids paper coatings, non-associative synthetic thickener/water retention additives performed better than either an associative synthetic thickener or CMC.

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