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A New OCC Wax & Hot Melt Measurement Technique

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Abstract

A new measurement technique is being developed to measure the concentration of wax and other contaminants in the old corrugated carton (OCC) process stream that melt and flow at process temperatures. In this method, conventional handsheets produced from process samples are dried under controlled conditions that cause the waxy components to melt and flow into the interstices of the surrounding fiber matrix. When re-wet the water acts as a stain on the fibers having no waxy coating, turning them a dark brown. The areas wet-out by the wax components remain light brown and contrast with the surrounding dark brown wet fibers. A modified scanning technique allows the measurement of these light brown areas. Thus, no dyes, special coated paper, or powder are needed to enhance the contrast between waxy contaminants and the background. The method holds promise of repeatability and is quite specific to waxes and contaminants that melt in the OCC process.

Introduction:

As part of an on going effort to develop a simple stickies measurement technique, the authors discovered a phenomenon that should prove valuable to investigators, process engineers, and quality control managers concerned with the quantification of contaminants that melt at process temperatures. It is known that waxes and hot melts when heated flow out and "wet" the fibers in their immediate area. What was not recognized is when the specimen hand sheet is re-wet after drying under controlled conditions the fibers containing no waxy matter are "stained" a dark brown and the waxy areas remain light brown. Measuring these light brown areas while the handsheet is wet, or damp, provides a measure of the wax concentration in the handsheet. The authors call this new technique the "RMD OCC Wet Specimen Method".

The RMD Test Apparatus:

Previously, the authors had discovered and developed a measurement technique to measure stickies that used a black palette and a box with a clear acrylic bottom. In this technique the wet specimen is placed on the black palette and gently rolled to remove the air between it and the face of the palette. The specimen and palette are then placed face down in the acrylic box and the entire apparatus placed on the image analysis scanner.

Preparing the Handsheet:

A few trials with the RMD Wet Specimen Test Apparatus demonstrated that the handsheet was two sided and under certain conditions could yield different counts on each side. Since the sheet is damp during the test and had to be turned over to measure the other side, the weight of the specimen hand sheet was increased to 2.4 gms.

Determining the Proper Curing Time:

It was also known that the blotter papers would absorb the waxes from the specimen if the handsheet was left in the dryer too long. Previous work with stickies had indicated this migration would occur and that the time the specimen was exposed to the heat was a critical part of the test. Some trials using a handsheet dryer with a surface

temperature of 190° C indicated the optimum time for the 2.4 gm 155 mm diameter handsheet was 7 minutes. This time was arrived at empirically by testing a standard concentration of wax in virgin pulp at different dwell times and looking at the facing blotter papers in water. When blotter is wet, the migrating wax shows as white against the gray translucent blotter fibers. The optimal time was clearly indicated at the point where the wax was developed the most on the handsheet and the least on the facing blotters.

The Test:

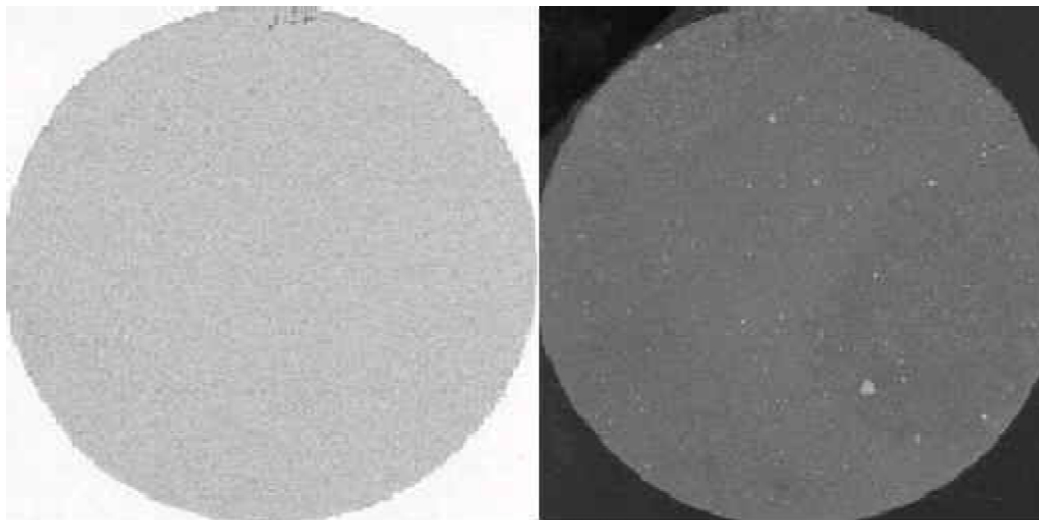


Figure 1: Images of the same 0.01% wax on OCC in virgin kraft. The left image is the specimen dry against a white background. The right image is of the wet specimen against a black background.

To measure the effectiveness of the test designed to take advantage of this wet staining phenomenon, handsheets were produced from a controlled quantity of virgin unbleached kraft adulterated with waxed corrugated cartons to produce a range of concentrations. The cartons were 30% wax coating by weight.

Five 2.4 gm handsheets were produced at each of these wax concentration levels, 0%, 0.0001%, 0.001%, 0.01%, 0.1%. These handsheets were all cured at 190° C for 7 minutes and tested using the following procedure.

Procedure:

The cured handsheets were analyzed individually. Each was placed in room temperature tap water, carefully removed when saturated, and placed on the black palette. The air was removed from between the palette and the specimen by gently rolling it with a soft rubber roll. The black palette was then placed face down in the clear acrylic box, and the entire apparatus was placed on the scanner and was scanned by the image analysis system.

Before placing the specimen/palette in the box any water drops left from previous tests were dabbed away and the clear bottom cleaned of any fiber and smudges.

Upon completing the test on the first side the damp specimen was carefully removed from the palette, turned over, rolled, and the backside was tested the same way as the first side.

Image Analysis:

Figure 1 shows a side by side comparison of images the same specimen wet and dry, as acquired by the Verity IA's Stickies analysis program. Figures 2 and 3 show the same area of this specimen magnified. The wax is clearly shown as the white areas contrasting with the dark brown wet kraft fibers in Figure 3.



Figure 2: Magnified section of the dry 0.01 % wax on OCC in virgin kraft, 2.4 gm hadsheet including the label mark at the top center. There is no evidence of wax.

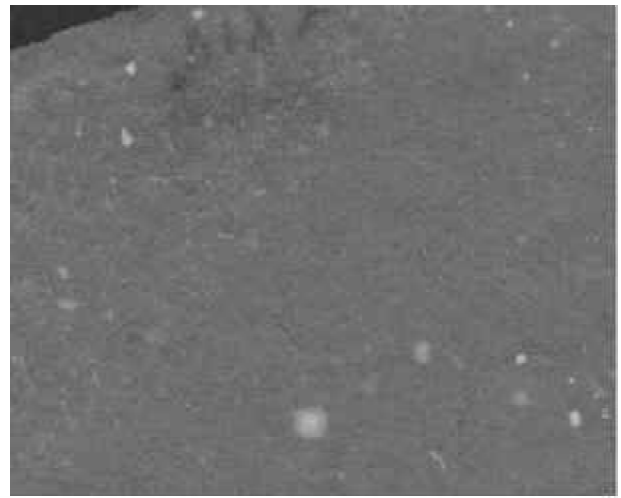


Figure 3: The same magnified section as Figure 2. The specimen is wet and placed on the black palette when the image is acquired. Shows the wax as light brown (shown here as light gray) areas against the wet dark brown fibers. (There is a slight rotational displacement between Fig's 3 & 4.)

The Verity IA image analysis system brings the entire image into memory where it calculates the threshold based upon the background reflectivity in the acquired image. By working on the current image held in memory instead of making a pre-scan requiring two scans, it is able to compensate for scan to scan variations that result from an inherent instability in most CCD based devices. As a result, it can distinguish objects whose reflectivity is very close to the background reflectivity level if necessary.

Image Analysis Calibration:

To calibrate the system and to be sure the scanner has stabilized, the Verity IA software compares the PPM results of at least two scans of the same specimen. If the results are within a preset tolerance, which for this series is 5%, then the results are accepted and automatically recorded in the Excel spreadsheet.

The tests run to date indicate a setting of between 15 and 25% contrast between the background and the measured waxes provides a good operating range.

Results:

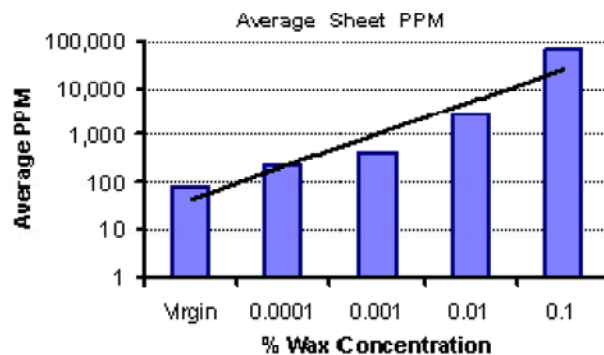


Chart 1: Plot of the average PPM as measured at 25% contrast for each of five wax concentrations in virgin kraft pulp (Addendum A). The wax concentration is by weight of the wax in OD pulp.

Both sides of five 2.4 gm handsheets were analyzed at each of five wax concentration levels: 0%, 0.0001%, 0.001%, 0.01%, 0.1%. These handsheets were all cured at 190° C for 7 minutes and tested using the procedure described above.

An object contrast setting of 25% was used to extract the size frequency distribution for the light brown areas larger than 0.06 sq mm against the dark water stained brown fiber. The PMM was then automatically computed for each hand sheet. The data from the five sheets were then averaged and reported as averages for the five handsheets as shown in addendum A and plotted in Chart 1.

The degree of variation was also calculated and reported in Addendum A as Figure 5. The authors began the investigation with three handsheets and moved quickly to five as it was soon discovered the sampling technique in the laboratory contributed to the variation within the test results. As a result the five sheets tested on both sides should be viewed as a single test.

These preliminary test and data will be followed with an examination of repeatability.

Instrument sensitivity:

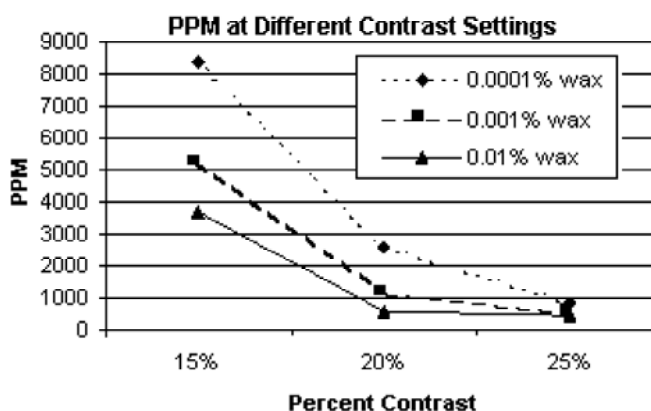


Chart 2: The effect of increased object contrast sensitivity upon the calculated PPM at variable levels of wax concentration. Data are plotted linearly.

Upon reviewing these data it became apparent an evaluation of the measurement response to variations in the instrument contrast sensitivity was necessary. Accordingly a short test of one hand sheet from each concentration group was conducted at three levels of sensitivity. The results are shown on chart 2 and in Figure 4.

These data indicate clearly that a greater differentiation of wax concentration can be achieved by running at higher sensitivity to low contrast image object. Follow-up tests for repeatability will incorporate an investigation to determine the optimal setting.

Object Contrast Setting	PPM		
	15%	20%	25%
0.01% wax	8353	2552	832
0.001% wax	5207	1098	520
0.0001% wax	3688	539	409

Figure 4: Data used in chart 2. Variable sensitivity. The lower the contrast setting the more sensitive the measurement.

Conclusion:

The RMD Wet Specimen measurement technique indicates the presence of wax in brown fiber and provides a PPM number for those objects extracted from the background as wax. The PPM shifts proportionally with the concentration of wax. Further investigation will be required to calculate the coefficient of conversion from PPM to wax concentration at a particular level of object contrast sensitivity and to determine the level of repeatability.

Addendum A

Summary data for Test Series 1. Five sheets of each wax concentration were made and tested. All except the Virgin and the very high concentration of 0.1% were tested on both sides.

		Test Summary Data									
Test Descript. :		5 Shts 1 side		5 Shts 2 sides		5 Shts 2 sides		5 Shts 2 sides		5 Shts 1 side	
		Vrgin		0.0001% wax		0.001% wax		0.01% wax		0.1% wax	
Test Time :		Thu Sep 10 16:21:40 19		Thu Sep 10 14:40:30 19		Thu Sep 10 15:09:00 19		Thu Sep 10 15:24:20 19		Thu Sep 10 16:12:06 19	
Size Category	Total	Ave Area	Total	Ave Area	Total	Ave Area	Total	Ave Area	Total	Ave Area	
	>sq mm	<=sqmm	Count	sq mm	Count	sq mm	Count	sq mm	Count	sq mm	
0.06	0.07	6	0.084	12	0.065	19	0.065	121	0.068	399	0.068
0.07	0.08	3	0.077	5	0.074	13	0.074	102	0.075	361	0.075
0.08	0.09	1	0.084	8	0.087	12	0.086	134	0.085	446	0.085
0.09	0.1	1	0.091	9	0.095	7	0.095	98	0.095	302	0.095
0.1	0.15	5	0.121	20	0.124	57	0.121	398	0.123	1408	0.123
0.15	0.2	7	0.182	19	0.171	25	0.173	269	0.172	1038	0.174
0.2	0.25	3	0.226	6	0.227	22	0.223	180	0.223	788	0.225
0.25	0.3	1	0.281	6	0.271	19	0.278	143	0.275	631	0.271
0.3	0.4	1	0.308	6	0.338	18	0.343	145	0.348	1003	0.348
0.4	0.6	3	0.477	9	0.494	16	0.514	151	0.486	1181	0.485
0.6	0.8			6	0.720	2	0.772	65	0.685	665	0.690
0.8	1			3	0.827	3	0.916	27	0.880	409	0.892
1	1.5	1	1.163	4	1.294	3	1.093	19	1.142	633	1.216
1.5	2					2	1.832	5	1.704	287	1.714
2	3			1	2.44	2	2.52	7	2.24	240	2.39
3	10			1	4.61	4	5.44	6	4.36	212	4.52
10	20							1	12.10	1	11.39
20	30										
30	40										
40	100										
Totals		32		115		224		1871		10004	
Ave Sheet PPM		76.90		217.61		458.00		2,870.20		63,318.70	

% Wax Concentration	Side 1 Test Number					Side 2 Test Number					Average	Standard Deviation	Coeff of Variation
	1	2	3	4	5	1	2	3	4	5			
Virgin	171	70	55	57	31						77	54	70.80%
0.0001	239	109	162	292	184	91	121	216	498	334	219	112	51.32%
0.001	120	235	1097	424	290	312	253	892	647	309	458	318	69.51%
0.01	3123	4467	1986	1919	738	4072	3599	4359	3241	1198	2870	1332	46.42%
0.1	19969	125994	57521	60308	52801						63319	38603	60.97%

Figure 5: PPM data from each side of five handsheets handsheets. Only one side of the handsheet was tested at the extremes of virgin and 0.1% concentration.