Quantification of block testing for coated paper substrates

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ABSTRACT: Block resistance is a critical property for coated paper and board substrate that will be rolled, stacked, or otherwise contact itself after coating. Small differences in the coated substrate's blocking can determine whether the substrate can be successfully used for its designated purpose. However, this crucial property is typically evaluated using a qualitative scale that is based on subjective operator ratings and impacted by factors that include: (1) sound of coated substrate during separation, and (2) force with which substrates are separated.

This paper tests the hypothesis that quantifying the block test by measuring the force required to peel samples apart improves the test by: (1) providing more standardized testing conditions by controlling peel force and rate; (2) more clearly differentiating samples that experience minimal to some blocking; and (3) maintaining customizability to evaluate customer-specific test conditions. The method developed in this study uses a standard block tester and block testing conditions, but it peels the coated paper samples using a hot tack/heat seal instrument with force measurement capabilities. This paper demonstrates, using the instrument's heat seal capabilities, that it can measure peel forces that represent the full range of observable block scores.

The efficacy of this method was evaluated by having a group of trained operators engage in a randomized, blind experiment where they assessed block resistance on a set of coated paper samples using a modified qualitative block scale and compared their results to force measurements collected using the proposed method. The sample set included two coatings that have successfully run in commercial trials with minimal blocking, and one coating that experienced significant blocking in commercial trials despite only exhibiting some blocking at standard block test conditions in laboratory testing. The quantitative test method presented in this paper clearly differentiated these samples, whereas the qualitative assessment could not predict which samples had suitable block resistance for commercial use. As any tensile tester capable of measuring with 0.1 N resolution can be used for the Quantitative Block Test, the proposed method can be widely adopted. Furthermore, this method can be used for any block condition.

Application: The development of new coatings for paper packaging requires robust and quantitative methods for performance evaluation. This paper reviews methods for evaluating coating block and describes a way to quantify block using force measurements.

As demand for paper-based packaging grows, waterborne barrier coatings offer an attractive solution to impart functional properties to paper substrates using an array of traditional and innovative coating methods. Many of these coating methods require the coated paper to be wound back into rolls after drying, often under elevated temperature and pressure conditions that increase the likelihood of blocking (coated paper sticking to itself) [1]. For applications that require double sided coating (e.g., sandwich wraps, bakery liners, etc.), even moderate blocking can lead to failure of the coating or tearing of paper fibers. In cases of significant blocking, entire rolls of coated paper can be irreversibly adhered together, rendering them useless and resulting in wasted material and coating line time.

Current standard methods for measuring block resistance rely on operators separating coated substrates that have been subjected to elevated temperature and pressure over time and assigning a qualitative rating or score. While these methods describe standard conditions at which coated samples should be pressed together, problems arise due to the subjective nature of these qualitative ratings or scores [1,2]. Block scores can vary based on operator perception, slight variations in the force used to separate the coated substrates, and the rate at which they are separated [3].

The former TAPPI Standard Test Method T 477 "Blocking resistance of paper and flexible materials" and the current ASTM D918-99 "Standard test method for blocking resistance of paper and paperboard" describe stacking four coated substrates together such that each stack contains one replicate of coated face-to-coated face, one replicate of coated face-to-uncoated face, and one replicate of uncoated face-to-uncoated face. The stack is then exposed to test conditions varying from 38°C–60°C and 44%–75% relative humidity (RH) at 0.5 psi for 21 ± 1 h. **Table I** contains the complete list of test conditions. After allowing the samples to cool for 30 min, the substrates are separated from each other and evaluated using the descriptions in **Table II**.

One modification of this Block Test method (samples evaluated either coated face-to-coated face or coated face-

Temperature, °C	Relative Humidity, %	Pressure, psi
38	75	0.5
49	75	0.5
60	75	0.5
38	44	0.5
49	44	0.5
60	44	0.5

I. Test conditions for ASTM D918 "Standard test method for blocking resistance of paper and paperboard."

to-uncoated face at 60°C and 50 psi for 1 h) provides a higher degree of distinction and has been shown to provide better alignment to industry experiences. **Table III** describes the 1–5 rating scale.

This method has provided more distinction among samples that remain visually undamaged after separation, but it is still inherently a qualitative evaluation and thus subjective to the individual operator. It then becomes important not only to consider the block score of a sample, but also the operator conducting the test. Furthermore, the portion of this scale that most correlates with block resistance on a production line is in the range that is most subjective. In efforts to continually improve the block resistance of barrier coating offerings, a coating was developed that outperformed its predecessor with a block score of 2 in customer trials, but still required some force to pull apart. The differentiating factor with this coating was that it was reproducibly easier to separate and made less sound during separation. To distinguish between these two results, the new material was given a block score of 1.5. However, this example highlighted the need for a more quantitative block testing method that utilizes consistent, measurable, and reproducible metrics to represent a material's block resistance.

Test methods to measure adhesive strength of coatings are commonly used in the field of peelable coatings [4], such as labels [5]. These methods include peel testing [6], microindentation [7], atomic force microscopy [8], and many others [4]. These test methods, however, often look to distinguish coatings where adhesion is desired, and as such, are often performed at temperatures far above the glass transition temperature of the coating. Measurements for block resistance of paper coatings, on the other hand, often occur at temperatures far closer to the glass transition temperature of the coating, making sample preparation and measurement conditions of particular concern [2]. As such, quantitative methods for measuring block resistance of paper coatings, or similar acrylic latexes, use methods that allow for standardized conditions, including rheometers [1], universal testers [2], and applied current in an electromagnet [9].

This paper presents a method by which the block scale can be quantified after using the same block tester used

Degree of Blocking Resistance	Description of Blocking
No blocking	No adhesion or cohesion between contiguous surfaces that slide freely upon one another. Surfaces of specimens are not marred when sheets are separated.
Slight blocking	Slight adhesion where adjacent surfaces do not slide freely. Upon separation of the sheets, the surfaces show occasional evidence of slight marring.
Considerable blocking	Adhesion or cohesion of contiguous surfaces where layers may be separated with difficulty. Surfaces will definitely be marred or partially destroyed upon separation.

II. Descriptive block rating scale for ASTM D918 "Standard test method for blocking resistance of paper and paperboard."

Block Score	Description of Blocking
1	Substrates separate freely/without any force.
2	Substrates separate easily, but a "sticking" sound is observable.
3	Substrates require force to separate, equivalent to a sticky note.
4	Substrates require force to separate; some fiber tear occurs upon separation.
5	Substrates require significant force to separate; significant fiber tear occurs upon separation.

III. Block score descriptions for the Modified Block Test.

in the current standards. The described method results in improved resolution in the block resistance regime of highest interest and replaces the operator-based subjective rating system.

MATERIALS AND METHODS

Coatings of five different barrier dispersion samples representing one to two compositions of three different chemistry types were prepared via hand drawdown to a dry coat weight of 6 or 10 g/m² on freesheet paper (basis weight: 58 g/m², PPS roughness: 4.8 µm, Gurley porosity: 25 s). The coated papers were dried in an oven for 2 min at either 80°C or 110°C.

ASTM block test

Coated papers were placed in a coated face-to-coated face configuration. Then, 0.5 psi of pressure was applied, and the samples were placed in a 60°C oven for 21 h. After the coated papers cooled, they were pulled apart, visually inspected, and given a block score based on the description in Table II. Tests were performed in triplicate.

Modified Block Test

Coated papers were placed in a coated face-to-coated face configuration. Then, 50 psi of pressure was applied, and the samples were placed in a 60°C oven for 1 h. After the coated papers cooled, they were pulled apart and given a block score based on the description in Table III. Tests were performed in triplicate.

Development of Quantitative Block Test

Coated paper samples were cut into strips that were 1 in. wide by approximately 12 in. long. These strips were loaded into the heat seal/hot tack tester and sealed at a pressure of 100 psi and temperatures ranging from 50°C to 190°C for 2 s. The increased pressure (100 psi) was chosen to offset the short test time of 2 s and ensure the temperature range resulted in block scores across the full range of the scale (1–5). After sealing, the samples were allowed to cool for 2 s with a blast of air and one of two peeling methods were used: (1) sealed samples were taken off the heat seal/hot tack tester, pulled manually, and given a block score according to Table III; and (2) sealed samples were immediately pulled on the heat seal/hot tack tester at a rate of 5 cm/s, the peak load peel force (N) was measured using the integrated load cell, and the sealing temperatures that yielded fiber tear were noted.

Quantitative Block Test

Samples exposed to the Modified Block Test conditions (50 psi, 60°C, 1 h) were loaded into the heat seal/hot tack tester and peeled at a rate of 5 cm/s. The peak load force (N) was reported. Tests were performed in triplicate.

An adaptable version of the Standard Operating Procedure for the Quantitative Block Test is described as follows:

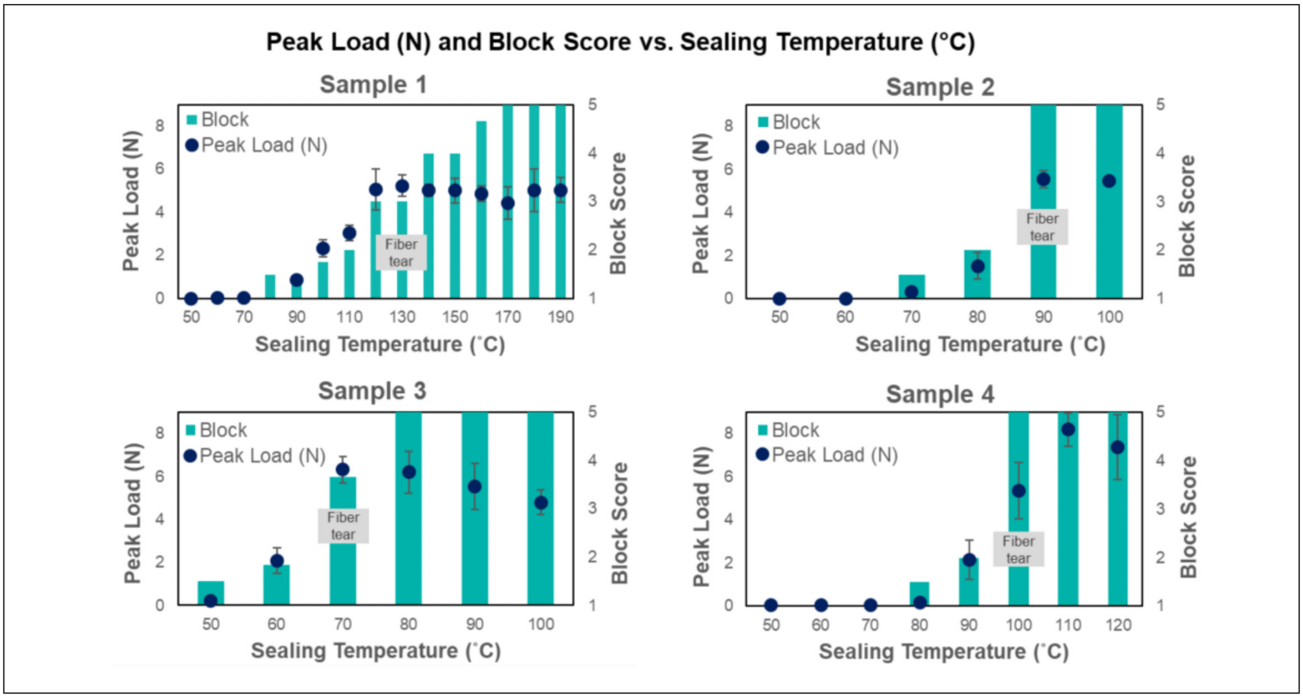
1. Heat oven to 60°C (or desired temperature).
2. Cut three samples of each coated substrate to 2 in. wide x 12 in. long strips (or other dimensions to best fit block testing and peel force measurement instruments).
3. Fold each sample strip in half so that the coated sides are facing one another (coated side facing inward).
4. Place folded strips in block testing apparatus and adjust spring to 50 psi.
5. Place block testing apparatus with samples into oven for 1 h (or desired time).
6. Allow block testing apparatus to cool until safe to touch, then remove samples and allow them to cool to room temperature (23°C).
7. Load samples into hot tack/heat seal tester and measure peel force.
8. Results are reported in force measurements (N); lower peel forces correlate to better block resistance.

RESULTS AND DISCUSSION

Method development

To successfully quantify the outlined Modified Block Test method, a correlation between the block scores assigned to manual pulls and the measured peel force must be established. Initially, samples were subjected to a temperature sealing ladder, then removed from the hot tack/heat seal instrument and pulled/assigned manually by a single operator to ensure that the full range of block performance (i.e., no blocking/block = 1 to considerable blocking/block = 5) for each binder was captured and evaluated. The identified temperature ranges were then used to conduct temperature ladders where both sealing and peel force measurement were subsequently conducted by the hot tack/heat seal instrument. The blue data points in **Fig. 1** highlight the response curves for each of the four different coatings, with Sample 1 exhibiting a more gradual increase in peak load peel force as temperature rises and Samples 2-4 exhibiting a sharper peak load peel force transition with temperature increase. Teal bars represent the manually assigned block score for each sealing temperature. A clear correlation exists between peak load peel force and block score, with higher block scores resulting in high peak load peel forces and lower block scores resulting in lower peak load peel forces. The conditions with the most noticeable difference between measured peak load peel force and manually assigned block score are those in the transition from good block resistance to poor block resistance (block score 2–4). This aligns with earlier observations that the subjective nature of manually assigning block scores in this range makes it difficult to accurately describe block resistance with the Modified Block Test alone.

Gray boxes indicate the condition at which fiber tear was first observed during substrate separation when measuring peel force. While Samples 2–4 first exhibit fiber tear at a seal temperature with a noticeably higher peak load peel force than the previous temperature and a Modified Block



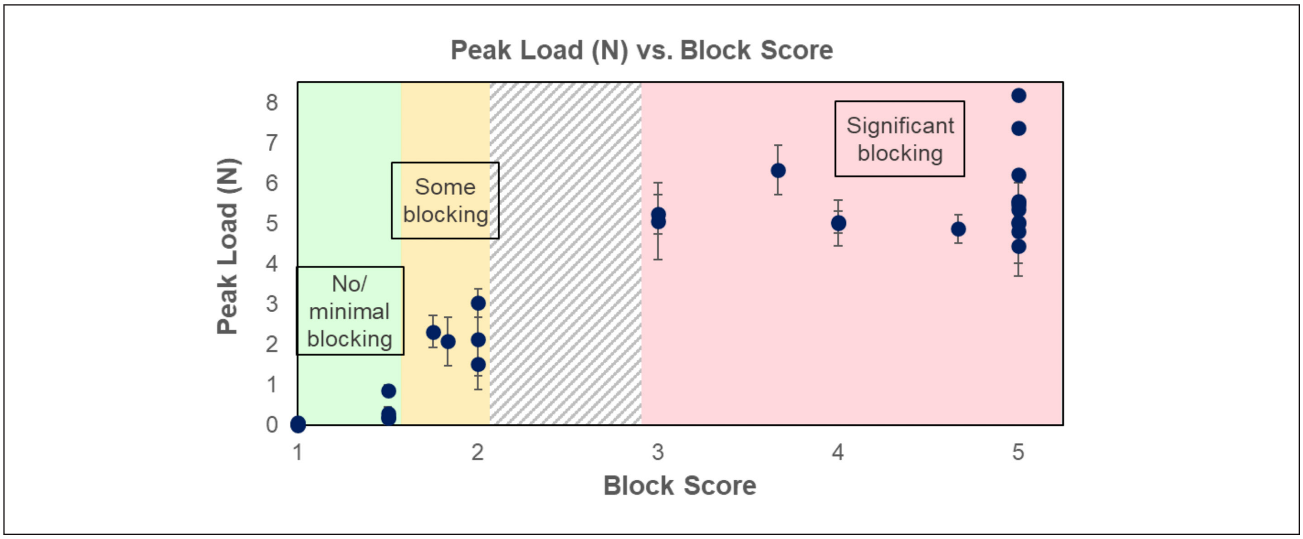
1. Peak load peel force (N) measured during separation of substrates at a 90° angle (blue points) and manually assigned block score (teal bars) as a function of sealing temperature for four different waterborne latex coatings. Gray boxes note the temperature at which fiber tear occurred during substrate separation when measuring peel force. Error bars represent the standard deviation of three replicates.

score where fiber tear is typically observed (block score = 4–5), the peak load peel force and block score at which Sample 1 first exhibits fiber tear is almost identical to that at the preceding temperature where no fiber tear is observed. This is likely a function of the gradual increase in required peel force as a function of temperature displayed by Sample 1, as a 10°C temperature increase only changes the blocking enough to induce fiber tear, but not enough to significantly increase the measured peak load peel force. These data display a clear relationship between manually assigned block scores and measured peak load peel forces that can be leveraged to quantify our current Modified Block Test method.

Peak load peel force measurements and manually assigned (by a single operator) block scores were plotted against one another without specific coating information to evaluate whether the relationship between the two metrics had any dependence on the coating identity (**Fig. 2**). The resultant graph suggests that the correlation is coating-agnostic, and across all materials evaluated, low peak loads align with low block scores and vice versa. The plot highlights the range of peak load peel forces (1.5–3.0 N) measured for samples with block scores around 2, in a way quantifying the difficulty of comparing two materials that both exhibit "slight blocking" but have small noticeable and reproducible differences in block resistance. While there is not sufficient resolution in the data to differentiate between a "loud, sticky" block 2 from a "quiet" block 2, a pairwise student's *t*-test identified statistically significant differences between three district block ranges:

1. **No/Low Block:** Peak load peel force < 1 N; average block score 1–1.5; samples are not adhered to each other at all or are so lightly adhered that they separate with minimal force and minimal sound. These materials are ideal for coaters/converters without formulation capabilities who will use a coating as supplied.
2. **Moderate Block:** Peak load peel force 1–3.5 N; average block score 1.75–2; samples are adhered to one another and require some force to separate but can be easily separated without damage to the coating or the substrate. These materials can often be utilized by customers with formulation capabilities and expertise to further improve block resistance prior to use.
3. **Significant Block:** Peak load peel force ≥ 4 N; average block score 3–5; samples are adhered to one another and require significant force to separate. Separation often causes damage to the coating itself, fiber tear of the substrate, or both. These materials present a challenge for unwinding rolls of coated substrate and unstacking coated cups, bowls, etc.

The range of peak load peel forces measured for samples in the Significant Block regime is quite wide, 4.4–8.2 N; however, this regime does not need further resolution, as



2. Peak load (N) measured during separation of substrates at a 90° angle as a function of block score (measured by manual pull from a single operator) for four different waterborne latex coatings. Error bars represent the standard deviation of three replicates. Color-highlighted regions indicate subsets of block scores that can be differentiated from one another with 95% confidence based on a pairwise student's t-test. Green: average block score 1–1.5 (peak load < 1 N); yellow: average block score 1.75–2 (peak load 1–3.5 N); red: average block score 3–5 (peak load ≥ 4 N). The area with gray lines does not contain any data points and therefore was not included in either the 1.75–2 or 3–5 block score regimes.

materials with this block performance are not likely to be considered for commercialization/end-use until the block resistance improves, at which point it moves into one of the other more highly-resolved regimes.

Method validation

With the effectiveness of the Quantitative Block Test method verified, coated-to-coated block results of Samples 1–4 were compared using all three methods described previously (**Table IV**). In this case, the Quantitative Block Test method was performed on coated paper samples that were subjected to the Modified Block Test conditions (60°C, 50 psi, 1 h). Under the ASTM D918 test conditions and rating system, none of the four samples demonstrated significant blocking.

The Modified Block Test method demonstrates the limitations of the ASTM method. Sample 3 exhibits the largest performance difference between the two methods by experiencing only slight blocking under the ASTM conditions, but full blocking with fiber tear under the Modified Block Test conditions. Additionally, Samples 1 and 2 differentiate themselves from Sample 4 by having a block score of 1.5 (minimal force to separate) vs. 1 (substrates separate without force). Samples 1 and 2 were far more difficult to differentiate using the Modified Block Test. Both required some minimal force to separate, but Sample 2 was louder than Sample 1 during the peel.

The Quantitative Block Test method removes ambiguity present in the other two block methods. The measured peak load force more clearly shows the difference between Samples 1, 2, and 4. Moreover, not only does Sample 3 exhibit a higher peak load force than the other three samples, the degree of fiber tear also matches that of the Modified Block Test. An additional benefit of using a heat seal/hot tack tester or other tensile tester to quantify block resistance is that it can be applied to any block condition. If a particular set of block testing conditions correlates to those on a given production line, samples can be evaluated using

Sample	ASTM D-918 Block Rating (60°C, 0.5 psi, 21 h)	Modified Block Test Score (60°C, 50 psi, 1 h)	Quantitative Block Test Peak Load, N (60°C, 50 psi, 1 h)
1	No blocking	1.5	0.12 ± 0.046
2	Slight blocking	1.5 (louder)	0.29 ± 0.142
3	Slight blocking	5*	5.82 ± 0.290*
4	No blocking	1	0.05 ± 0.000

IV. Comparison of coated-to-coated block testing results from the three methods discussed in this paper: ASTM D-918; an internally used modification of D-918; and the newly developed Quantitative Block Test (* indicates fiber tear).

Total Sample 1 pulls	18	%
Scored as 1.5	7	38.9
Scored as 2 but differentiated from Sample 5	4	22.2
Total Sample 1 differentiated from Sample 5	11	61.1

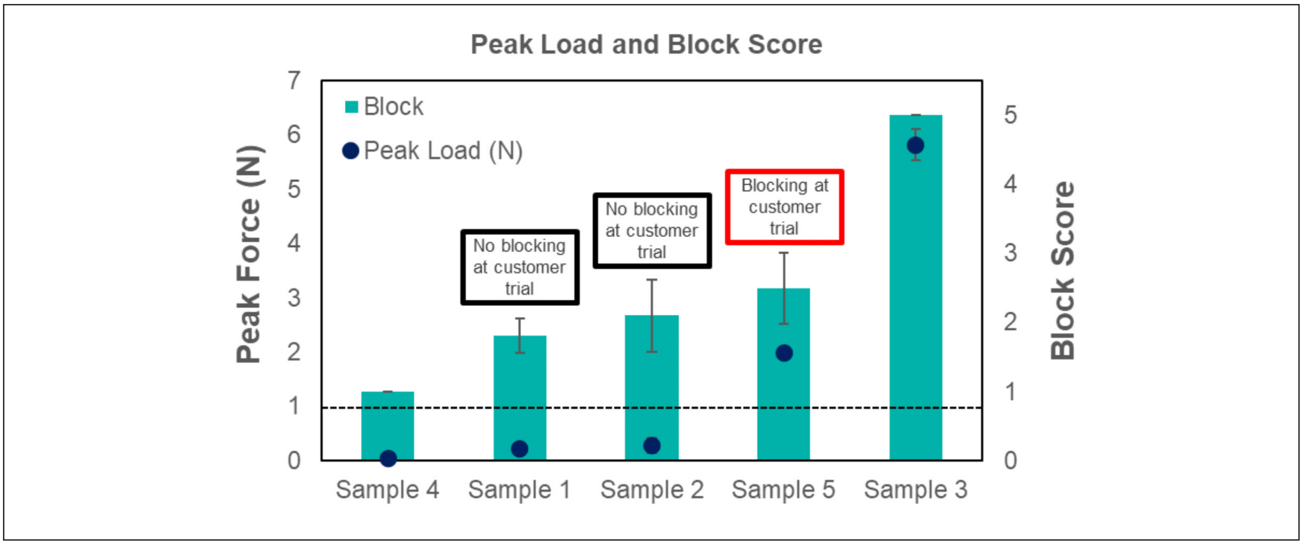
Operator	Sample	Block Score	Operator	Sample	Block Score
A	Sample 5	3	D	Sample 1	1.5
A	Sample 1	2	D	Sample 5	2
A	Sample 1	2	D	Sample 5	2
A	Sample 5	3	D	Sample 5	2
A	Sample 5	3	D	Sample 1	2
A	Sample 1	2	D	Sample 1	1.5
B	Sample 1	2	E	Sample 5	3
B	Sample 1	2	E	Sample 5	2
B	Sample 5	2	E	Sample 1	1.5
B	Sample 1	2	E	Sample 1	1.5
B	Sample 5	2	E	Sample 5	2
B	Sample 5	2	E	Sample 1	2
C	Sample 1	1.5	F	Sample 1	1.5
C	Sample 1	2	F	Sample 1	1.5
C	Sample 5	3	F	Sample 5	3
C	Sample 5	3	F	Sample 5	3
C	Sample 1	2	F	Sample 5	3
C	Sample 5	2	F	Sample 1	2

V. Results of a blind, randomized study comparing block resistance of Sample 1 to Sample 5 using the Modified Block Test. Six different trained operators are represented with letters A-F. Green results highlight replicates of Sample 1 that were rated with a block score of 1.5 (the intermediate block score added to the scale to represent Sample 1's performance), and yellow results highlight replicates that were rated with a block score of 2, but were still differentiated from Sample 5 (scored with a 3 when typically given a block score of 2).

those conditions, a tensile tester, and the same distinctions presented here.

To validate the Quantitative Block Test method, the example described in the introduction was revisited and the materials were evaluated head-to-head in both the Modified Block Test and the Quantitative Block Test. Sample 5 represents a first-generation material that resulted in significant blocking during a customer trial, and Sample 1 represents the next-generation material with improved block resistance that has run successfully during customer trials. As described earlier, Sample 5 is typically rated with a block score of 2. During development, Sample 1's block resistance was noticeably better than Sample 5's, but coated sheets of paper did not separate on their own, which is the metric to achieve a block score of 1. Thus, Sample 1 was given a block score of 1.5. **Table V** highlights the results of a blind study with six trained operators conducting the Modified Block Test on a randomized set of Samples 1 and 5. Operators only assigned Sample 1 a block score of 1.5 (the intermediate score that was added to the block scale to describe Sample 1's block resistance during development) in 39% of tests. In an additional 22% of tests, Sample 1 was assigned a block score of 2, but Sample 5 was assigned a block score of 3, highlighting cases where operators did not assign the "correct" block score to each sample but were still able to differentiate between block resistance of the two samples. Taken together, operators were only able to identify block performance differences between Sample 1 and Sample 5 61% of the time, despite the critical difference observed in coating line performance.

Samples 1 and 5, along with Samples 2, 3, and 4 from earlier studies, were then evaluated using the Quantitative Block Test. **Figure 3** shows peak load peel force (N) values measured during Quantitative Block Testing compared to Modified Block Test scores for each of the five samples. Samples 1, 2, and 5 have similar block scores and notice-



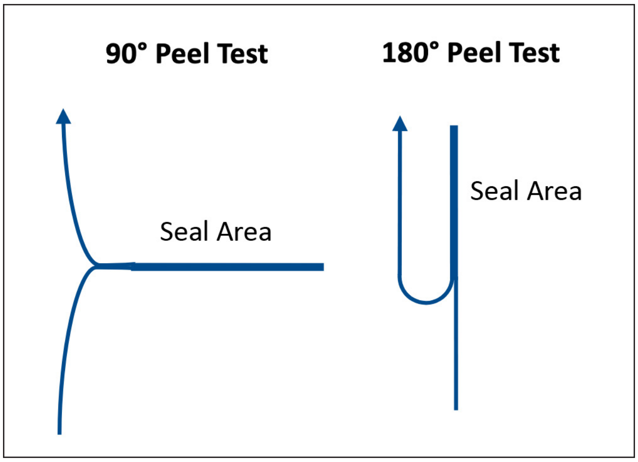
3. Peak load peel force (N) measured during separation of substrates at a 90° angle (blue points) and manually assigned block scores (teal bars) for five different waterborne latex coatings. Text boxes include important performance distinctions reported in customer trials. The black dotted line represents the recommended "pass" threshold for the Quantitative Block Test (<1 N peak load). Block scores were collected by six trained operators, with each operator assigning scores to 2–3 replicates for each sample in randomized order. Error bars represent the standard deviation of three replicates for measured peak loads and 12–18 replicates for block scores.

ably more variation compared to Samples 3 and 4 when evaluated using the Modified Block Test, despite critically important block resistance differences in customer trials. These results highlight both the importance of reliably and confidently identifying subtle performance differences during product development and the shortcomings of the Modified Block Test. In contrast, the peak load peel forces for Samples 1 and 5 are significantly different from one another, making it easy to differentiate between the two samples. Additionally, the recommended threshold for no/low blocking outlined in the Method Development section, a peak load peel force <1 N, is met for Sample 1 but not for Sample 5, again providing a quantitative distinction between the two materials.

Additional considerations

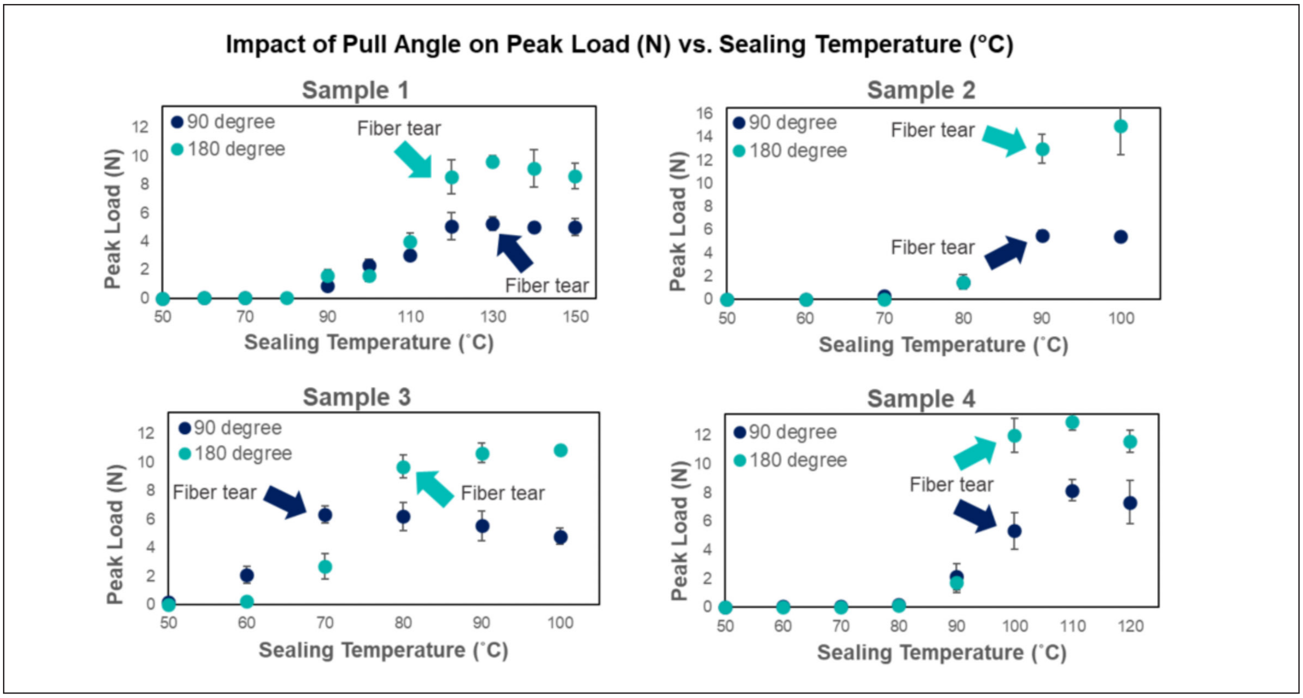
When implementing the Quantitative Block Test across labs with different available equipment, there are considerations that must be taken to ensure optimal performance. One such consideration is the relatively low peel forces exhibited by samples with low block scores. Heat seal/hot tack testers with integrated load cells are ideal for this sort of measurement, because the load cells are designed to accurately measure the force range of interest. Other kinds of tensile testers could work just as well for the peeling portion of this test, but particular attention should be paid to the sensitivity of the load cell (target resolution < 0.1 N).

Another potential difference between experimental setups is the geometry of the peel test. The samples in the study above were pulled with a 90° peel test in which the angle between the direction of peel and the seal is 90° (**Fig. 4**). Other experimental setups may be designed in a 180° peel test configuration in which the angle between the direction of peel and the seal is 180° (Fig. 3).



4. Schematic of the difference between a 90° peel test and a 180° peel test configuration.

The effect of peel orientation was examined using our Modified Block Test method (**Fig. 5**). For all samples, the largest difference between a 90° and 180° peel orientation came during the transition from no sealing to full sealing. A 180° peel orientation demonstrated higher peel forces when the samples experienced significant block to the point of fiber tear. The temperature at which fiber tear occurred either was identical between the two configurations or only changed by 10°C. Most importantly, however, the peel forces in the region of the sealing curves where the samples experienced little block were similar regardless of orientation. This demonstrates the effectiveness of using peel force to measure block, as two common tensile test



5. Peak load (N) measured during separation of substrates at a 90° (blue) and a 180° (teal) angle as a function of sealing temperature for four different waterborne latex coatings. Error bars represent the standard deviation of three replicates.

Sample 1		
Pull Speed, m/s	Peak Force	Standard Deviation
0.002	0.07	0.006
0.050	0.07	0.006
0.300	0.05	0.010
0.590	0.07	0.012

VI. Measured peak load peel forces (N) for Sample 1 as a function of peel rate. Each sample was evaluated in triplicate.

orientations allow for differentiation between samples of borderline block resistance. The impact of peel rate on the measured peak load peel force (N) was also evaluated and found to have no impact (**Table VI**). These findings demonstrate the versatility of the Quantitative Block Test method, as changes in peel orientation and rate do not appreciably change peak load peel forces within the no-to-low blocking regime.

CONCLUSIONS

This paper details a method for quantitative block testing of coated paper substrates using a hot tack/heat seal instrument with force measurement resolution as low as 0.1 N. After demonstrating that this instrument is capable of measuring peel forces that represent the full range of block scores, it was found that the force that represents each block score is independent of polymer chemistry. With the force measurements standardized, the results of this quantitative method were compared against the qualitative Modified Block Test in a head-to-head study where a group of six trained researchers engaged in a randomized, blind experiment where they measured the block resistance of a set of coated paper samples that experienced a range of block resistance in a commercial trial using the qualitative Modified Block Scale. While the Quantitative Block Test method was able to clearly differentiate these samples, the qualitative assessment could not predict which samples had suitable block resistance. Since any tensile tester with measurement resolution of 0.1 N can be used for the Quantitative Block Test, this method can be widely adopted. Furthermore, this method can be used to evaluate samples at any set of block conditions. If a particular set of block testing conditions correlates to those on a given production line, samples can be evaluated using those conditions, a tensile tester, and the same distinctions presented here. **TJ**

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ABOUT THE AUTHORS

Test method improvement and quantification is critical to evaluation of experimental candidates for product development. The improved block test method introduced here provides a more quantitative means of evaluating a key performance metric based on the amount of force required to separate pieces of coated substrate.

The most difficult aspect of this research was the need to measure samples that had been exposed to test conditions outside of the equipment to enable pairing with existing block test conditions. We addressed this by creatively modifying the procedure and generating templates to ensure correct sample insertion into equipment.

Through this study we learned how to operate a new piece of equipment and how to modify it to evaluate related but different properties from its targeted use. The most interesting finding was the correlation between qualitative block scores and quantitative force measurements across multiple different coating chemistries.

Mills may be able to correlate their qualitative, but ultimately most critical to end-use, block performance data from winding and unwinding rolls of coated substrate to the quantitative rating scale proposed in the manuscript. This would provide the R&D community a consistent, quantitative metric for acceptable anti-block performance.

This test method is also currently compatible with flexible substrates due to the sample loading mechanism. We are currently working towards an equipment modification that would enable compatibility with thicker substrates such as paperboard.

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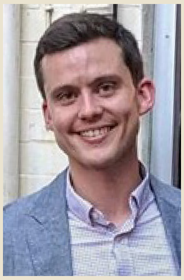

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