Effect of pressure and time on water absorption of coated paperboard based on a modified Cobb test method

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ABSTRACT: This manuscript presents the study of water absorption by paperboard subjected to water at high hydrostatic pressure based on a modified Cobb tester. The new tester is based on TAPPI Standard Test Method T 441; however, the water column can reach up to 550 mm.

The evaluation consisted of measurements of water absorption for coated and uncoated paperboard at different exposure times from 5 s to 45 s and water column heights from 10 mm to 500 mm (corresponding to hydrostatic pressures 98 Pa and 4.9 kPa, respectively). The coatings were formulated as a combination of styrene acrylate (SA; two binder levels) and two types of ground calcium carbonates (differing particle sizes) to form the two pre-coating structures: open and closed. The coating weight was 6 g/m^2 applied on 210 g/m^2 solid bleached board (SBB). In addition, 210 g/m^2 uncoated boards were studied. Characterization of the coatings was performed with scanning electron microscopy (SEM), mercury intrusion, and roughness.

It was found that the new device properly mimics the conditions of the current Cobb tester. The characterization of the coating also confirmed the presence of more open/larger pores of open coatings, confirming the desired coating structure. The absorption of boards was mainly driven by exposure pressure by comparing with exposure time. This was already evident after shorter periods of exposure time at 5 s and also 15 s exposure time. Paperboards with open coatings showed slightly higher absorption than other boards.

Application: Within its limitations, the proposed new testing device offers an easy way to study the water absorption of coated paperboards subjected to high external pressure.

Water transport through fibrous networks like paperboard and paper is a complex phenomenon that is driven, in part, by their intrinsic properties [1]. Capillary forces induced by the liquid menisci between porous walls have been found as one of the driving factors for liquid absorption in porous material [2-4]. As with the fibrous base stock, the liquids move through the coating layer of the coated boards due to capillary forces. The capillary pressure is influenced by the capillary radius, the liquid surface tension, the contact angle of the liquid meniscus, and length of the capillary channel [5-9]. The capillary forces increase with narrow channels, and many small pores are preferrable to few larger pores for capillary absorption. Factors such as pigment type, particle size and shape, and binder concentration can significantly influence the pore structure and thus the water absorption of coated paperboard [10]. As the penetration through the coating layer reaches the paper substrate, the voids in the substrate are filled with liquid. At this point, the liquid begins to be absorbed by the substrate fibers. This action causes the expansion of the fibers and therefore a reduction of the void for liquid motion [11].

Paperboard absorption can also be driven by external forces [12]. This can be observed, for instance, when dispersion coatings are applied on substrates. In this scenario, the absorption is increased due to the pressure at which the substrate is subjected by blade, rod, application rolls, or other elements of the coater machine [11,13]. A similar situation is observed when printing ink is applied on coated paperboard by printing press [14]. In other applications, the effect of pressure on absorption might also be of importance, particularly when the material is exposed for a long time to liquids. It has been experimentally proven that external pressure can be decisive in the water absorption of uncoated and coated boards. In fact, if the external pressure is large enough, it can have a larger influence than the capillary action [12]. Theoretical approaches have been reported to predict the effect of external pressure on the absorption external pressure [14,15]. Pressure driven absorption is favored by a pore structure with few large pores.

Traditionally, water absorption of uncoated or coated paper, paperboard, or corrugated board has been determined by the Cobb test. The Cobb test measures the amount of water absorbed by a defined surface area of a sample (quoted in g/m^2) in a set period of time, which is usually 60, 120, or 1800 s and known as $Cobb_{60}$, $Cobb_{120}$, or $Cobb_{1800}$, respectively, as measured by TAPPI Standard Test Method T 441 om-98 “Water absorptiveness of sized (non-bibulous) paper, paperboard, and corrugated fiberboard (Cobb test).” The applied period of time includes both the time a sample has been exposed to water and the time needed to handle the sample after the exposure to water. Usually, the handling time is about 15 s. For example, for the $Cobb_{60}$, the sample will be directly exposed to water of the hydrostatic pressure for a period of 45 s, (45 + 15 = 60 s), while for Cobb 120, the sample will be directly exposed to water for a period of 105 s (105 + 15 = 120 s).

The Cobb test is characterized by its simplicity and ease of implementation. The only pieces of equipment needed are a scale, drier roll, stopwatch, and the tester itself. However, as the standard has been defined, it is only possible to study the effect a minimum water column height of 10 mm (corresponding to 98 Pa; calculated according to $P = \rho g h$) [16], limiting the analysis of the impact of the pressure on the board absorption in relation to, for example,the coating process. The objective of this investigation was not to design a tool for replacing the standard Cobb tester; rather, the goal was to create a tool to study the impact of the external pressure on the absorption of coated paperboards based on a new and simple absorption apparatus with a higher Cobb ring/column inspired from the standard Cobb tester. In this case, with the higher Cobb ring/column, the column of water can reach up to 550 mm of water height (corresponding to ~5.4 kPa, calculated according to $P = \rho g h$) [16]. While the column height required to achieve the actual pressure in a coating process would be several meters, this device is a first step in assessing the effects of the coating process and increased pressure on penetration. The advantage of such an approach is simplicity to measure and that no tool with pressurized chamber is needed to vary the applied pressure during water absorption.

MATERIALS AND METHODS

Description of substrates and pre-coatings

An uncoated solid bleached paperboard (SBB) of 210 g/m² from Iggesund Paperboard (Iggesund, Sweden) was used as a substrate. Different pre-coating formulations consisting of a combination (by changing pigment and binder ratios) of styrene acrylate (SA) and ground calcium carbonate (GCC) were applied to paperboard to provide controlled porosity of the surface. In order to obtain different pre-coating absorption properties, the formulations were designed to obtain pre-coatings with a more controlled porosity. These included those such as open pre-coating (more porous) and closed pre-coating (less porous) surfaces using GCC Hydrocarb® 60 (average particle size, PS, of 1.4 um) and Hydrocarb® 90 (average PS of 0.7 um), respectively, from Omya North America (Cincinnati, OH, USA). The coating compositions are given in **Table I**. In line with a traditional work of practice, using combinations of different fillers with different quantities of the binder, we were able to obtain coatings with different porosities. Pre-coating target weights were 6 g/m² and 14 g/m² for all pre-coating types. The application of pre-coatings was conducted using a pilot scale coater at Iggesund Paperboard. The coating was applied using a blade coater. The web speed was 300 m/min, and the drying was performed with an infrared (IR) dryer followed by air dryers. It is important to keep in mind that the intent of this work was not to investigate the performance of different coatings. Instead, the goal was to create substrates with different known porosities for the investigation of the impact of external pressure on water absorption associated with the coating process.

Coating Components	Open Pre-coating, pph	Closed Pre-coating, pph
Hydrocarb 60	100	—
Hydrocarb 90	—	100
Latex Dow XZ 94329,02	5	15
Solids content, %	64.8	65.7
Brookfield viscosity, cP	1440	1840

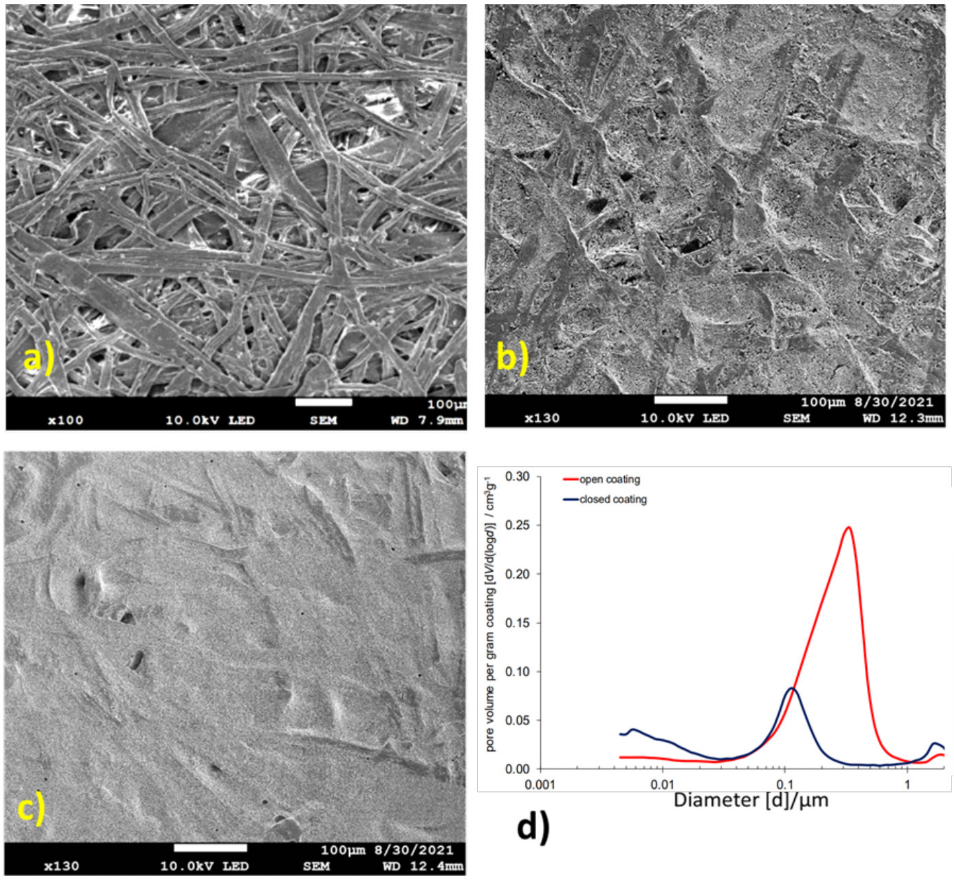
I. Coating composition. Solids content and viscosity tested at 22°C.

Characterization of the tested boards

Uncoated and coated boards were characterized for roughness, thickness, grammage, and contact angle of deionized water. Further, surface analysis based on scanning electron microscopy (SEM) was completed. Roughness (S10) was measured with a PPS Parker Print Surf 58-06 at 1,000 kPa of clamping pressure. The sample thickness was measured with an analogue dial caliper gauge COMPAC type 532G. The coating grammage was determined based on the difference in weight between uncoated and coated samples of a 100 cm² sample according to TAPPI T 410 om-98 "Grammage of paper and paperboard (weight per unit area)." All measurements were conducted in quintuplet.

Both porosity and pore size distribution were characterized using a Micromeritics Autopore V mercury porosimeter (Micromeritics; Norcross, GA, USA). The maximum applied pressure of mercury was 414 MPa, which is equivalent to a Laplace throat diameter of 0.004 µm [6]. For this testing, coating samples were applied to an impermeable substrate (aluminium foil). The drawdown method, as well as the interface between the coating and the foil, will result in some initial pore volume contribution over the large pore diameter range. The data for these samples have, therefore, been normalized at ~1.3 µm, and the resulting curves are shown in **Fig. 1d**.

In order to establish the presence of closed and open pre-coating, microscopic observation of the board surface was conducted with a Leo Gemini 1530 scanning electron microscope (Carl Zeiss AG; Oberkochen, Germany) at 10



1. Scanning electron microscope (SEM) images taken with (a-c) backscattering detector, and d) pore size distribution of the open and close pre-coating: a) 210 g/m² uncoated substrate; b) open pre-coating of 6.1 g/m²; and c) closed pre-coating of 6.6 g/m².

kV with secondary electron detection. Prior to observation, the samples were coated with a 20 nm layer of gold using a sputter coater.

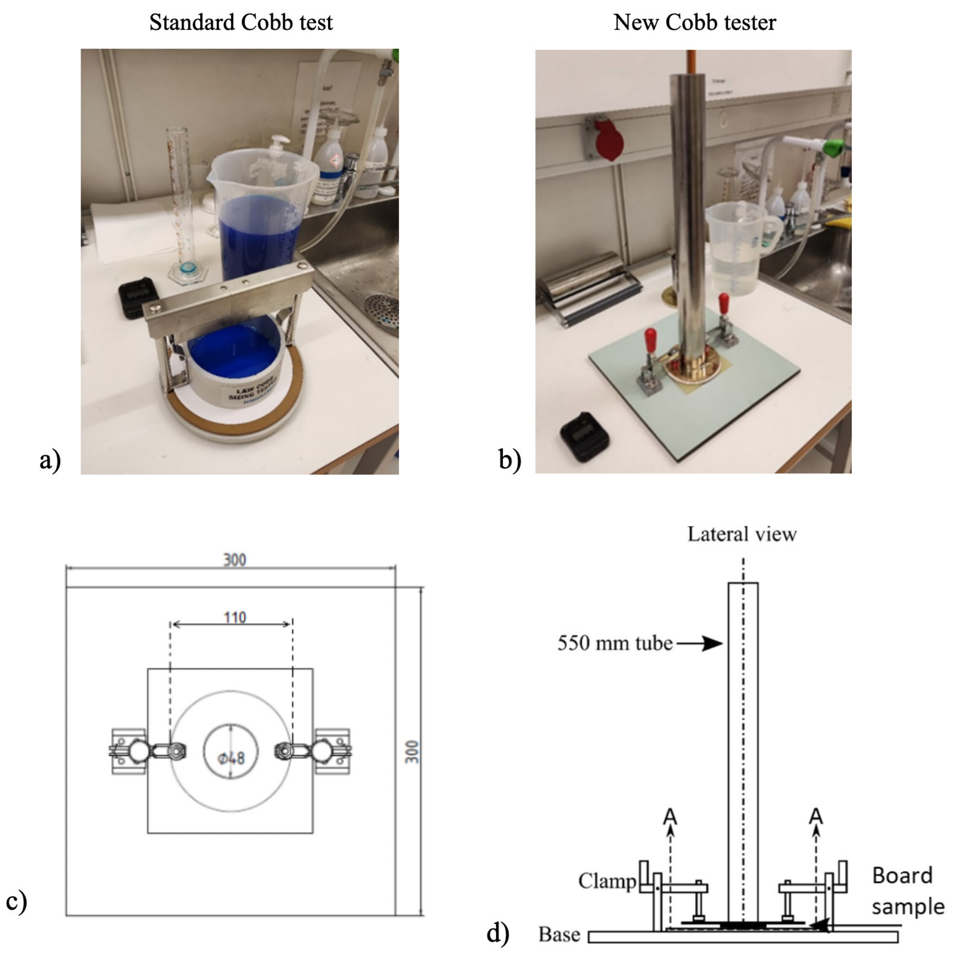
Description of the new Cobb tester

Pictures of the standard and new Cobb tester are shown in **Figs. 2a–2b**, respectively. Top and side view schematic illustrations of the new Cobb tester are presented in **Figs. 2c–2d**. Like the standard Cobb testers (used according to TAPPI T 441 om-98), a device that follows the same principle was proposed where a board sample is placed underneath a tube or ring that holds an amount of deionized water for a given time. The new Cobb tester consists of a stainless-steel tube of 50 mm in diameter and 550 mm in length with a 110-mm-dia. disk at the bottom side. The exposed sample size is 48 mm (internal tube diameter corresponding to a test area ~18 cm²). This disk has a 1-mm-high ring of 10-mm thickness that evenly distributes the pressure on the board to maintain the water on the surface of the exposed board area without affecting the structure of the board. The tube is maintained in place by two toggle clamps that are in contact with the 110-mm disk. The board samples are placed underneath the disk and on top of a rubber mat to avoid direct contact with the surface of the tester base/bottom plate. The base was made with a fiberboard plate of 300 mm × 300 mm that provides adequate stability to the tester.

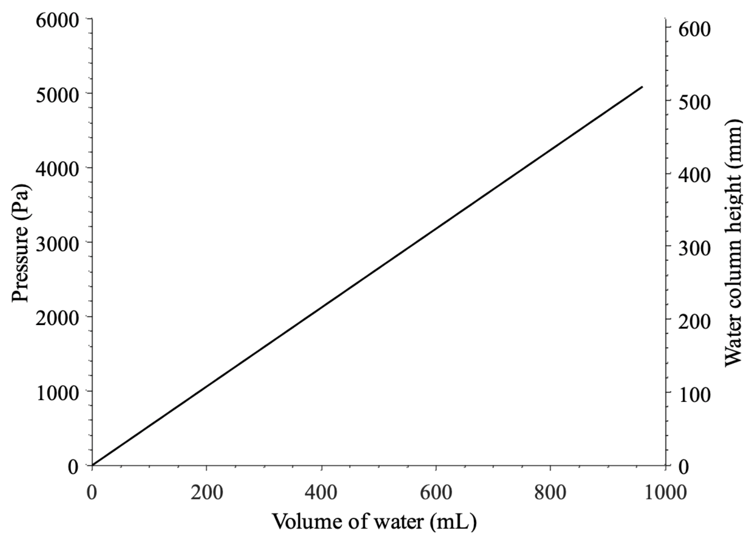
As the height of water column increases, the pressure on the boards increases proportionally. **Figure 3** shows the relationship between pressure, amount of water, and water column height of the new tester. With this new tester, it is possible to achieve up to 5.4 kPa of hydrostatic pressure when the test column is completely filled with water, compared to 98 Pa in the standard Cobb tester.

Measurement of absorption based on the new Cobb tester

The absorption measurements were conducted according to TAPPI T 441 om-98, with the exception that the board samples were exposed to different water column heights from 10 mm (standard Cobb tester corresponding to 98 Pa) to 500 mm (new Cobb tester corresponding to 49 KPa) during a period from 20 s to 60 s. It is important to note that 15 s are needed to take the samples from tester to conduct the



2. a) Standard Cobb tester with test area of 100 cm²; b) new Cobb tester with test area of 18 cm²; and (c-d) diagram of the new Cobb tester.



3. Relationship between column hydrostatic pressure, column height, and volume of water of the new Cobb tester.

Sample	Board Grammage, g/m²	Pre-coating Weight, g/m²	Thickness, mm	PPS Roughness S10, µm
210 g/m² base substrate (uncoated)	210 ± 1.0		0.26	7.3 ± 0.11
Open pre-coating (target coat weight 6 g/m²)		6.08 ± 0.52	0.26	5.3 ± 0.06
Closed pre-coating (target coat weight 6 g/m²)		6.58 ± 0.56	0.27	3.8 ± 0.14

II. Evaluated substrates and their characteristics.

measurements; therefore, the actual exposure time under pressure is 5 s and 45 s for $Cobb_{20}$ and $Cobb_{60}$ test, respectively. Prior to testing, the samples were conditioned for at least 24 h at 23°C and 50% relative humidity (RH), and testing was conducted at the same conditions. The average and standard deviation of water absorption of all the samples corresponding to each board type were calculated for analysis. As recommended by the TAPPI T 441 standard, samples were evaluated in quintuplet for all the measurements.

In order to prove that the new tester is comparable to the standard device, the $Cobb_{60}$ test was conducted under standardized conditions, meaning that the column height was 10 mm and the testing period was 60 s. These measurements were conducted for samples with both high and low water absorptivity with uncoated 210 g/m2 board and board coated with closed pre-coating (thick coating layer ~14 g/m²) board, respectively. A thick coating layer has been used to minimize the impact of the baseboard on the absorption process. In addition, $Cobb_{60}$ with a water column height of 500 mm was conducted with the roughest sample to evaluate the sealing between the ring and the board surface on the new tester. This scenario represents the highest pressure that the new tester can deliver with the least secure sealing conditions. This portion of the testing confirmed that the new tester could supply enough pressure on the board to maintain the water only on the studied board area without affecting the board structure. To visualize the spreading pattern of the water on the board substrate, methylene blue was used as a dye. The testing was conducted in quintuplet.

RESULTS AND DISCUSSION

Sample characterization

Details of the samples and their substrate characteristics are presented in **Table II**. As expected, the roughness of boards was higher for uncoated samples, reaching a PPS value over 7 µm. As expected, the coated boards showed lower roughness compared with uncoated boards, and the lowest roughness was achieved with closed pre-coatings. The same was evident from the SEM analysis of the substrates (Fig. 1).

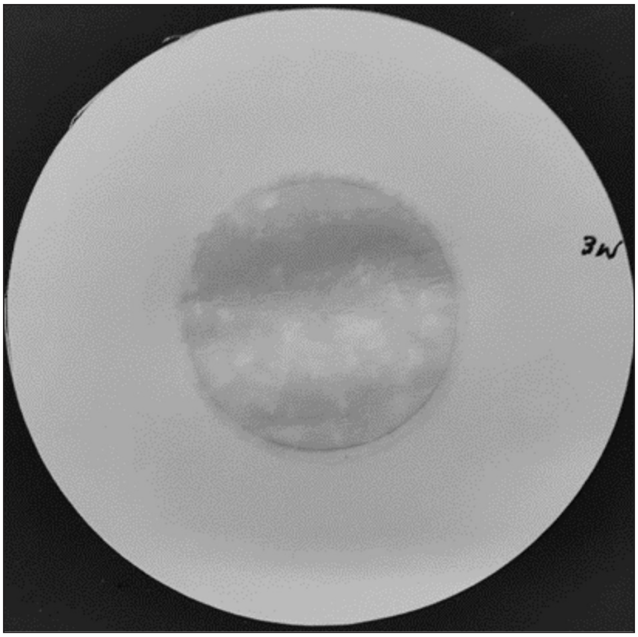
The uncoated 210 g/m² base substrate (Fig. 1a) had a quite rough and more open surface, while precoated substrates (Figs. 1b–1c) had a more closed and smoother surface. By comparing the precoated substrates, it is evident that closed pre-coating (Fig. 1c) provided a smoother and less porous surface than the substrate coated with open pre-coating (Fig. 1b). The open pre-coating had the presence of small voids in the surface. These spaces were well distributed over the entire coating surface. This observation can be correlated with the roughness measured for closed and open coating; however, this observation cannot be extrapolated to visually differentiate the roughness within each coating type. Interestingly, the closed coatings presented some small cavities; nevertheless, after further analysis with and without backscattering detector, it was concluded that the cavities were only superficial. Figure 1d shows the pore volume in form of mercury intrusion curves for the open and closed pre-coatings. The mercury intrusion data are indicative of the pore structure of the coated samples. The dominant pore size for the open pre-coat was 0.34 µm, while the dominant pore size for the closed pre-coat was at the finer pore diameter of 0.11 µm. The curve continues to rise at finer pore diameters, which may be due to the breakthrough (collapse) of the pigment structure by mercury or because there are finer pores in this coating.

Comparison of standard and new Cobb tester

Evaluation of sealing for new tester

Figure 4 shows the pattern after the dye after measurements with the new Cobb tester. The image shows that the ring had an adequate sealing, and no signs of leaking beyond the limits of the studied area were noted. In addition, no damage was observed on the board surface due to the pressure applied from the tester to keep water in place. The minimum indentations presented on samples after testing can be compared with those found after tests conducted in the conventional rig.

The comparison between the standard and the new tester was conducted under standardized $Cobb_{60}$ conditions (column height was 10 mm and the testing period was 60 s), and the results from the standard and the new tester showed that, on average, the results were the same (when comparing the standard and the new tester on uncoated samples and on coated samples, respectively); however, the variation of the results for coated samples (thick coating layer) was found slightly larger but within the same range for the new tester (**Fig. 5**). This is a reasonable result, as the area studied for the new tester is smaller than that of



4. Pattern formed by the dyed water on studied boards after the Cobb test.

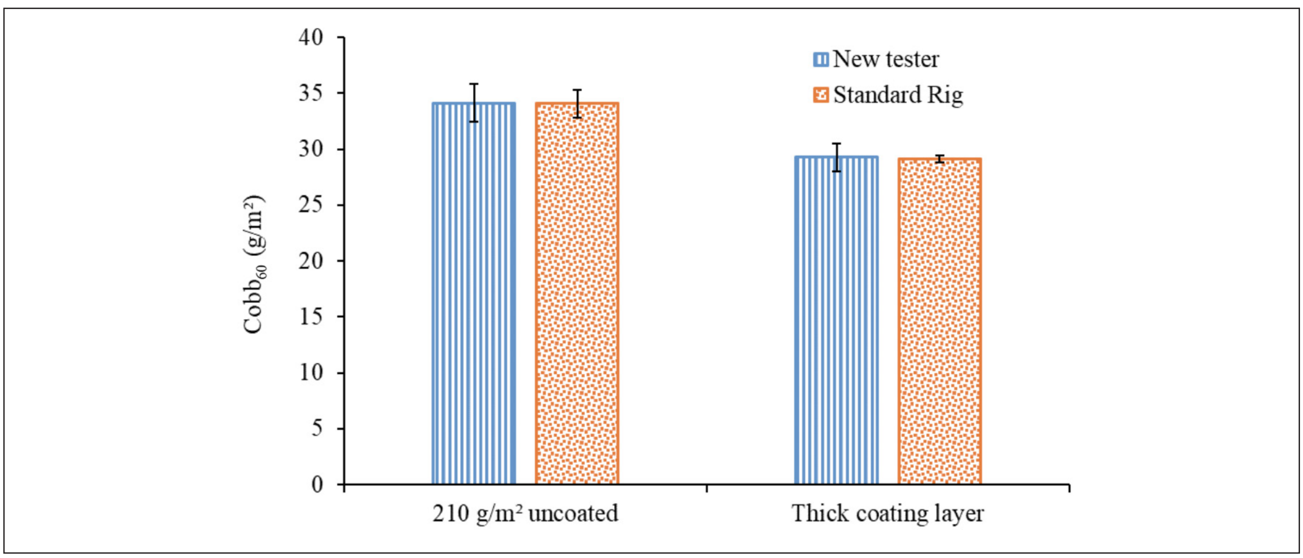
the conventional rig. This implies that the new tester takes account of fewer irregularities of the board surface, and therefore each variability weighs more in the final representation of the surface characteristics. However, the increase of variability was not significant. In addition, when comparing uncoated and coated samples, it is evident that coated samples show lower absorption (as expected).

Effect of the pressure and time on the water absorption of boards

Figure 6 shows the water absorption as a function of time and pressure (column height) for all the studied boards, and details of the water absorption results are presented in **Table III**. Both the water column pressure and the time had impacts on the absorption. However, the pressure of the water column was the predominant factor for the increase of absorption. For example, for a given pressure, the increase of absorption as a function of time was found in the range of a few grams. For both uncoated and coated boards, the absorption measurements became inconsistent at time of 60 s for high pressures as the boards became saturated with water. Therefore, these test points were not considered for further analysis. The highest absorption was found for the uncoated 210 g/m² boards due to their exposed fiber surface, while it was lower for open pre-coatings and it was lowest for closed pre-coatings. This was evident even after shorter periods of time. **Figures 7 and 8** show water absorption for shorter periods of time ($Cobb_{20}$ corresponding to 5 s exposure time and $Cobb_{30}$ corresponding to 15 s exposure time).

In general, for shorter periods of time, there was insignificant difference in the absorption when comparing 5 s exposure time (Fig. 7) and 15 s exposure time (Fig. 8), as the results were in the same range. As already mentioned, the highest absorption was found for the uncoated 210 g/m² boards due to their exposed fiber surface. As the pressure increased from ~0.1 kPa (corresponding to 10 mm water column) to ~5 kPa (corresponding to 500 mm water column), the water absorption increased from ~23 g/m² up to ~230 g/m² (Figs. 7–8).

Among the coated board samples, closed pre-coatings gave lower water absorption than open pre-coatings (Figs. 7–8). This was probably due to the presence of a higher pore volume and porosity in open pre-coatings observed in the SEM micrographs and pore volume measurement (Fig. 4b and Fig. 4d). These areas provide direct paths for the water into the coatings and, therefore, an effective

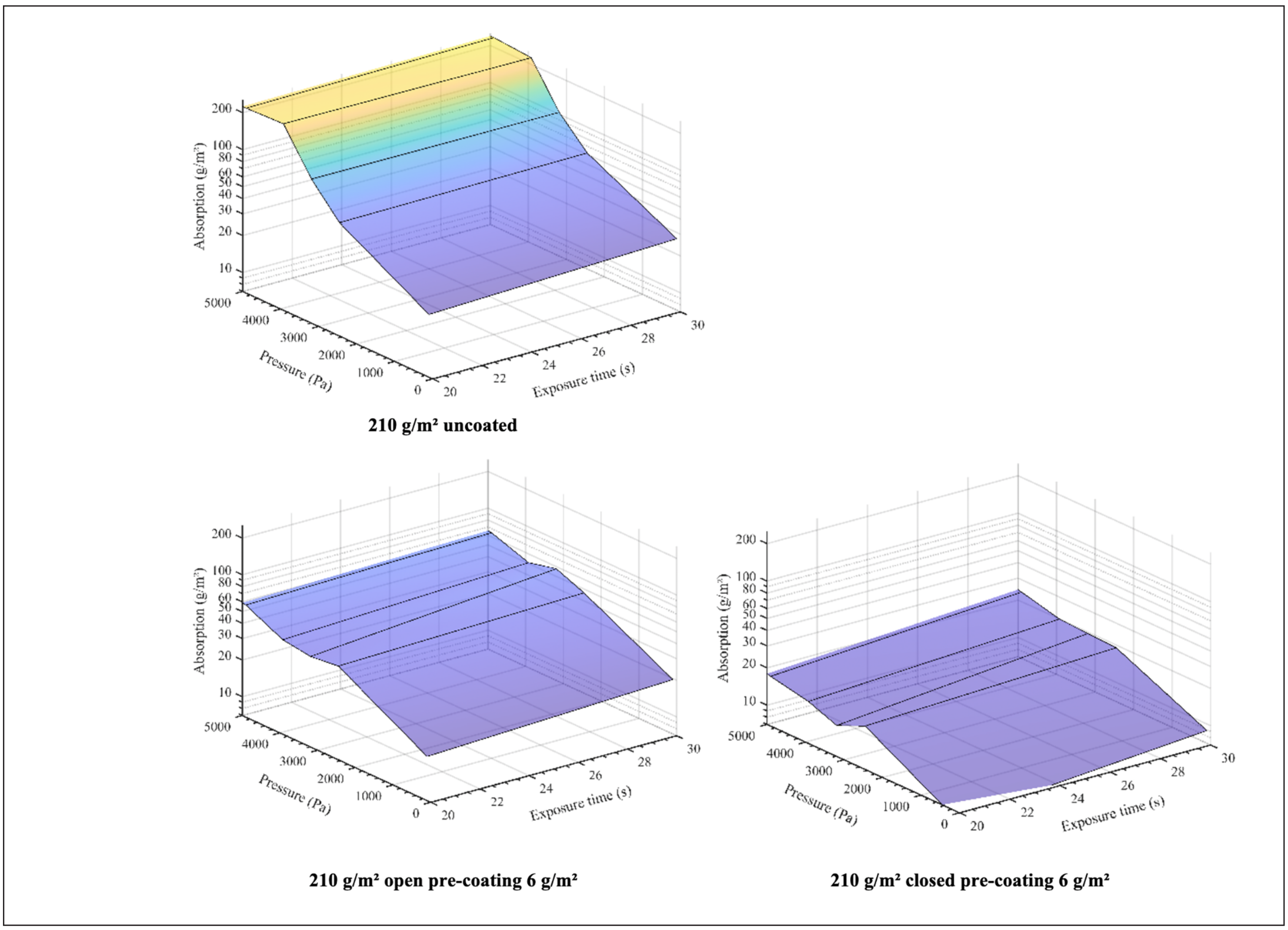


5. $Cobb_{60}$ conducted with conventional and new tester on uncoated board and a board coated with a thick layer of closed pre-coating (~14 g/m²). Error bars represent standard deviation.

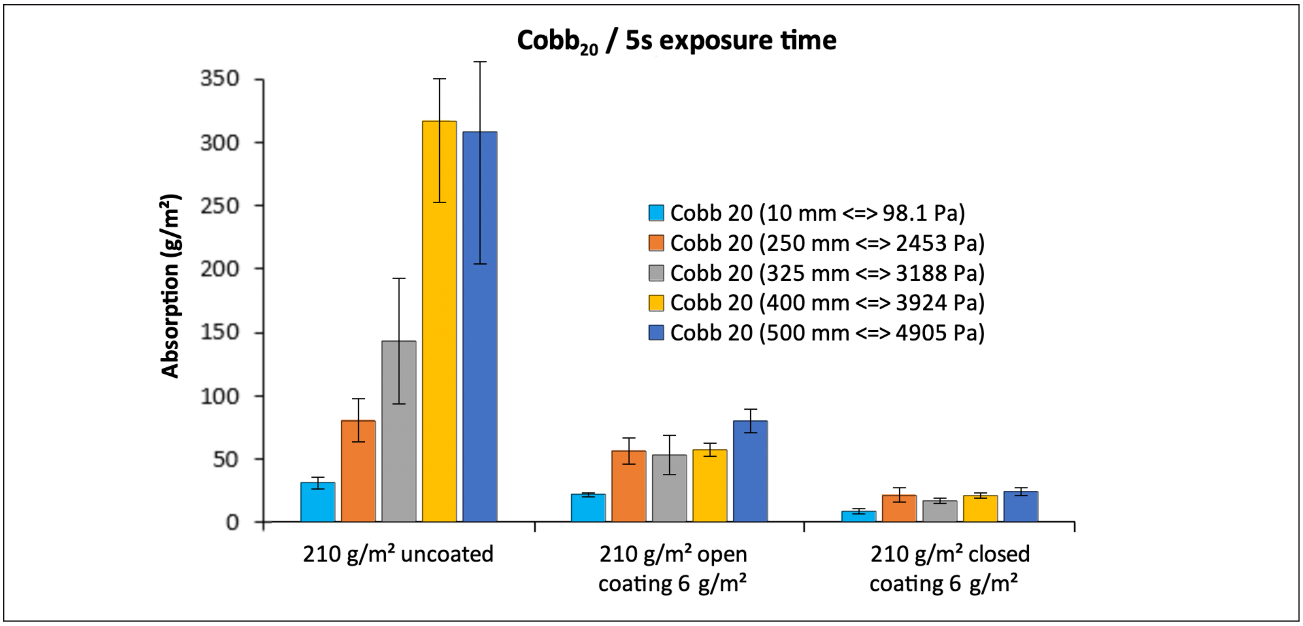
Boards	10 mm (98.1 Pa)			250 mm (2.453 Pa)		
	$Cobb_{20}$	$Cobb_{30}$	$Cobb_{60}$	$Cobb_{20}$	$Cobb_{30}$	$Cobb_{60}$
210 g/m², uncoated	22.69 ± 3.40	26.68 ± 3.87	34.14 ±1.65	58.66 ± 12.40	61.58 ± 6.04	85.24 ± 13.62
210 g/m², open coating 6 g/m²	16.10 ± 1.10	19.59 ± 3.40	27.01 ± 1.37	41.05 ± 7.28	46.56 ± 11.57	56.18 ± 15.22
210 g/m², closed coating 6 g/m²	6.05 ± 1.47	8.86 ± 1.39	18.58 ± 1.39	15.66 ± 3.94	19.01 ± 4.49	28.95 ± 4.72

Boards	325 mm (3.188 Pa)		400 mm (3.924 Pa)		500 mm (4.905 Pa)	
	$Cobb_{20}$	$Cobb_{30}$	$Cobb_{60}$	$Cobb_{20}$	$Cobb_{30}$	$Cobb_{60}$
210 g/m², uncoated	104.36 ± 35.99	103.71 ± 26.68	230.98 ± 46.76	226.87 ± 83.00	224.39 ± 75.83	239.48 ± 32.67
210 g/m², open pre-coating 6 g/m²	38.68 ± 11.58	57.69 ± 8.63	41.70 ± 3.51	50.88 ± 10.15	58.23 ± 6.69	64.93 ± 14.94
210 g/m², closed coating 6 g/m²	12.42 ± 1.41	19.45 ± 2.88	15.23 ± 1.34	19.99 ± 2.02	17.61 ± 2.23	23.88 ± 3.06

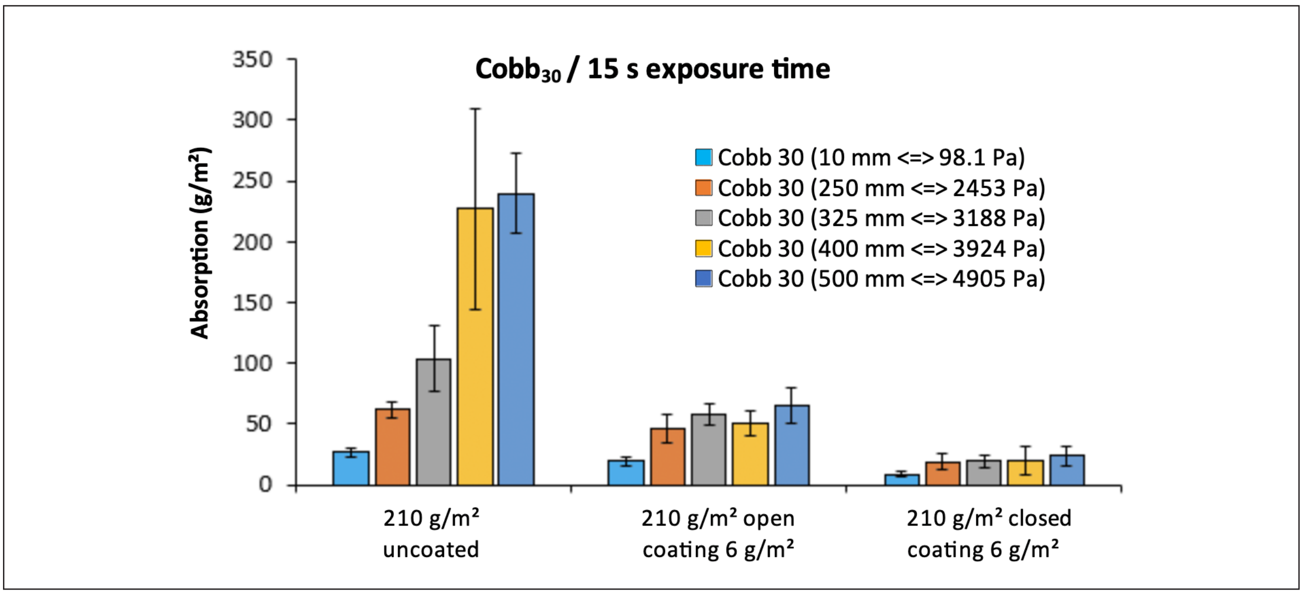
III. Average and standard deviation of Cobb test conducted at different exposure times and pressures (water height) for the studied boards.



6. Average water absorption at different exposure times and pressure of water.



7. Average water absorption of studied substrates at 20 s exposure time and different hydrostatic pressure (from ~0.1 kPa to ~5 kPa). Error bars represent standard deviation.



8. Average water absorption of studied substrates at 30 s exposure time and different pressure of water (from ~0.1 kPa to ~5 kPa). Error bars represent standard deviation.

mechanism for controlled water absorption. Traditionally, the coatings for printed application are made porous, i.e., to have controlled absorption of the inks. On the other hand, coatings with closed surfaces (less porous) tend to cover/close the fiber structure of the substrate surface more efficiently. The result is a reduced amount of water absorbed into the coating. As with the uncoated board samples, the pressure of the water column was the main factor for absorption, especially between water pressure at 0.1 kPa (column height of 10 mm) and 2.4 kPa (column height of 250 mm). At higher pressure, the differences were insignificant when comparing 5 s exposure time (Fig. 7) and 15 s exposure time (Fig. 8).

All results show that the proposed new tester could be used as an indicative tool for evaluation of the impact of the external pressure on water absorption. Its simplicity and similarity to the standard Cobb test makes the system an attractive method for measuring water absorption at different pressures. The operation of the tester can be compared to the standard Cobb test so that minimum extra equipment and training are required. According to the results, the new tester achieved similar results and slightly higher variability than those normally found by the standard testing device. At the moment, the new tester does not offer a practical way to analyze absorption at shorter time intervals that might be of the interest. For example, coating pressure exposure time (pressure pulse) under the blade or rod is much shorter than 1 s. However, this could be an opportunity for addressing a new research question as to where new simple tools that can handle shorter time intervals could be developed and tested.

CONCLUSION

The study presented the analysis of uncoated and coated board absorption subjected to different pressures based on a new testing device. According to the results, the new tester achieved similar results and slightly higher variability than those normally found by the standard testing device. When comparing exposure pressure and time, the absorption of boards was primarily driven by exposure pressure. This was evident even after shorter periods of exposure time of 5 s and 15 s. The highest absorption was found for the uncoated 210 g/m^2 boards due to their exposed fiber surface, while it was lower for pre-coated boards. It was found that boards coated with open pre-coatings absorbed a larger amount of liquid in comparison with closed pre-coated boards.

The proposed new tester is a simple device that allows analysis of board absorption subjected to different pressures. Its simplicity and similarity to the standard Cobb test makes the system an attractive method for measuring water absorption at different pressures. It might be suitable not only for coated board but also for other types of materials like paper, paperboard, and corrugated fiberboard. The operation of the tester can be compared to the standard Cobb test, so minimum extra equipment and training is required. Despite its simplicity, the proposed new tester provides a useful method for examining boards subjected to high pressures at different times. It was found that the new device accurately simulated the conditions of the conventional Cobb tester; therefore, measurement can be compared to standard methods.

Unfortunately, the new tester does not offer a practical way to analyse absorption at very short time exposure durations. This might be of the interest, for example, for coating where the pressure exposure time (pressure pulse) under the blade or rod is much shorter than 1 s. In addition, it might not be suitable for testing boards at long times and high pressure. However, this observation is dependent on the characteristics of the board. The weaker the board to the presence of liquid, the shorter exposure time and lower pressure can be applied during testing. **TJ**

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ABOUT THIS PAPER

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About this journal

ABOUT THE AUTHORS

We developed this testing method because we needed a simple method for testing the impact of external pressure on absorption in a coating process, which is a new concept. We discovered that absorption of boards was mainly driven by exposure pressure by comparing with exposure time. Thus, this new tool/method could be utilized for testing associated with the dispersion coating process of paper and paperboard.

For mills, this tool/method could be easily used to evaluate and/or predict behavior of paper coatings or substrates. During its development, we found it was difficult to work with short time intervals, such as 1–2 s or less. Therefore, the next step is to tune the tool/method and try to work with higher external pressure and shorter time intervals.

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sortium consisting of Cellcomb, Omya International, Holmen Iggesund Paperboard, and UMV Coating Systems.

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