Impact of different calendering strategies on barrier coating pickup

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ABSTRACT: Paper was pre-calendered in a pilot scale configuration with a traditional soft nip calender and a metal belt calender. All calendering strategies reduced surface roughness and permeability of the samples, but different strategies affected the surface roughness and permeability differently. The metal belt calender seemed to have a larger effect on the large-scale variations compared to the soft nip calender.

Six test points from the pilot calendered papers were chosen for laboratory coating studies. Uncalendered paper was included as reference samples. The calendered samples and the reference were pre-coated with a regular pigmented coating consisting of a ground calcium carbonate (GCC) pigment and a styrene acrylate (SA) latex. Both uncoated and pre-coated substrates were barrier coated with a polyvinyl alcohol (PVOH) in one and two layers. The coating pickup was determined gravimetrically, and the barrier properties were evaluated with TAPPI Standard Test Method T 454 grease resistance test.

All samples needed two PVOH coating layers to form a grease barrier. The uncalendered sheets showed the best results with one coating layer, but this was at the expense of a higher coating pickup compared to the calendered sheets. The barrier coating pickup could be reduced by a combination of high temperature metal belt calendering and pre-coating. The high temperature and long residence time in the nip enabled plasticization of the fibers. This led to an irreversible deformation, even after water application. This meant that the smoothness obtained during calendering would be less affected by water-induced roughening during the coating operation.

Application: Most works on barrier research have previously focused on either coating formulations or coating processes. This work complements earlier research by demonstrating that the amount of required coating color can be reduced through an appropriate calendering strategy and use of pre-coating.

In barrier coating, a continuous film is formed on the top of the paper or paperboard surface to achieve a barrier against oxygen, water, or oil and grease. The transport of gases and liquids will depend on the impermeability and thickness of the barrier material and the amount of defects (pin holes and cracks) in the coating layer. A more homogeneous barrier layer will give a better coverage and thus improve barrier performance. The barrier properties will therefore depend on the material properties of the barrier, the coating operation, and the substrate smoothness and porosity.

Schuman et al. [1] studied the pretreatment of paper and board and its impact on the barrier performance of dispersion coated products. The barrier properties were characterized as the water vapor transmission rate of barrier coated board. Both pre-calendering and pre-coating improved the water vapor barrier of a latex-based formulation applied with dispersion coating. Schuman et al. concluded that a smooth surface is beneficial for the final barrier performance [1]. A later study where polyvinyl alcohol (PVA) was applied as a barrier coating indicated that the smoothness of the substrate is the governing factor for barrier performance [2]. By improving the wettability of the surface with Corona treatment, an improved uniformity of the barrier coating could be obtained. An improved coating uniformity would result in better coverage of the surface and improved barrier performance.

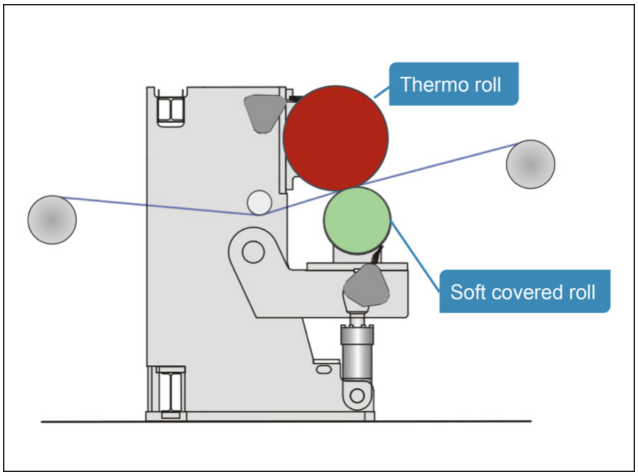
The smoothing effect of calendering is achieved by the replication of the smooth roll surface onto the surface of the substrate [3]. The replication is further enhanced by increased pressure and the softening effect of the fibers at high temperatures [4]. During compression, the thicker parts of the substrate will carry most of the load [5,6], and smoothing is thus achieved by compressing the floc areas more than the non-floc areas, introducing density variations [7-9]. The smooth surface after calendering therefore comes at the expense of density variations within the sheet.

Due to compression, stresses are built into the sheet that relax when water is applied to it during the coating operation. Some of the smoothing effect of calendering is therefore lost when a coating is applied. Since the floc areas are mostly compressed, the floc areas will expand the most when water is applied. Engström and Lafaye [10] studied the effect of pre-calendering on lightweight coated (LWC) papers. In this study, the smoothing effect of pre-calendering was lost as the coating was applied to papers. Their conclusion was that pre-calendering has no positive effect on the final properties. However, it is emphasized that an unheated calender roll was used in this study. Chin and Yuon [11] studied the roughening of calendered papers after water application. Papers calendered without a heated roll showed reversible changes after calendering, and significant roughening was observed when water was applied. A higher calendering temperature introduced irreversible changes in the surface structure, leading to a lower roughening after water application.

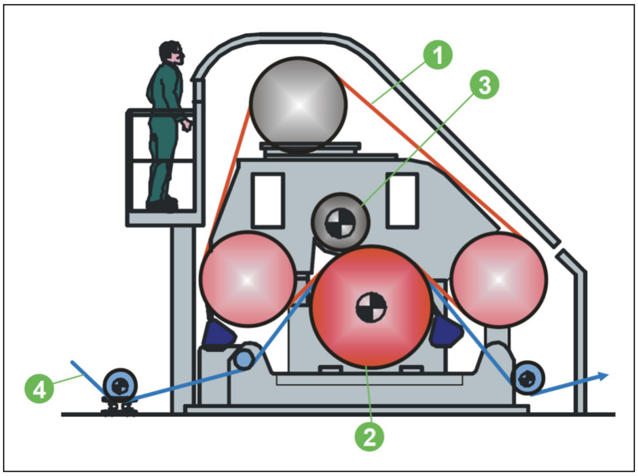
Ghosh and Hart [12] studied the impact of web temperature on the densification during calendering. Sheet density increased considerably with sheet temperature. This is due to the thermal softening of the fibers, leading to a higher degree of plasticization of the sheet. Plasticization of the sheet is necessary to obtain irreversible deformation in the thickness direction, even after water application [11].

Rautiainen et al. [13] applied different calendering strategies in combination with curtain coating for pigmented coatings. Metal belt calendering and soft nip calendering were compared. The metal belt calender combines a metal belt with a heated nip. The metal belt will give a longer residence time in the nip and a more effective heat transfer to the web. The improved heat transfer will plasticize the web and give a larger deformation of the web, resulting in improved smoothness. The metal belt calender improved the large-scale smoothness to a higher extent than the traditional soft nip. Combined with the curtain coating process, this improved the coating uniformity of pigmented coated substrates.

The aim of this work has been to investigate how different pre-calendering strategies affect the barrier properties after coating. Paper was calendered in pilot scale with a metal belt calender and in a traditional soft nip calender. A limited number of test points were then chosen based on surface roughness and permeability. Samples from these test points were then coated in a laboratory bench coater with a pigmented pre-coating and polyvinyl alcohol (PVOH) barrier coating.



1. Schematic illustration of the soft nip calender used in the calendering trials.



2. Schematic illustration of the metal belt calender used in the trials. The different parts of the calender are shown: (1) steel belt, (2) thermo roll, (3) deflection compensation roll, and (4) web.

MATERIALS AND METHODS

A 55 g/m² uncoated and uncalendered paper was used for the trials. The paper was calendered at the Valmet pilot plant in Järvenpää, Finland. A traditional soft nip and a metal belt calender were used for the trials, as shown in **Fig. 1** and **Fig. 2**, respectively. The line loads for the traditional soft nip calender were 250, 350, and 450 kN, and the temperature of the heated roll was 200°C. The line loads with the metal belt calender were 25, 50, 75, 100, and 125 kN/m, and the temperatures of the heated roll were 120°C and 170°C. The web speed during the trials was 1000 m/min.

The surface properties of the calendered substrates were characterized at the pilot plant for Parker Print Surf (PPS)-10 surface roughness according to ISO Standard 8791-4:2021 “Paper and board — Determination of roughness/smoothness (air leak methods) — Part 4: Print-surf method,” and the Bendtsen surface roughness was determined according to ISO Standard 8791-2:2013 “Paper and board — Determination of roughness/smoothness (air leak methods) — Part 2: Bendtsen method.” Bendtsen air permeability was determined at the laboratories of RISE in Stockholm according to ISO Standard 5636-3:2013 “Paper and board — Determination of air permeance (medium range) — Part 3: Bendtsen method.” Sheets with similar PPS or similar Bendtsen air permeability were chosen for the coating trials.

The pilot coated papers were then coated in a bench coater from RK PrintCoat Instruments Ltd. (Royston, UK) using a wire wound rod giving a wet deposit thickness of 12 µm. A pre-coating consisting of a ground calcium carbonate (GCC) pigment was used with a styrene acrylate (SA) latex and carboxymethyl cellulose (CMC). The GCC pigment was Covercarb 60 from Omya (Oftringen, Switzerland), and the SA latex was HPB 69 from Trinseo (Wayne, PA, USA). A PVOH, Poval 15-99 from Kururay Europe (Hattersheim am Main, Germany), was used for the barrier coatings. The solids content of the pre-coating was 50%, and

	Pre-coating	Barrier Coating
Dry content, %	50	15
GCC, pph	100	
SA latex, pph	15	
CMC, pph	0.5	
PVOH, pph		100
GCC: ground calcium carbonate; SA: styrene acrylate; CMC: carboxymethyl cellulose; PVOH: polyvinyl alcohol.		

I. The pre-coating and barrier coating formulations used in the laboratory coating trials. The proportions of coating components are given as part per hundred (pph) pigment for the pre-coating and parts per hundred of PVOH for the barrier coating.

the solids content of the barrier formulation was 15%. All coatings were then dried in an oven at 105°C for 10 min. The barrier coating was applied in one or two layers. The coating formulations with the dry content are given in **Table I**.

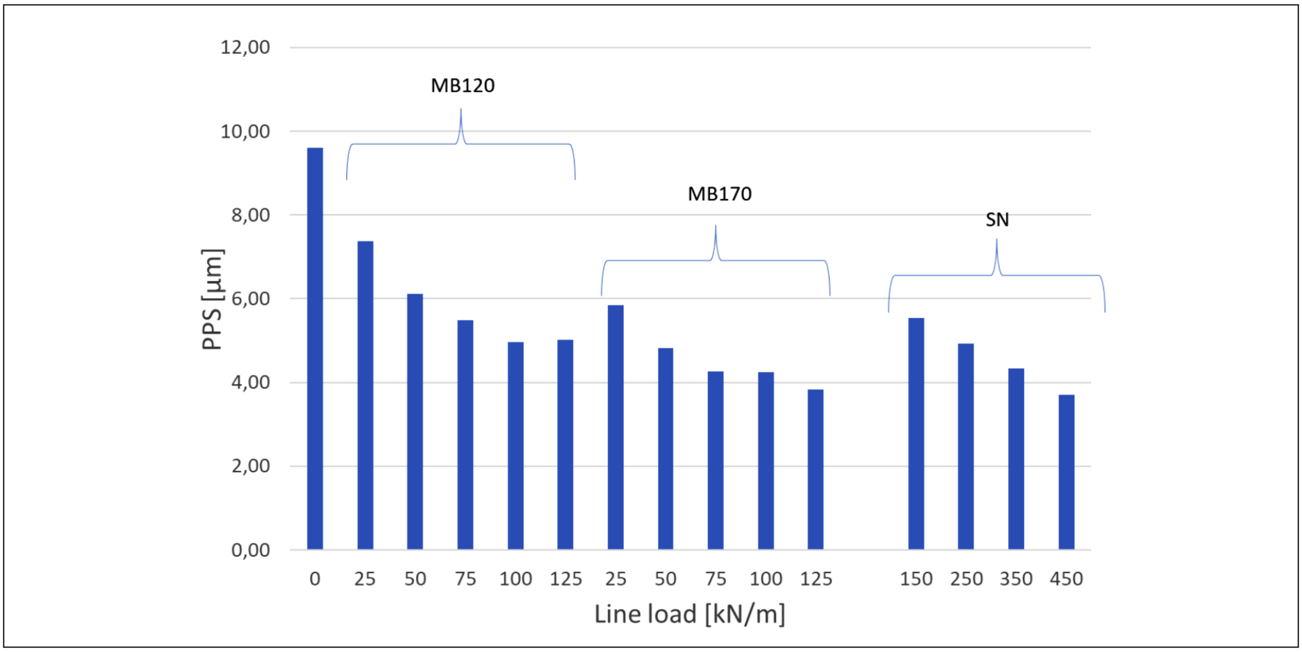
To determine the roughening effect of water during coating, uncalendered and calendered sheets were coated with deionized water in the bench coater. A wired wound rod with a wet deposit of 4 µm was used, and the sheets were dried in an oven at 105°C for 5 min. The topography of the untreated sheets and the water coated sheets were determined with OptiTopo, a photometric stereo imaging method [14]. Height maps, 10*10 mm², and height distributions were calculated to determine the surface topography in different size scales.

The coating pickup was measured gravimetrically by cutting out rounds of uncoated and coated substrates. Before the grammage determination, the rounds were placed in an oven to dry. The coating samples were compared to uncoated references. The oil barrier properties of the PVOH coated sheets were determined using TAPPI Standard Test Method T 454 "Turpentine test for voids in glassine and greaseproof papers."

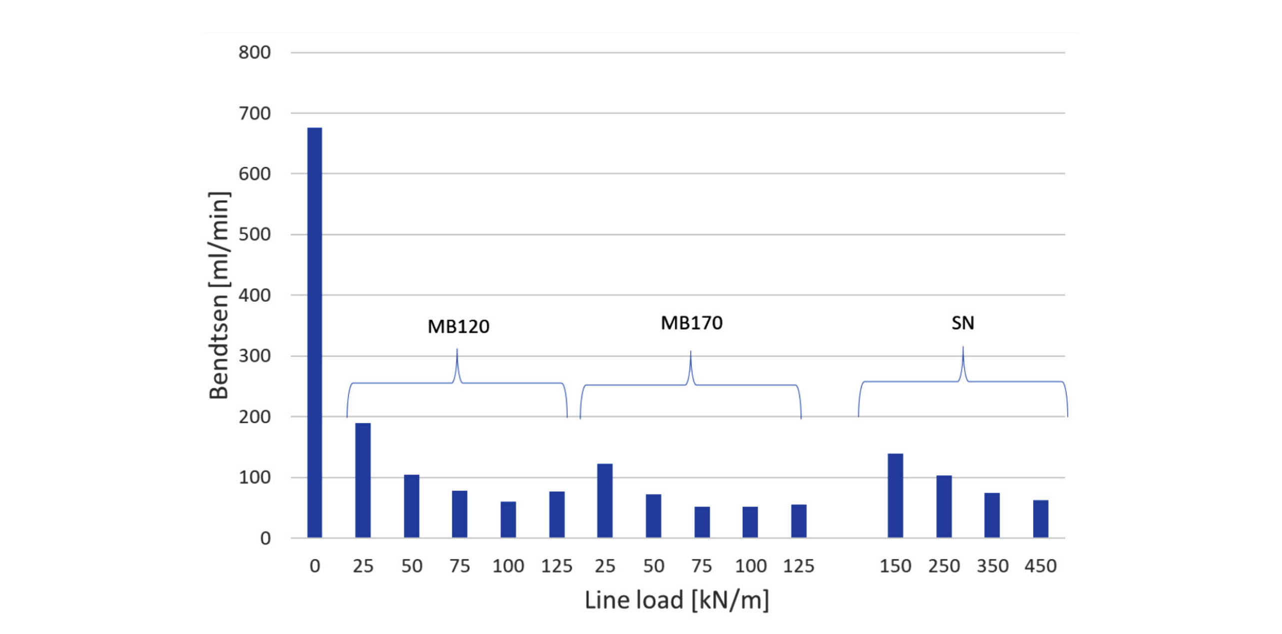
RESULTS AND DISCUSSION

Figures 3–4 show the surface roughness of the paper samples after calendering. The PPS roughness seemed quite similar between the higher temperature of the metal belt and the soft nip calender. It seemed that the metal belt calender had a higher impact on the Bendtsen roughness than the soft nip calender. Since the Bendtsen roughness is more sensitive to large scale roughness, it seems reasonable to assume that the metal belt calender had a larger impact on large scale roughness than the soft nip. This is in line with the observation of Rautiainen et al. [13]. Both the PPS and Bendtsen roughness decreased when the calendering temperature increased in the metal belt calender. It also seemed that the PPS was more affected by temperature than the Bendtsen roughness.

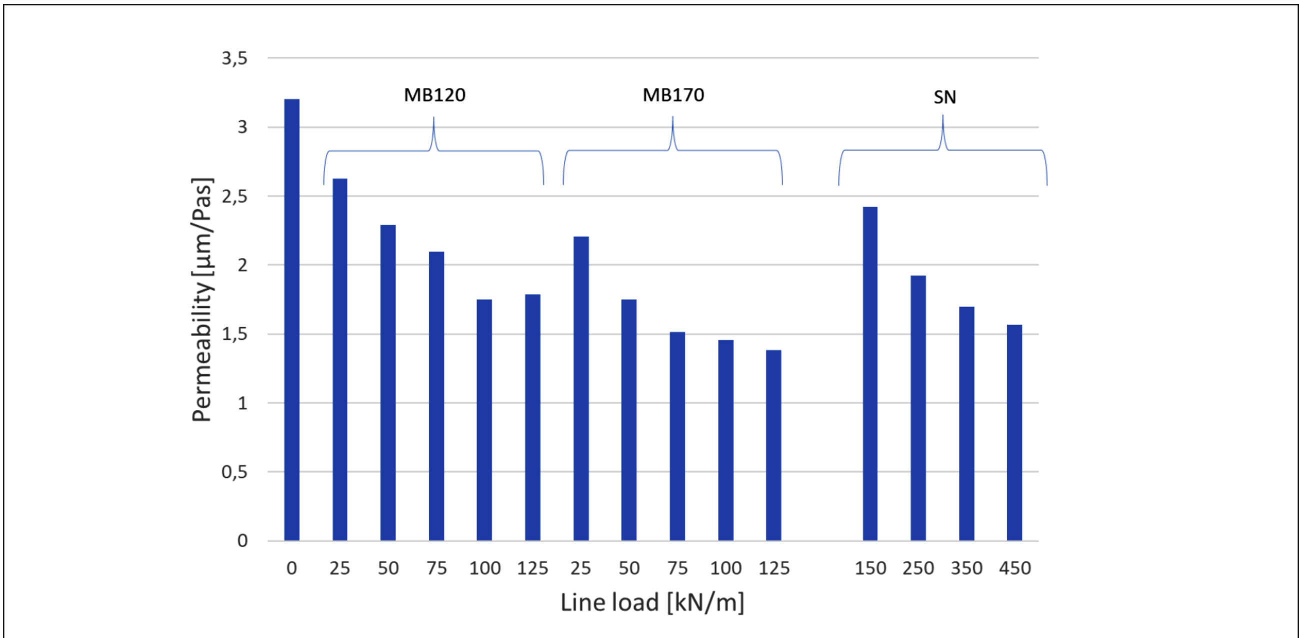
Figure 5 shows the air permeability of the calendered paper. As the paper was compressed, the structure densified and improved smoothness, leading to a reduced air permeability. As the line load increased, the permeability decreased. The decrease was also more pronounced for the metal belt calender compared to the soft nip calender. Increased temperature in the metal belt calender, 170°C, significantly decreased the air permeability. Temperature had



3. The Parker Print Surf (PPS) surface roughness after calendering. MB120 refers to metal belt calendering at 120°C, MB170 refers to metal belt calendering at 170°C, and SN refers to soft nip calendering.



4. The Bendtsen roughness after calendering. MB120 refers to metal belt calendering at 120°C, MB170 refers to metal belt calendering at 170°C, and SN refers to soft nip calendering.



5. The Bendtsen air permeability after calendering. MB120 refers to metal belt calendering at 120°C, MB170 refers to metal belt calendering at 170°C, and SN refers to soft nip calendering.

a more pronounced effect on the Bendtsen air permeability than the Bendtsen roughness.

From these results, five papers and an uncalendered reference were chosen, as shown in **Table II**. Sheets were chosen in order to compare soft nip calendered and metal belt calendered sheets with similar PPS roughness, similar Bendtsen roughness, or similar air permeabilities. A test point with a lower metal belt calendering temperature was also chosen.

Coated papers

The permeability of the uncoated and pigmented coated paper is shown in **Table III**. The permeability decreased considerably with calendering, as seen in Fig. 4. Pre-coating also decreased the permeability for all samples, and the result also differed between calendering conditions. A lower permeability before coating gave a lower permeability of the coated paper. The largest difference in air permeability between uncoated and pre-coated paper was ob-

Sample and Calendering	Bendtsen Roughness, mL/min	Bendtsen Roughness, mL/min	Air Permeability, µm/Pas
SN 200°C – 450 kN/m	3.71	61.8	1.57
MB 170°C – 100 kN/m	3.83	55.2	1.39
SN 200°C – 350 kN/m	4.34	74.7	1.7
MB 170°C – 30 kN/m	4.82	72.2	1.75
MB 120°C – 125 kN/m	4.96	59.7	1.75
Uncalendered	9.6	676	3.2
PPS: Parker Print Surf; SN: soft nip calender; MB: metal belt calender.			

II. The substrates chosen for coating studies.

Sample and Calendering	Permeability	
	Uncoated, µm/Pas	Pre-coated, µm/Pas
SN 200°C – 450 kN/m	1.565	0.664
MB 170°C – 100 kN/m	1.385	0.576
SN 200°C – 350 kN/m	1.695	0.696
MB 170°C – 30 kN/m	1.75	0.646
MB 120°C – 125 kN/m	1.75	0.721
Uncalendered	3.2	0.703
SN: soft nip calender; MB: metal belt calender.		

III. The permeability of the uncoated and pigment coated paper samples.

tained for the uncalendered paper, partly due to an initial high air permeability. Metal belt calendering at the higher temperature, 170°C, seemed to give a lower air permeability after pre-coating compared to similar soft-nip calendered samples and the paper calendered with the metal belt at the lower temperature after pre-coating.

The coating grammage of different coating layers, pre-coatings, and PVOH coatings are given in **Table IV**. Note that the coating grammage of the PVOH-coating is given as the total amount of PVOH. Some general observations were made when comparing the pre-coating pickup and the PVOH coating pickup:

- The uncalendered papers showed a considerably higher amount of pre-coating, probably due to higher porosity and roughness. This also explains the large decrease in air permeability after pre-coating.
- The uncalendered papers showed about the double amount of PVOH coating pickup compared to the pre-calendered papers. This was valid for both papers with pre-coating and the papers without pre-coating.
- The soft nip calendered papers also showed a slightly higher pre-coating grammage than the metal belt calendered papers.

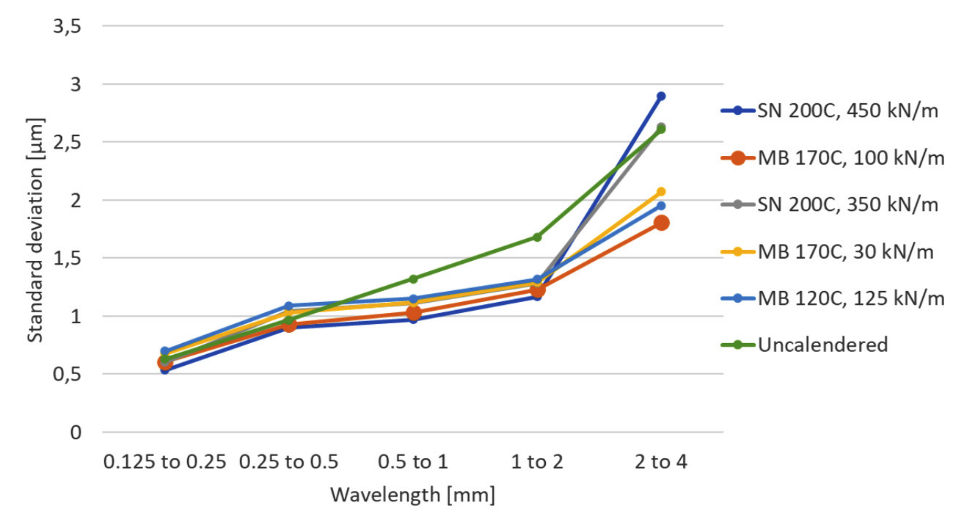
A slight difference could be observed between the two different calendering methods. The soft nip calendering gave no or small reductions in PVOH coating pickup when a pre-coating was used. When the higher temperature, 170°C, was used in the metal belt calender, a lower amount of PVOH coating pickup was observed when a pre-coating was used. In this case, combining metal belt calendered and a pre-coating gave a significantly lower PVOH coating pickup compared to the combination of soft nip calendered and pre-coating.

Roughness before and after water application

The roughness before and after water application is shown in **Fig. 6** and **Fig. 7**, respectively. The results are presented as height standard deviation at different wavelengths representing different size scales. Calendering reduced the surface variations considerably compared to the uncalendered paper (Fig. 6). The soft nip calendered papers at 200°C and line loads 450 kN/m and 350 kN showed lower variations in mid- and low-scale area, 0.125–2 mm, and higher variations in the large-scale area, 2–4 mm, compared to the metal belt calendered papers. Before water application, metal belt calendered papers at 120°C and 125 kN/m

Uncoated			Pre-coated			
Sample and Calendering	PVOH Coating		Sample and Calendering	Pre- coating, g/m²	PVOH Coating	
	One Layer, g/m²	Two Layers, g/m²			One Layer, g/m²	Two Layers, g/m²
SN 200°C – 450 kN/m	1.82	3.66	SN 200°C – 450 kN/m	7.55	2.24	3.85
MB 170°C – 100 kN/m	1.88	3.01	MB 170°C – 100 kN/m	5.18	1.01	1.89
SN 200°C – 350 kN/m	1.66	3.25	SN 200°C – 350 kN/m	7.43	1.71	3.04
MB 170°C – 30 kN/m	1.10	3.99	MB 170°C – 30 kN/m	6.40	1.65	2.58
MB 120°C – 125 kN/m	1.50	3.58	MB 120°C – 125 kN/m	7.80	2.82	4.16
Uncalendered	3.21	6.01	Uncalendered	17	1.43	4.92
PVOH: polyvinyl alcohol; SN: soft nip calender; MB: metal belt calender.						

IV. The pre-coating and PVOH coating grammages of calendered and coated paper samples. The coating grammage of two layers of PVOH is the total amount of PVOH (i.e., layer1 + layer2).



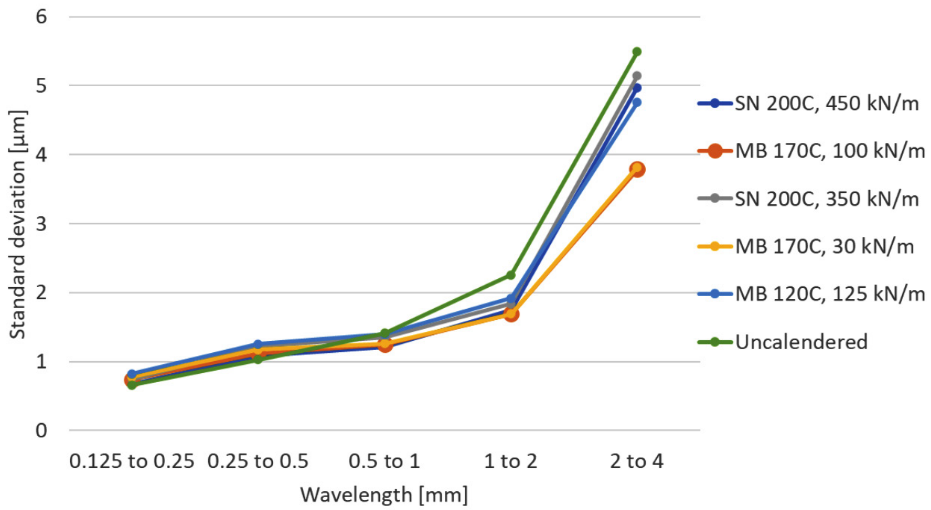
6. The surface variations at different wavelengths before water applications. The surface variations are given as the height standard deviation. MB120 refers to metal belt calendering at 120°C, MB170 refers to metal belt calendering at 170°C, and SN refers to soft nip calendering.

showed similar patterns in surface variations at different size scales as the metal belt calendered paper at 170°C. In this case, the higher line load, 125 kN, could compensate for the lower temperature.

After water application, the roughness increased for all papers and the surface variations increased to a higher degree in the larger wavelengths (Fig. 7). The uncalendered paper showed the highest surface variations of the papers after water application. All calendered papers gave similar surface variations after water application at low- and medium-scale wavelengths, 0.125–2 mm.

At longer wavelengths, 2–4 mm, the soft nip calendered papers and the metal belt calendered paper at 120°C had similar surface variations after water application (Fig. 7). The large-scale surface variations of these papers were close to the uncalendered paper. The metal belt calendered paper at 170°C and line loads 30 and 100 kN/m showed significantly lower large-scale surface variations after water applications. The longer nip residence time in the metal belt calender would enable the heat to penetrate further down in the sheet and enable plasticization of the fibers. This would lead to lower surface variations at longer wavelengths.

After water application, the metal belt calendered papers at 120°C showed considerably higher large-scale variations than the papers calendered at 170°C after water applications. The variations of the paper calendered at 120°C was on the same level as the uncalendered paper after water application. It seems plausible that there was not enough heat to plasticize the paper when the lower temperature



7. The surface variations at different wavelengths after water applications. The surface variations are given as the height standard deviation. MB120 refers to metal belt calendering at 120°C, MB170 refers to metal belt calendering at 170°C, and SN refers to soft nip calendering.

was used in the metal belt calender. This led to a higher amount of roughening at large wavelengths due to water-induced relaxation of built-in stresses.

Grease resistance

The paper samples coated with one layer of PVOH showed immediate penetration, except for the uncalendered sample, as shown in **Table V**. Surprisingly, the uncalendered papers showed the best barrier performance. This was, on the other hand, achieved by a large amount of coating pickup, as shown in Table IV. In fact, one layer of PVOH on the uncalendered paper had roughly the same grammage as two layers PVOH coating on the calendered papers. Coating a paper with these kind of coating grammages would make these papers much more costly than the calendered papers.

One of the samples, the metal belt calendered paper at 120°C and line load of 125 kN/m (MB 120°C, 125 kN/m), did not pass any of the TAPPI T 454 tests, even with a pre-coating and two layers of PVOH. This sample was also the roughest of the calendered samples. In this case, the pre-coating also had a visible positive effect, since the pre-coated sample showed penetration after 1200 s compared to 900 s for the sample without pre-coating. It therefore seems that two layers of PVOH were needed to cover the paper substrates enough to give a good grease barrier.

CONCLUSION

During these trials, no paper with one PVOH layer passed the grease resistance test, while most papers with two PVOH layers passed the grease resistance tests. The only exception was the paper calendered at the lowest tempera-

Samples with No Pre-coating			Samples Pre-coated with GCC/Latex		
Sample	One Layer	Two Layers	Sample	One Layer	Two Layers
SN 200°C – 450 kN/m	5	1800	SN 200°C – 450 kN/m	5	1800
MB 170°C – 100 kN/m	1	1800	MB 170°C – 100 kN/m	5	1800
SN 200°C – 350 kN/m	5	1800	SN 200°C – 350 kN/m	5	1800
MB 170°C – 30 kN/m	5	1800	MB 170°C – 30 kN/m	5	1800
MB 120°C – 125 kN/m	5	900	MB 120°C – 125 kN/m	5	1200
Uncalendered	1500	1800	Uncalendered	1200	1800

PVOH: polyvinyl alcohol; SN: soft nip calender; MB: metal belt calender.

V. The results from the TAPPI T 454 grease resistance test for PVOH coated paper samples. A sample is considered to have passed the test if the sample reaches 1800 s.

ture (metal belt calendered paper at 120°C with a line load of 125 kN/m). It is therefore hard to make direct comparisons between the calendering strategies and the role of the pre-coating just by using the grease resistance tests.

All the calendered and single PVOH coated papers failed the grease resistance tests quite immediately, and all except one passed the same test with two PVOH layers. It is likely that the second layer was near the limit of when a grease barrier was established. This was especially so since the low temperature metal belt calendered paper failed the test with a second layer. It therefore seemed relevant to compare the PVOH coating pickup of the papers as long as the papers passed the TAPPI T 454 grease resistance test.

Different calendering strategies gave different results on the amount of pre-coating and PVOH coating, and the thermal softening of the fibers seemed to be of central importance. Thermal plasticization of the fibers resulted in a lower water-induced roughening (Fig. 6 and Fig. 7). This is in line with earlier results in literature [11-12]. The lower water-induced roughening would result in a more homogeneous coating distribution and also a lower coating pickup [10]. The more homogenous surface will also give an improved coverage and a lower coating pickup in the following PVOH coating steps, as shown in Table IV. All pre-calendering strategies had a positive effect, giving a reduced coating pickup compared with the uncalendered paper.

The soft nip calendered paper showed a relatively high pre-coating pickup. Ghosh and Hart [12] showed that a high sheet temperature is necessary to obtain densification of the sheet. The residence time in the traditional soft calender nip at 1000 m/min is extremely short. This means that the heat will not have time to penetrate the sheet and enable full plasticization of the fibers. Consequently, the soft nip calendered paper showed higher surface variations at longer wavelengths than the metal belt calendered papers at 170°C (Fig. 6). More coating grammage would be needed to cover the large surface variations, which would lead to a higher coating pickup. The high coating pickup of the paper metal belt calendered paper at 120°C was probably due to water-induced roughening at larger wavelengths (Fig. 7). This was probably caused by insufficient plasticization of the fibers due to the lower calender roll temperature.

ABOUT THE AUTHORS

We wanted to investigate how different pretreatment strategies can be applied to improve the performance of barrier coatings. During this work, we had the oppurtunity to perform pilot calendering trials with many different calendering strategies.

Barrier coating has gained a lot of research interest in recent years. At the same time, we have not been able to find any research works that have tried to explain the role of both pre-calendering and pre-coating on the final barrier performance. This work therefore complements earlier research on barrier coating.

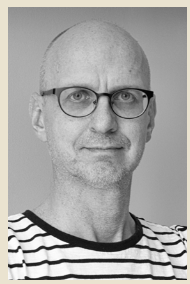
A challenging aspect of this work was to find a methodology to compare the barrier performance of papers that had been pretreated in different manners without mixing too many parameters. This was achieved by choosing among numerous calendered samples to obtain samples with either similar short scale roughness (PPS), long scale roughness (Bendtsen), or porosity (Bendtsen air permeability). These samples were then coated to a point where they would provide an acceptable grease barrier. Evaluation of barrier performance thus became more of an evaluation of how much coating was needed to obtain full coverage of the surface, i.e., the coating pickup.

Our main discovery was that reduction in large scale roughnesses and thermal softening during pre-calendering had a large impact on coating coverage. In the process, previous knowledge about calendering, pre-calendering, and traditional pigmented coating proved useful in the interpretation of the results. This was also the origin of the idea to measure the roughness before and after water application on the calendered substrates. It was most interesting to find that the pre-calendering strategy had such an impact on the pre-coating and barrier coating pickup.

The results of this work show that coating coverage can be improved by a combination of appropriate calendering strategy and pre-coating. This enables reduction of barrier coating color with maintained barrier performance, and thus, cost savings. If we find funding in the future, we would like to perform the same pre-coating and barrier coating trials at pilot scale.

Rättö

Junel



Valtakari

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A high temperature combined with a long nip residence time, metal belt calendering at 170°C, gave full plasticization of the fibers and less water-induced roughening. This resulted in a lower pre-coating pickup. In the next step, the PVOH coating pickup was reduced further by the smooth surface created by the GCC based pre-coating. No such reduction was observed for the metal belt calendered paper at 120°C or the soft nip calendered paper. These papers also showed a higher pre-coating pickup.

An appropriate calendering strategy combined with precoating can thus be used to optimize the barrier performance and coating pickup. **TJ**

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