Multilayering of conventional latex-based dispersion coatings containing small amounts of silica nanospheres: Runnability on a pilot scale flexographic coater and barrier performance

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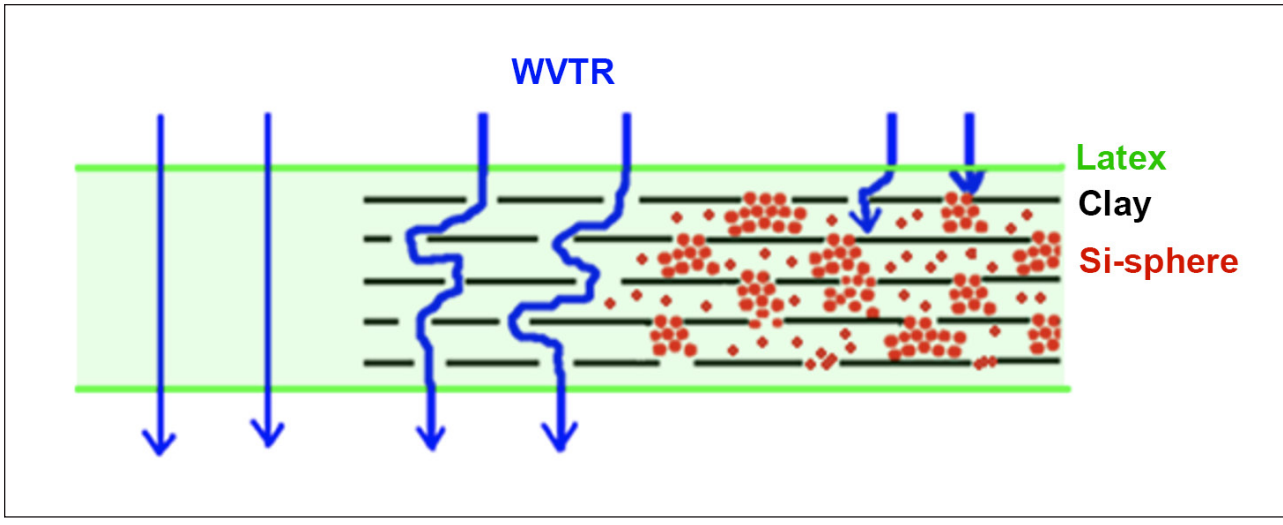
ABSTRACT: The addition of functional coatings to packaging materials is a key requirement for increasing their performance and creating innovative packaging solutions. Flexography, a cost-effective printing method commonly used to print information and graphics directly onto a wide variety of packaging substrates, shows good potential for applying functional coatings. In this study, conventional clay-latex coating formulations containing approximately 1.3 wt% silica nanospheres were applied to a linerboard using a pilot scale flexographic printing web press. The performance of multilayered silica nanosphere-based coatings was compared with conventional coatings containing talc and/or wax dispersion in terms of coating grammage, runnability, and barrier performance. Coating grammage increased with an increased number of coating layers and a significant decrease in both the water vapor transmission rate (WVTR) and the direct water uptake of water (Cobb 120 wettability test) was observed for coatings containing silica nanoparticles. In general, the silica nanosphere-based coatings performed better than talc-based coatings. Talc/wax-based coatings had the highest variation in surface roughness due to an uneven distribution and variations of coating layers.

Application: This study presents a novel way for creating barrier dispersion coatings and applying them onto paper-based substrates. By utilizing nanomaterials and multilayering, it is possible to create low grammage coatings with high water and moisture barriers. As similar approach could be used for development of oxygen and carbon dioxide barriers.

Paperboard is one of the most commonly used packaging materials, often specifically engineered and treated to provide certain functionality. One of the most important functionalities is the moisture barrier, because it increases packaging and product shelf-life [1]. The moisture barrier properties are characterized by direct liquid water uptake (Cobb test) and water vapor transmission rate (WVTR) measurements. In the production of coated linerboard products, a method commonly used to enhance the barrier performance is the application of barrier dispersion coatings. These normally consist of mineral pigments (commonly clay and talc) in a polymer latex (typically styrene-butadiene, styrene-butyl acrylate, or polyvinyl acetate) [2]. The role of the latex is to form a film and that of the pigment is to improve barrier properties (providing additional mechanical barrier and increasing the length of the diffusion pathway for permeants) and assist with printability, glueability, and blocking issues [3-6]. Most dispersion coatings based on latex, clay, and talc, readily meet the requirements for sustainability by being fully repulpable and easily compostable [7].

The use of kaolinite clay or hydrophobic materials, such as talc and hydrophobic clay, in coatings to improve their barrier properties has been reported elsewhere [5,7-11]. However, the disadvantage with hydrophobic pigments is in their potential issue with dispersing and processing. It is known that plate-like clay and talc filler materials with greater aspect ratios provide a more effective barrier layer [7,9]. These filler materials are traditionally used to hinder and increase the diffusion path length of the water vapor, as illustrated in **Fig. 1**. However, clay and talc particles do not provide a fully impervious water vapor barrier. The gaps/cavities between the particles in the latex coating often result in unwanted pathways for moisture, thereby undesirably increasing the WVTR [12]. One way of filling up the gap/cavity between clay and talc particles to improve barrier performance is to use silica nanospheres (SiO_2). The spherical morphology of silica nanoparticles enables them



1. Schematic description of a cross section of coating layer containing latex binder, kaolin clay, and silica spheres. Blue arrows: water vapor transmission rate (WVTR); light green: latex binder; black lines: kaolin flatty clay; red dots: = nanosilica (Si) spheres.

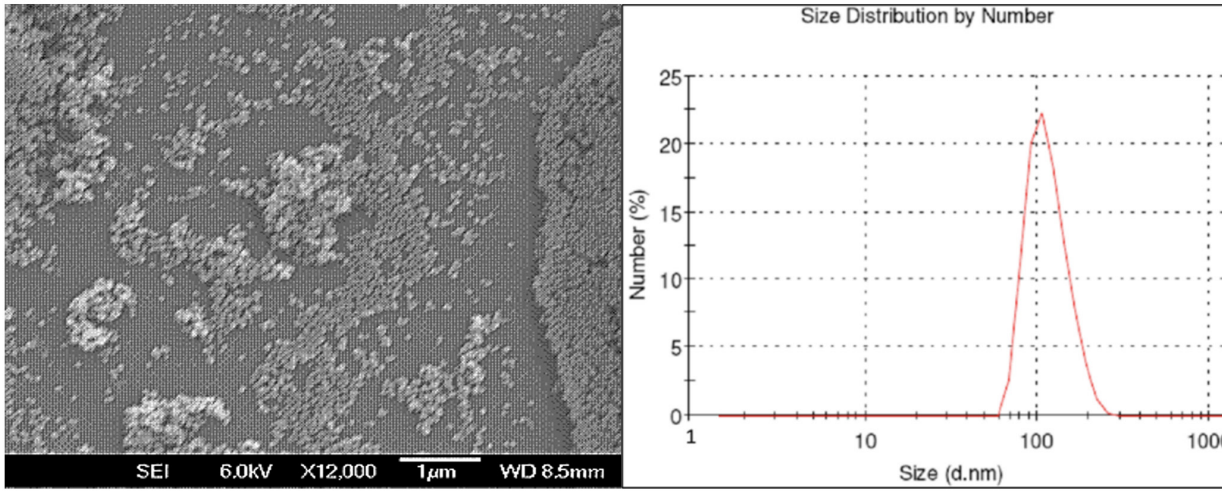
to readily occupy these gaps/cavities as the coating is formed and consolidated. As such, they are more evenly distributed across the coating layer and occupy interfacial spaces and voids of the coated film where the larger clay platelets cannot fit, as shown in Fig. 1. Quite often, due to the hydrophobic properties of talc- and wax-based coatings, it is not possible to apply double or multiple layers of the coating in a conventional way. Flexographic printing enables multilayering and provides better barrier properties [13,14].

This paper demonstrates the use of latex-clay based barrier dispersion coatings containing a small addition (approximately 1.3%) of silica nanospheres applied by a multilayering flexographic printing/coating process. Use of silica nanospheres in combination with flexographic multilayering to low coating grammages provides a significant decrease in both the water vapor transmission rate and the direct water uptake. This represents a new approach in the development and performance of barrier dispersion coatings for paper-based materials [13,14]. Multilayering flexographic printing/coating of up to 6 layers was produced and evaluated in terms of coating runnability, coating grammage, surface characteristics, and moisture barrier performance.

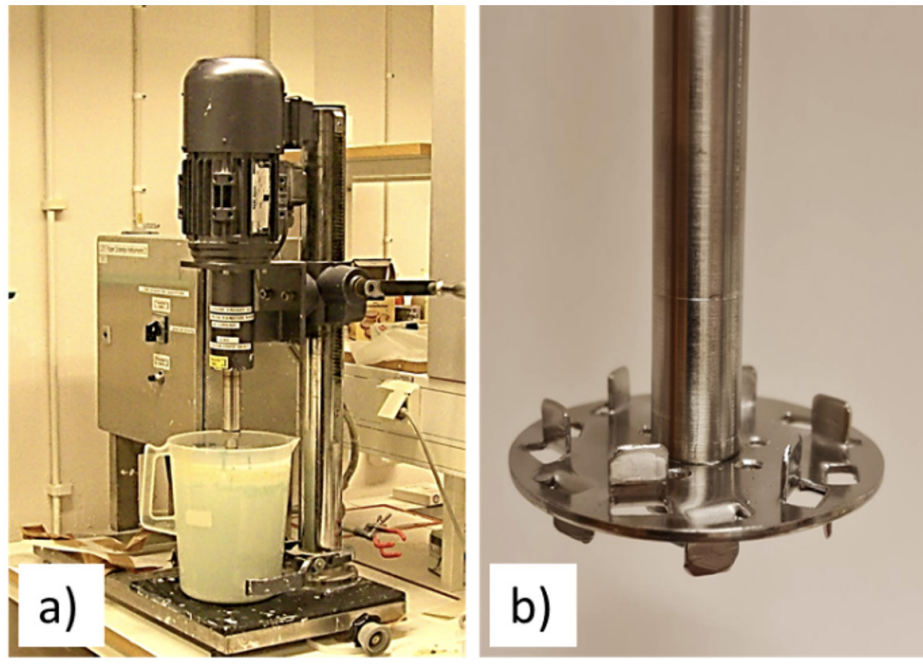
EXPERIMENTAL

Materials

A commercially available white linerboard (supplied by BillerudKorsnäs AB, Sweden) with a grammage of 132 g/m² was used in this study. The linerboard was coated/printed with a coating formulation comprising a binder, clay, talc, silica nanospheres, colorant, and antifoamer components.



2. Scanning electron microscope (SEM) image of silica nanospheres prepared according to the method of Stöber et al. [15], along with particle size distribution diagram.



3. High shear mixer (a) and impeller blade (b) used for preparation of coating formulations.

The clay was an Australian flatting grade kaolin clay used for paper coating that has good coverage, high brightness, and an average particle disc (plate) diameter of 2.33 µm, disc thickness of 0.067 µm, and density of 2.6 g/cm^3. The binder was a styrene-butadiene latex (DL699PA) from Trinseo Holdings Asia Pte. Ltd. (Singapore), supplied as a 50% dispersion with an average particle size of approximately 100 nm. The talc (PPEXP-069) was from Luzenac America (Houston, TX, USA), supplied as powder. The colorant was a water based colorant Aquabatch Blue #5, supplied by Pacific Inks Ltd. (Auckland, New Zealand). For some coatings, a small amount of wax and/or silica nanosphere dispersion was used. Wax emulsion (ME 36840R) supplied by Michelman (Cincinnati, OH, USA) is an emulsion containing paraffin and polyethylene at a pH of approximately 9.5. The silica nanosphere dispersion used in the study was prepared according to the method of Stöber et al. [15]. Solids content of silica dispersion was 14.34%, with most of silica particles/nanospheres in the size range of 80–110 nm, as shown in **Fig. 2**. A small amount of Defoamer Nopcomaster DF 122 (Nopco Paper Technology Australia; Rowville, VI, AUS) anti-foaming agent diluted (5% solids dispersion) was also used.

Preparation of coating formulations

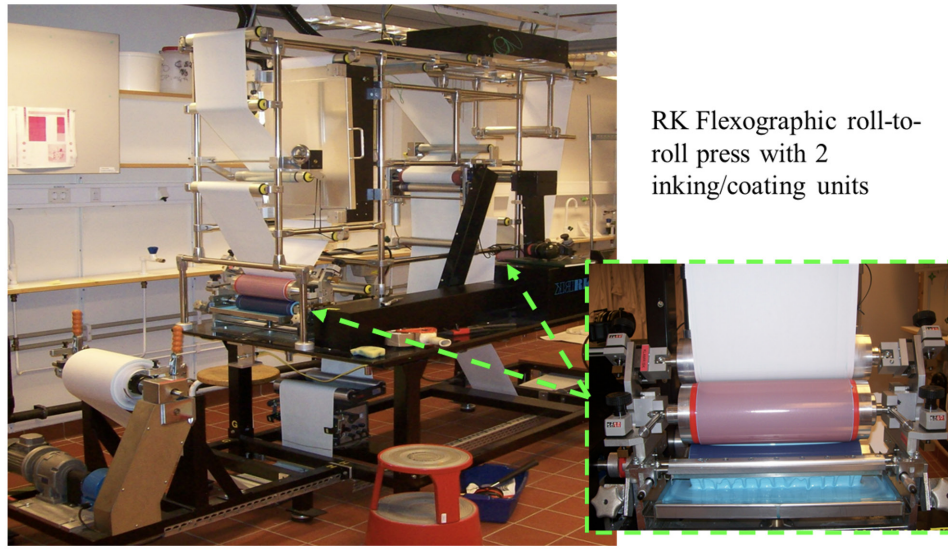
Four different coating formulations were prepared using a high shear mixer (DT Paper Science Instruments; Raisio, Finland) equipped with and impeller blade, as shown in **Fig. 3**, to provide sufficient mixing of silica nanospheres. Mixing speed ranged from 800 rpm at the beginning to 1800 rpm at the end of addition.

All four coatings were mainly based on kaolin clay, with two of these coating formulations containing a small amount of talc with and without wax, respectively, and the other two coating formulations containing a small amount of silica nanospheres with and without wax, respectively. **Table I** shows the four coating formulations. The finished coatings contained a pigment:latex ratio of approximately 47:53% d/d.

The first step of the preparation procedure involved mixing the dry kaolin clay powder with the latex binder, and the second step was the addition of talc or silica nanospheres, respectively, followed by the addition of wax. At the end of this preparation process, a small dose of an

Dry Material	T, pph	TW, pph	S, pph	SW, pph
Total pigment	100	100	100	100
Kaolin clay	87.2	87.2	97.3	97.3
Silica nanospheres	0.0	0.0	2.8	2.8
Talc	12.8	12.8	0.0	0.0
Wax	0.0	8.9	0.0	8.9
Latex	112.8	112.8	112.8	112.8

I. Recipes of the four coating formulations: T = coating containing talc; TW = coating containing talc and wax; S = coating containing silica nanospheres; SW = coating containing silica nanospheres and wax (pph = parts per hundred part of total pigment).



4. RK Flexographic roll-to-roll press with two inking/coating units at Karlstad University in Sweden.

anti-foaming agent solution was added to the formulation (~0.5 g per 1 kg of final coating formulation), followed by the addition of the colorant (~0.4 g per 1 kg of final coating formulation). The small dose of colorant was added for easier matching of the coating/printing stations. After the addition of all the components, the coating formulation was kept under stirring at 1800 rpm for 10 min. The final solids content of all the coatings was approximately 50 wt% and the final pH was 8.3–8.5. The viscosity of the coatings was in the range of 27 to 30 s, as measured by Zahn Cup #2 (corresponding to approximately 70–90 cP).

Coating process

All coatings were applied to the linerboard substrates using a flexographic roll-to-roll laboratory/pilot printing press (RK Printcoat Instruments Ltd.; Royston, UK) equipped with two in-line coating/printing units, as shown in **Fig. 4**, and located at Karlstad University in Sweden. Each unit was equipped with a ceramic anilox roll (120 lines/cm, 8 ml/m^2), a printing plate (DS2, 1.70 mm, 59 Shore A) from FlexoPartner AB Sweden (now Marvaco Oy, Helsinki), and a turbo air dryer/heater mounted directly after the unit. As the printing press was equipped with two in-line coating/printing units, three successive runs were completed to provide 6 coating layers for each formulation. The printing press setup was as follows: speed of 50 m/min, nip pressure, and drying air remained constant during the entire coating/printing process.

Characterization of coated samples

All coated samples were characterized in respect to their coat weight, WVTR, direct water uptake, and surface roughness. All characterization measurements were performed on samples preconditioned in a test environment maintained at standard ISO 23/50 conditions (23°C and 50% relative humidity). The results for each test are reported as mean ± standard deviation of six measurements.

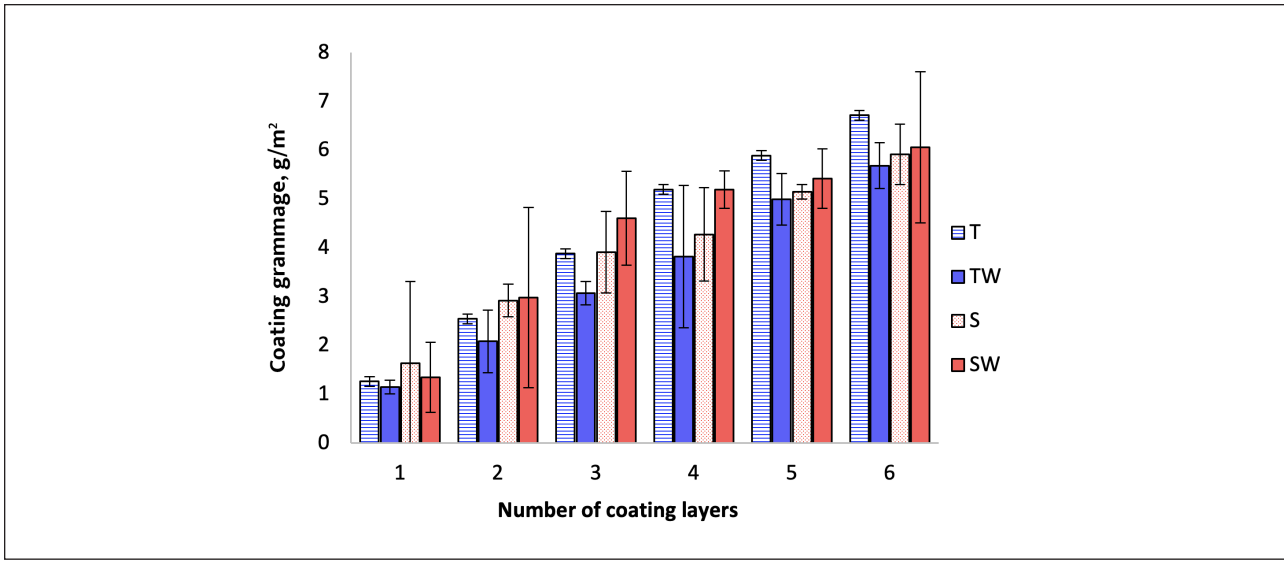
The coat weight (g/m^2) was determined according to the TAPPI Standard Test Method T 410 om-98 "Grammage of paper and paperboard (weight per unit area)." The WVTR (g/m^2/day) was measured at standard ISO 23/50 conditions (23°C and 50% relative humidity), adopting the ISO 2528:2017 "Sheet materials — Determination of water vapour transmission rate (WVTR) — Gravimetric (dish) method" approach by using dry calcium chloride ($CaCl_2$) flakes as the desiccant. For the Cobb 120 measurements (direct water uptake during 120 s), TAPPI T 441 om-98 "Water absorptiveness of sized (non-bibulous) paper, paperboard, and corrugated fiberboard (Cobb test)"/ISO 535:1991 "Paper and board — Determination of water absorptiveness — Cobb method" was followed.

Surface roughness and profiles were obtained using a Wyko NT 3300 surface profiler from Veeco Metrology Group (Veeco; Plainview, NY, USA). All characterization measurements were performed in a test environment maintained at standard ISO 23/50 conditions (23°C and 50% relative humidity).

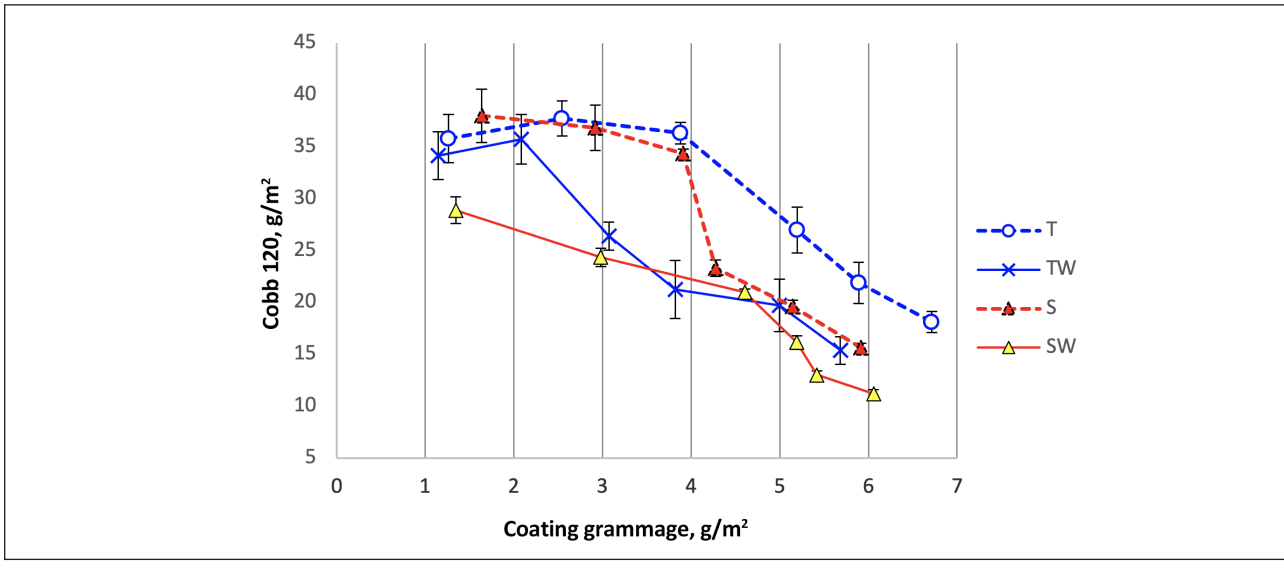
RESULTS AND DISCUSSION

Runnability along with the barrier properties of the coatings printed/coated onto the white top linerboard were evaluated during 4 h runtime. No major runnability issues were encountered during the printing/coating trials. There was no building-up or blocking of the cylinders — anilox or on the roll. Cleaning of the equipment at the end of the trial was easy.

Figure 5 shows the coat weights as a function of the number of coating layers. As expected, the coat weight increases with the number of layers. In general, there was no significant difference in the final applied coat weight be-



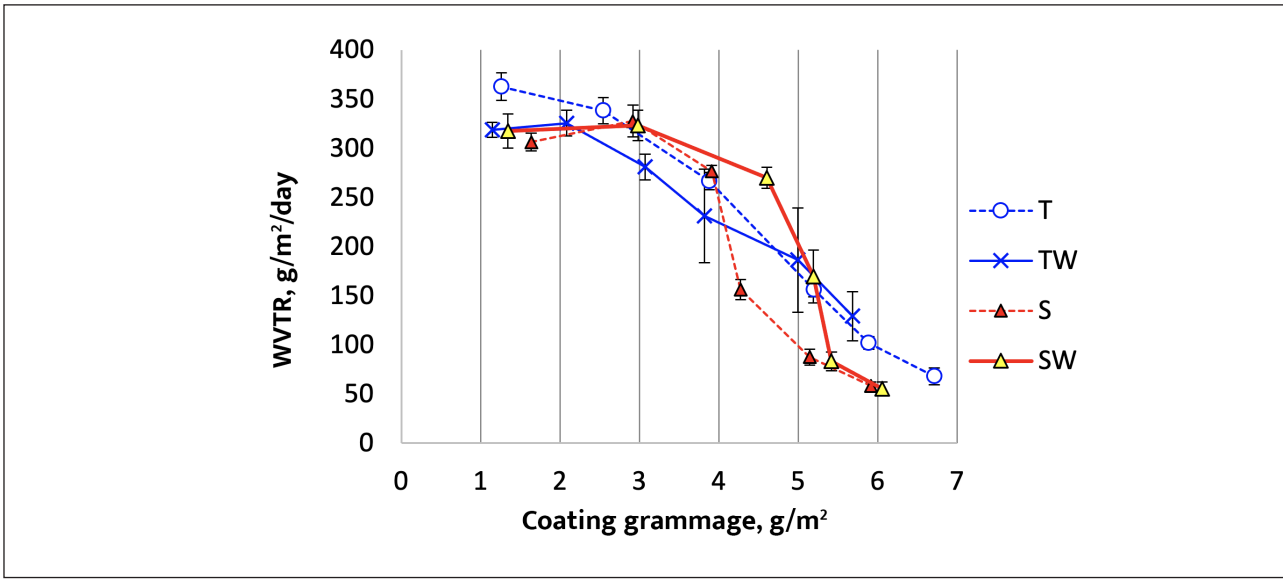
5. Coating grammage (g/m²) as a function of the number of coating layers (T = coating containing talc; TW = coating containing talc and wax; S = coating containing silica nanospheres; SW = coating containing silica nanospheres and wax).



6. The Cobb 120 values as function of the coating grammage (corresponding to number of coating layers).

tween different coatings, indicating similar coating transfer for all coatings. However, for the 5 and 6 layers, there were some significant differences between coating containing talc (T) compared with coating containing talc and wax (TW) and coating containing silica particles (S). Coating T showed slightly higher coating transfer to the substrates than coatings TW and S. In addition, the coating containing silica nanospheres and wax (SW) was in the same range as coatings TW and S, with the differences not being significant.

The barrier properties of the coating were evaluated in the form of direct water uptake (Cobb 120) and moisture barrier (WVTR). **Figure 6** shows the results of the Cobb 120 measurements for all coated samples as a function of coating grammage. The number of coating layers is also directly related to coating grammage. In general, for all coated samples, the Cobb 120 value decreased with increasing coating grammage (number of coating layers). After applying the first coating layer, the Cobb 120 value for the SW coating (corresponding to 1.34 g/m² coating grammage) is significantly lower than for all other coatings. For the TW coating, after applying about 3 g/m² of coating, the Cobb 120 value decreased to the same range as for the SW coating. However, to achieve coating grammage of about 3 g/m², 2 layers of the SW coating were needed, while for the TW coating, 3 layers were needed to achieve a similar coating grammage. This can be explained by a bigger coating transfer to the substrate (per coating layer) for the SW coating than for the TW coating. For the T and the S coating, the Cobb 120 value is the same for the first 3 coating layers. After applying 4 coating layers, the Cobb 120 value



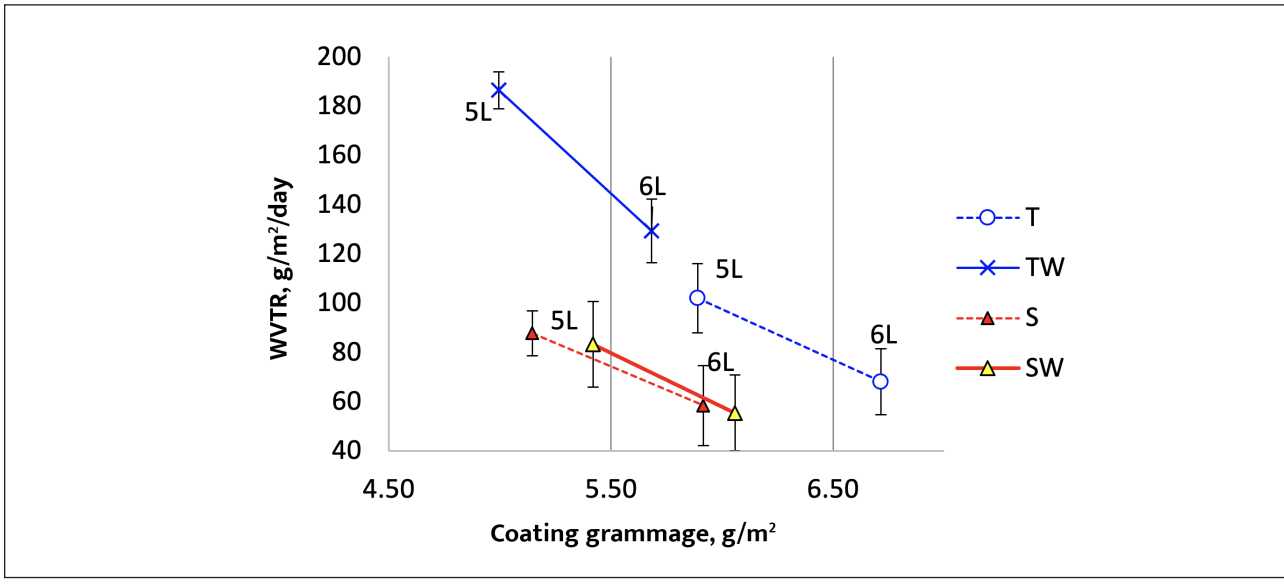
7. The water vapor transmission rate (WVTR) values as a function of the number of coating layers (coating grammage).

is much lower for the S coating (4 layers corresponding to 4.3 g/m² coating) than for the T coating (4 layers corresponding to 5.4 g/m² coating).

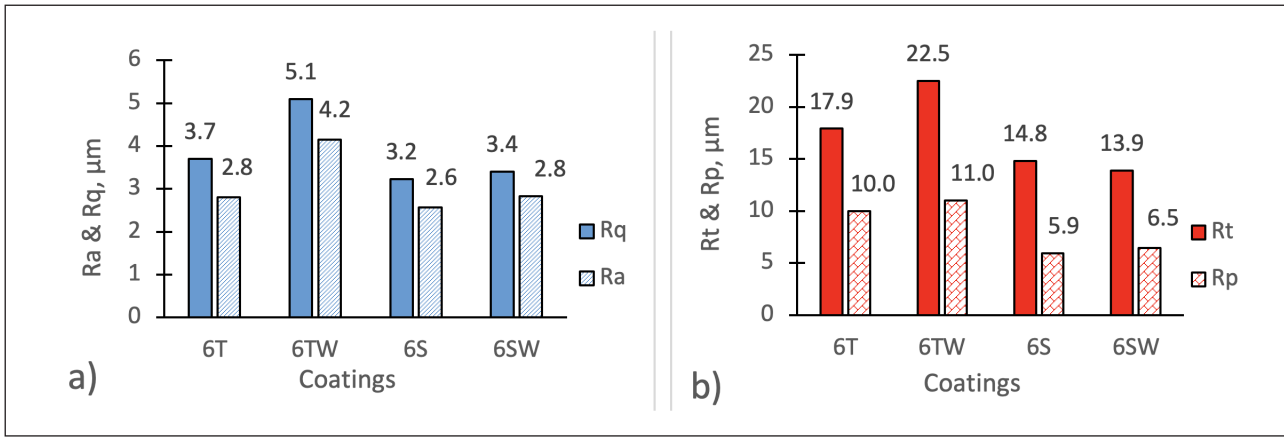
After applying 5 layers of the coating, the Cobb 120 values for the TW coating (5 g/m² coating) and the S coating (5.2 g/m² coating) are in the same range and much lower than for 5 layers of the T coating (5.9 g/m2 coating), while this is not the case for the SW coating. The Cobb 120 value for the SW coating is significantly lower already after applying 4 coating layers (corresponding to 5.4 g /m² coating). However, when comparing only the T and the TW coatings (overall view), there is a significant difference where the Cobb 120 value is lower for the TW coating containing a small amount of wax. The same is evident when comparing the S and SW coatings, where the Cobb 120 value is significantly lower for the SW coating containing a small amount of wax. Our findings are supported by the results presented by Mesic et al. [13], where wax-based coatings, multilayered by flexography had lower Cobb 120 values than wax-free coatings. Schuman et al. [6] reported migration of the wax to the surface and a corresponding improvement in coat quality of dispersion coatings containing wax. In addition, the six layers of SW coating (corresponding to 6.1 g/m² coating) show the lowest Cobb 120 value when compared with all other coatings. It seems that in addition to the desirable hydrophobic effect of the wax, a small amount of silica nanospheres incorporated into latex-clay based coatings fills the spaces and voids between clay particles, providing an additional mechanical barrier that results in a more desirable lower Cobb 120 value.

The results of the barrier performance of the coatings measured by WVTR as a function of coating grammage are presented in **Fig. 7**. The number of coating layers are again related to the coating grammage. In general, the WVTR values decrease steadily with the increasing coating grammage for all coating samples. An overall view of the barrier performance is that the WVTR values for the S and SW coatings were in about the same range for 5 coating layers (corresponding to 5.2 g/m² and 5.5 g/m² coating, respectively) and for 6 coating layers (corresponding to 5.9 g/m² and 6.06 g/m² coating, respectively), while for the same number of coating layers (corresponding to 5.0 g/m² and 5.7 g/m² coating, respectively), the TW coating showed a much higher WVTR value than all other coatings. A closer view of the 5 and 6 coating layers (**Fig. 8**) shows that a better WVTR performance was obtained for 6 coating layers containing silica nanospheres for S and SW (about 58 g/m²/day and 55 g/m²/day, respectively), while the worst WVTR performance was obtained for the TW coating (129 g/m²/day).

Finally, we can say that the 6 thin coating layers with a total coating grammage of about 5.9–6 g/m² coating gave significantly good WVTR performance for the S and SW coatings. The T coating also show a low WVTR value for 6 coating layers, but the coating grammage of it was higher than for the S and SW coatings. Our results are supported by work presented elsewhere [13,16]. For the light coat weights, the clay plates are better aligned and provide a more difficult and tortuous pathway for the diffusion of water vapor through the layer-by-layer formation of the coating, achieving low WVTR values. In addition, for the S and SW coatings, the small amount of silica nanospheres incorporated in latex-clay based coatings most likely fill the spaces and voids between clay particles, providing additional physical barrier to the diffusion of water vapor, resulting in a slightly better WVTR barrier. Cairns et al. [17] reported that the presence of spherical silica particles provides beneficial attributes to the interior structure of the coated films, resulting in a better WVTR value. The slightly worse WVTR barrier properties for the TW coatings (con-



8. The WVTR values for 5 and 6 coating layers/coating grammage (5L = 5 coating layers; 6L = 6 coating layers).



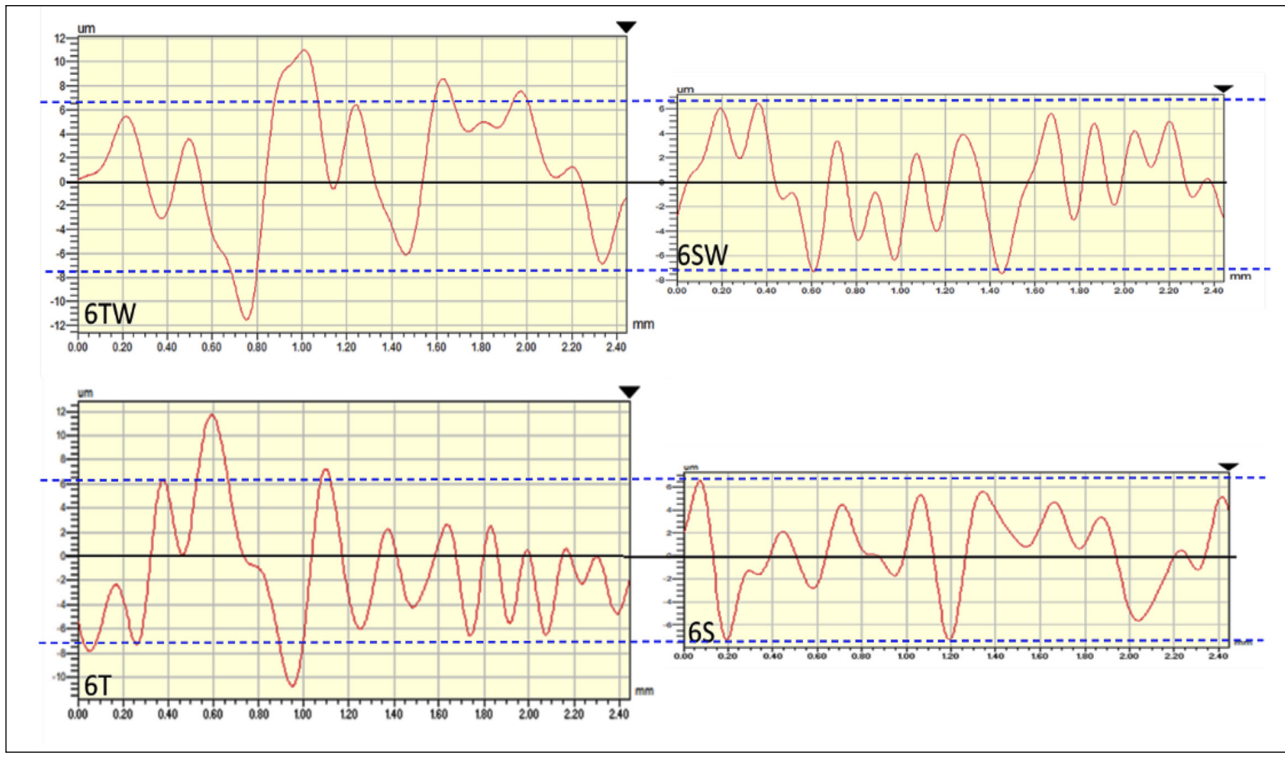
9. (a) Surface roughness of the different coatings in form of average surface roughness (Ra) and mean square average roughness (Rq), and (b) maximum profile peak height (Rt) and maximum height of the profile (Rp).

taining a small amount of wax) could probably be related to the uneven distribution of the coating layers as result of hydrophobicity, surface tension, and viscoelasticity [14] of the coating during application and drying.

Figure 9 presents the surface roughness after 6 layers were applied in the form of average surface roughness (Ra), mean square average roughness (Rq), maximum profile peak height (Rt), and maximum height of the profile (Rp). The surface roughness parameters were filtered by using digital Fourier transforms and low pass filter (LPF) with 10/mm cutoffs. The LPF cutoff 10/mm filter corresponds to middle scale variations greater than 100 µm [18,19].

Figure 9a shows that the highest surface roughness in form of Ra and Rq were for the TW coating. The roughness for all other coatings (T, S, and SW) was quite low, with no significant differences between them.

Figure 9b shows the middle scale (Rt) and maximum height (Rp) profiles. The middle scale variation in form of Rt was largest for the TW coating; it was slightly lower for the T coating and was lowest for the S and SW coatings. The variations in form of maximum height of the profile Rp were higher for both talc-based coatings (T and TW) when compared with silica nanosphere coatings (S and SW). Both the T and W coatings were in the same range. The same behavior is evident when comparing S and SW coatings. The highest variation for the TW coatings is probably related to hydrophobicity, surface tension, and viscoelasticity, resulting in uneven distribution and variations of TW coating layers [14]. Conversely, the silica nanosphere-based coating (SW) with a small dose of wax had similar roughness properties to the silica nanosphere-based coating (S) without wax, which could likely be related to the alkalinity of the silica nanospheres. It seems that the silica nanospheres have a bigger impact on the even distribution of the coating across the paperboard substrate surface than the wax does. The silica nanospheres dispersion is slightly



10. Surface profile (XY → 2.4 mm x 1.9 mm) for 6 layers of different coatings.

alkaline, which promotes a more even dispersion of both silica nanospheres and the clay, as well as a more even distribution and deposition of the coating itself across the paperboard. Cairns et al. [17] reported that for latex coatings incorporated with clay and silica spheres, there was an even dispersion and distribution of the silica nanospheres and clay particles across the coating surface and within the coating itself.

The surface profiles of the coated layers presented in **Fig. 10** are consistent with the roughness trends presented in Figs. 8a-8b. The profile variations were largest for the TW coatings and lower for the T coating, and they were lowest for the S and T coatings. For the TW coatings, the surface profile is more like "waviness" that can likely be explained by an uneven distribution of the hydrophobic property provided by the wax.

CONCLUSIONS

No major runnability issues were encountered during the printing/coating trial with the 4 experimental coatings tested in this work. No building-up or blocking of the cylinders, anilox, and on the roll occurred.

The coat weight increased with an increase in the number of layers. In general, there was no significant difference in the final applied coat weight between the different coatings, indicating a similar coating transfer for all coating layers.

For all coated samples, the Cobb 120 values decreased with increasing number of coating layers, and hence increased coating grammage. In general, both wax-based coatings (TW and SW) had lower Cobb 120 values when compared with respective coatings without wax (T and S), which is a result of the hydrophobicity of the wax-based coatings.

The Cobb 120 value for the silica nanosphere-based coating containing a small amount of wax (SW) was significantly lower than all other coatings, especially at low grammage, which is a result of both the wax hydrophobicity and the additional physical barrier provided by silica nanospheres.

The WVTR performance for the S and SW coatings was in the same range and much lower than that for the TW coating, which is a result of the additional physical barrier provided by silica nanospheres. A higher WVTR for the TW coating could be related to the uneven distribution of the coating layers as result of hydrophobicity provided by the wax.

The T coating also showed a low WVTR value for 6 coating layers, but the coating grammage of it was higher than it was for the S and SW coatings.

The S and SW coatings had the lowest surface roughness and profile variation, while the TW coating had the highest variation of the surface roughness and profile, which is probably related to uneven distribution and variations of coating layers as a result of hydrophobicity provided by wax. **TJ**

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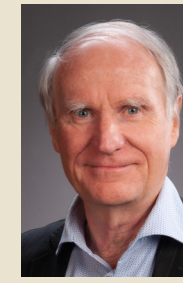

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We chose this research topic as a means of developing a novel approach to development and application of barrier coatings. This research is in line with our previous research and offers a potential solution for the industry in this area.

The most difficult aspect of this study was scaling up of preparation and application of coating formulation. For the application, we went from bench equipment to a pilot roll-to-roll web coater, which more closely mirrors commercial application. It was hard, but we did find adequate pilot equipment. Our most interesting finding was that despite hydrophobicity and the use of nanomaterials, it was possible to do multilayering of coating.

For mills, the information presented here offers a potential alternative solution for off-line coating that avoids issues with applying several layers of hydrophobic coatings using a traditional coater. Our next step is commercial trials.

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