

## Top layer coatability on barrier coatings

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### ABSTRACT

The influence on top layer coatability by different surface characteristics of dispersion coated barrier layers was studied. The barrier layer consisted of platy pigments such as talc and kaolin combined with different amounts and types of latexes. Coating of the top coat was carried out using reverse gravure technique where the dispersion consisting of mineral pigments and latex is applied under slight pressure onto the barrier coated substrate. Wettability and coatability were measured as a function of surface energy and surface roughness of barrier layer and surface tension of the top coat dispersion. Plasma and corona surface treatments were used for increasing surface energy and wettability without affecting surface topography. The most important aspect to predict coatability was the wettability and especially the water contact angle since the top coat is an aqueous solution. Decreasing the surface tension of top coat dispersion also improved coatability whereas roughness had only a minor effect.

### INTRODUCTION

In conventional paper coating, mineral pigments, such as clay or talc, are used to improve the print quality, gloss, brightness and opacity of the paper product. The use of mineral pigments in dispersion coating to provide improved barrier properties has recently gained increased attention [1–9]. Dispersion coating has also been claimed a more environment-friendly alternative with regard to composting and repulping than conventional lamination or extrusion coating [10,11]. Different types of dispersion polymers, commonly known as latexes, have been used for improving barrier properties against humidity, grease and oxygen. One challenge of dispersion coated surfaces has been poor printability due to the low surface energy [12–21]. The print quality, especially when using water-based inks has been poor, resulting in smearing, ink refusal and print mottle, due to improper ink transfer or adhesion [12–28]. This can to a certain extent be counteracted by a surface treatment, such as corona or plasma, but the discharge treatment has been reported to decrease the barrier properties [27,28]. A new method for improving the wettability is use of UVC radiation. The germicidal wavelength of 254 nm has been shown to degrade organic compounds such as dispersing agents from the pigment surface resulting in an increased polarity [29,30].

New value-added products with novel functionalities, e.g. paper or board based printed devices (sensors, displays etc.) are drawing more attention. For these to come into everyday use, devices with reasonable electrical performance and practically negligible production cost are required. A multilayer-coated paper-based substrate that is suitable for printed electronics and functionality has been developed [31,32]. In this multilayer structure, shown in figure 1, a thin topcoating consisting of mineral pigments is coated on the top of a dispersion coated barrier layer. The topcoating provides well-controlled sorption properties through controlled thickness and porosity thus enabling optimized printability of functional materials. The penetration of ink solvents and functional materials stops at the barrier layer, which not only improves the performance of the functional material, but also eliminates potential fiber swelling and de-bonding caused if solvents are allowed to penetrate into basepaper [33].

From manufacturing point of view, the surface properties of the barrier layer and the surface tension of the top coat formulation must be well understood. Incompatibility of barrier and topcoat material might cause insufficient wetting of the barrier layer by the top coat dispersion, which leads to uneven top coat thickness or uncoated spots. The objective of this study was to understand the required surface properties of the barrier layer in order to make coating of the top coat possible. Surface energy of the barrier layer was studied, with main focus on water contact angle since the topcoat is an aqueous solution. Furthermore, the influence of surface tension of top coat dispersion on coatability was investigated.

## MATERIALS AND METHODS

The multilayer coating structure under consideration in the current work consists of a precoating and a smoothing layer, on which the barrier layer is coated. The thin and porous topcoating provides the final smoothness and controllable sorption properties. The coatability of the topcoating layer on the barrier layer is studied by varying the materials in the latter with the aim of creating as different as possible surface characteristics in terms of surface energy and surface roughness. Additional changes in the surface energy of the barrier layer were induced by using different surface treatment methods, such as plasma, corona and UVC radiation. An alternative way to influence the wetting properties is controlling the surface tension of the topcoat dispersion, which in this study was decreased by addition of iso-propanol.

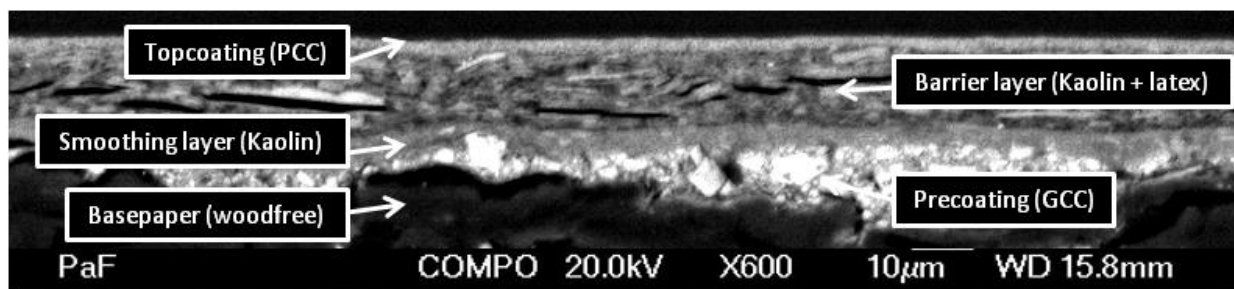


Figure 1. Cross-section SEM image showing the layer structure, topcoating (reverse gravure), barrier layer (reverse gravure), smoothing layer (blade), precoating (blade) and basepaper.

### Barrier layer formulation

A precoated basepaper ( $107 \text{ g/m}^2$ ) was first blade coated with a  $10 \text{ g/m}^2$  kaolin layer to smoothen the surface. On this smoothing layer various barrier layers were coated. The used pigments in the barrier layer were talc (Mondo Minerals B.V., C10B) and kaolin (Imerys Minerals Ltd., Barrisurf HX) both having a shape factor higher than 50. As barrier latexes two styrene-acrylate copolymer latexes were used, latex SA1 (Ciba Finland Oy) (particle size 130 nm and  $T_g 7,5^\circ\text{C}$ ) and latex SA2 (Styron Europe GmbH) (particle size 110 nm and  $T_g 0^\circ\text{C}$ ). Latex Exp1 and Exp2 are experimental latexes (Styron Europe GmbH). The barrier coatings were carried out using reverse gravure technique on a MiniLabo (Yasui Seiki Co.) laboratory scale reel to reel coater. Table 1 shows the experimental design for the barrier layer. The goal was to create substrates with as different as possible surface properties, including surface energy, hydrophilicity and roughness, while maintaining adequate barrier properties. The critical pigment volume concentration (CPVC) for both the talc and the kaolin is at approximately 30 pph latex addition level (equals a pigment volume concentration (PVC) of 57%). Samples with an addition level of 10 pph latex, which is less than the CPVC, had insufficient barrier properties as well as uneven coating layers. The barrier layer thicknesses are calculated from the coating grammage assuming zero porosity.

Table 1. Experimental design for the barrier layer. The thicknesses are calculated from grammage assuming zero porosity.

Filler	Binder	Binder amount (pph)	Grammage (g/m <sup>2</sup> )	Thickness (μm)
Talc	SA1	10	-	-
Talc	SA1	30	39,6	21,3
Talc	SA1	50	32,4	19,4
Talc	SA1	70	30,9	20,0
Kaolin	SA1	10	-	-
Kaolin	SA1	30	40,4	21,8
Kaolin	SA1	50	34,4	20,6
Kaolin	SA1	70	23,6	15,3
Kaolin	SA2	10	-	-
Kaolin	SA2	30	35,4	19,1
Kaolin	SA2	50	30,3	18,8
-	SA1	100	23,3	23,3
-	Exp1	100	33,9	33,9
-	Exp2	100	25,0	25,0

### Top coat formulation

The top coat formulation consisted of aragonite PCC (Specialty Minerals Nordic Oy, Opacarb 3000) combined with 10 pph of styrene-butadiene latex (Styron Europe GmbH, DL 920) as binder. In order to make the top coat layer as thin as possible the dry solids content was lowered to 44%. The Brookfield viscosity was adjusted to 500 mPas by addition of 2,5 pph of carboxy methyl cellulose (CMC) (Noviant Oy, Finnfix 5). The surface tension of the dispersion was 49,4 mN/m. In order to decrease the surface tension, water was partially replaced by isopropanol. A replacement of 2,5% of water gave a surface tension of 43,3 mN/m while a replacement of 20% gave 35,2 mN/m. Reverse gravure coating technique was used, since it enabled coating of thin (7 g/m<sup>2</sup>) top coat layers onto the barrier layer. Blade coating of such thin top coats resulted in all top coat dispersion being scraped off from the barrier layer and was therefore impossible to use.

### Surface treatment

Both corona and plasma treatments were performed in pilot scale in a roll-to-roll process at normal atmosphere (air). In the corona treatment plasma state occurs between two electrodes, of which one is a grounded metal roll under the substrate. The atmospheric plasma is generated using a dielectric barrier discharge. The treatment gas, argon in this case, is fed between the electrodes and the grounded back up roll where the treatment gas breaks down due to the high voltage electric field, resulting in nonequilibrium plasma rich in excited and electronic states. The substrate to be treated is fed through the plasma. Electrode design, suitable power supply, impedance matching and controlled gas flow help to minimise the filamentary discharges, which are typical for a corona discharge [34]. Both corona and plasma were run at low and high power levels. Table 2 lists the treatment parameters. UVC surface treatment was performed using a germicidal ozone free UVC lamp ( $\lambda_{\text{max}}$  254 nm) supplied by Heraeus. The used irradiation intensity was 50 mW/cm<sup>2</sup> at 254 nm for one minute.

Table 2. Parameters for pilot scale corona and argon plasma surface treatments.

Treatment	Power	Speed	Width	Efficiency value	Gas feed	Frequency
Corona low (CL)	1300 W	50 m/min	500 mm	52 Wmin/m <sup>2</sup>	-	24,8 kHz
Corona high (CH)	1300 W	20 m/min	500 mm	130 Wmin/m <sup>2</sup>	-	24,8 kHz
Plasma low (PL)	780 W	50 m/min	380 mm	41 Wmin/m <sup>2</sup>	30 l/min	28,3 kHz
Plasma high (PH)	780 W	20 m/min	380 mm	103 Wmin/m <sup>2</sup>	30 l/min	28,3 kHz

#### Contact angle, surface energy and surface tension measurement

The contact angles (three parallel measurements, standard deviation  $< 2^\circ$ ) were measured in ambient conditions (RH =  $22 \pm 3\%$ , T =  $22 \pm 2^\circ\text{C}$ ) using a CAM 200 contact angle goniometer (KSV Instruments Ltd). The contact angles were calculated as a function of time using the software supplied with the instrument, which utilizes both a circular and a Laplace fit to the projected drop curvature. Main focus was set on contact angles for water, and the values presented in this article were read 0.5s after the first contact between the paper and the drop. This time was sufficient for droplet stabilization on the surface. In order to avoid the time dependent decay of surface energy of the treated surfaces all contact angles were measured within two days after the treatment. For calculating the surface energy the apparent contact angles of the probe liquids, water, ethylene glycol (EG), and diiodomethane (DIM) were measured. The calculations were performed using the theory proposed by Owens and Wendt [35], Kaelbe [36] and Fowkes [37] subdividing the surface energy into polar and dispersive parts. No roughness corrections were done. The surface tension was measured according to the pendant drop method and Young-Laplace equation using the same CAM 200 goniometer.

#### Surface topography characterization

Surface roughness was measured using a Parker Print Surf (PPS) tester manufactured by Lorentzen & Wettre. The measurement interval of the PPS roughness is  $0,6 - 6 \mu\text{m}$ . A JEOL JSM-6335F field emission scanning electron microscope (SEM) was used for surface visualization. A NTEGRA Prima (NT-MDT) atomic force microscopy (AFM) was also used for analyzing the topography of the samples. Topographical imaging was carried out in semi-contact mode. All the images ( $1024 \times 1024$  pixels,  $20 \times 20 \mu\text{m}$  and  $100 \times 100 \mu\text{m}$ ) were measured in ambient conditions (RH =  $24 \pm 3\%$ , and RT =  $24 \pm 1^\circ\text{C}$ ). The Scanning Probe Image Processor (SPIP, Image Metrology) software was used for the roughness analysis of the images.

#### Water Vapor Transmission Rate (WVTR) Measurement

The barrier properties were measured using the cup method according to the ASTM (E 96/E96M-05) standard. The used conditions were relative humidity of 85% and temperature of  $23^\circ\text{C}$ . Two parallel measurements were performed during three days each.

#### Evaluation of coatability

The result of the coated topcoat (reverse gravure) layer was visually evaluated on a scale from 1 to 4. Figure 2 shows examples of the coating layer quality. On this scale grade 1 means no wetting, grade 2 inadequate wetting resulting in areas being uncoated, grade 3 small defects, which means on the limit to required wetting, and grade 4 proper wetting and adequate coatability. From research point of view grade 3 is the most interesting one indicating the wetting is slightly improper. From manufacturing point of view only grade 4 is acceptable and results in a defect free coated layer.

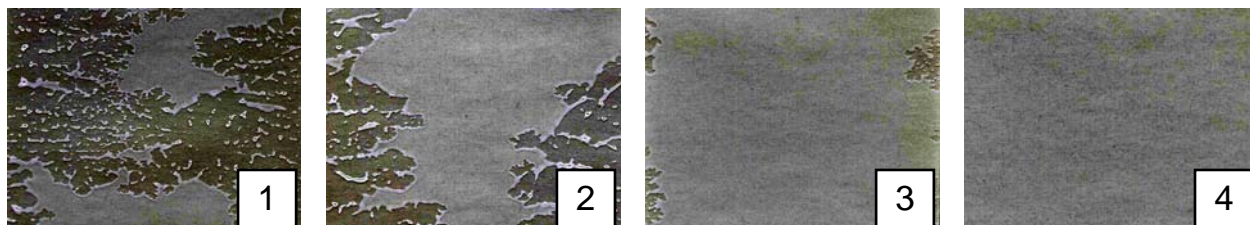


Figure 2. The result of the coatability was visually evaluated on a scale from 1-4. The contrast is artificially increased to better show the differences between the barrier and topcoating layers.

## RESULTS

### Barrier layer properties

#### Surface chemistry

Figure 3 shows example SEM images of the barrier layer surfaces. In cases where filler pigments are used, they are clearly visible on the barrier layer surface. Table 4 summarizes the PPS and AFM surface roughness measurements as well as water contact angles for the barrier coating layers. The contact angles were measured for all the samples both as untreated and as a function of surface treatment method and power. Since talc is a hydrophobic pigment the water contact angles on those barrier layers using it as filler were ca 20° higher when compared to kaolin. Differences between the latexes can also be observed. The SA2 latex generally gave a lower water contact angle than the SA1 latex. Latexes were used in different amounts and for amounts over 30 pph (below CPVC) the latex seemed to be the dominating material determining the water contact angle. Both plasma and corona surface treatments were carried out in pilot scale at two different power levels. Both surface treatment methods increased the surface energy; the high power level of both decreasing the contact angle for water on average with 21° and the low power level with 12°. The largest changes in surface energy could be induced when kaolin was used as filler pigment. The effect of surface treatment on the surface energy was smallest with talc and the coatings having latex contents higher than 50 pph. UVC treatment was also tested but its effect on surface energy was minimal.

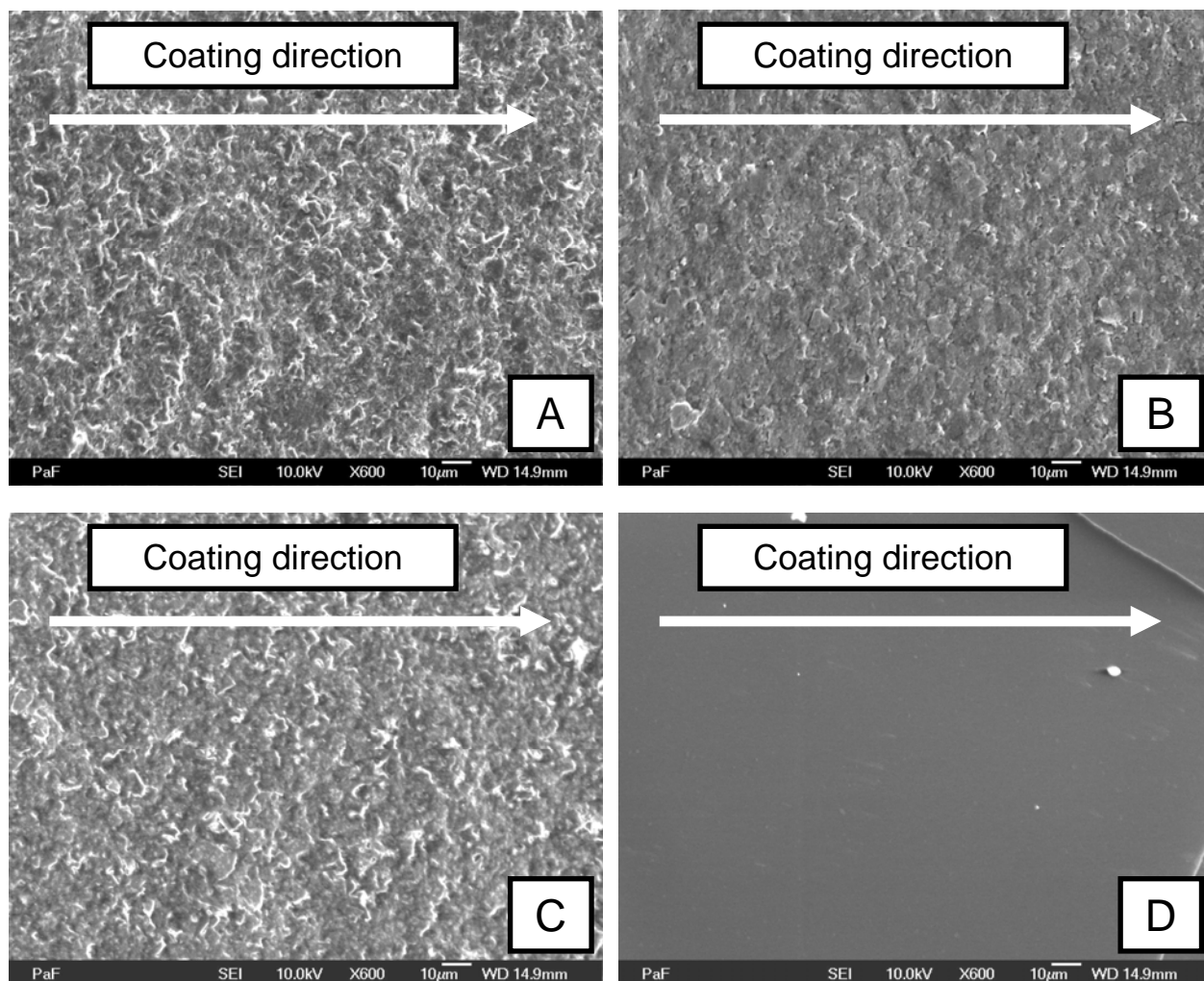


Figure 3. SEM images of the surfaces, A: Kaolin + 30 pph SA1 latex, B: Kaolin + 30 pph SA2 latex, C: Talc + 30 pph SA1 latex and D: 100 pph Exp2 latex. Cracks in the barrier layer (D) are caused by sample preparation for SEM-imaging.

The water contact angle seems to be almost linearly dependent on surface energy as can be seen in table 3. The table also lists the dispersive (d) and polar (p) components of the surface energy. It appears that both the kaolin and the latex SA2 increase the polarity of the barrier layer surface. As expected using talc as the filler results in the lowest surface energy.

Table 3. Surface free energy, dispersive and polar components for the surfaces shown in Figure 3

Barrier layer formulation	Figure 3	CA° (water)	Surface energy (mN/m)		
			d	p	tot
Kaolin + 30 SA 1	A	95	21,23	2,29	23,51
Kaolin + 30 SA 2	B	84	17,35	7,13	24,49
Talc + 30 SA 1	C	114	18,88	0,07	18,94
100 pph Exp 2	D	105	20,3	0,72	21,01

Table 4. Roughness and contact angle for water for the differently surface treated substrates. UT=untreated, CH=corona high, CL= corona low, PH= plasma high and PL=plasma low.

Barrier layer formulation		PPS	AFM Roughness		WVTR	Water contact angle (°)				
Pigment	Binder	( $\mu\text{m}$ )	RMS 20 (nm)	RMS 100 (nm)	( $\text{g}/\text{m}^2/\text{day}$ )	UT	CH	CL	PH	PL
Talc	10 SA 1	1,12	-	-	-	100	72	81	74	76
Talc	30 SA 1	2,48	268	508	8	114	103	109	93	108
Talc	50 SA 1	2,03	-	-	13	113	107	108	97	109
Talc	70 SA 1	1,75	-	-	14	113	107	108	103	109
Kaolin	10 SA 1	1,37	-	-	-	76	45	51	49	52
Kaolin	30 SA 1	2,15	212	611	36	95	33	72	46	45
Kaolin	50 SA 1	2,10	228	454	16	113	47	97	70	92
Kaolin	70 SA 1	1,78	-	-	-	116	100	110	93	109
Kaolin	10 SA 2	2,82	-	-	-	60	47	52	50	52
Kaolin	30 SA 2	1,46	222	427	89	84	78	79	76	79
Kaolin	50 SA 2	1,10	150	249	79	81	79	80	78	80
-	100 SA 1	0,86	37	-	0	113	108	109	104	110
-	100 PE 1	0,73	-	-	-	109	92	95	91	98
-	100 PE 2	0,61	-	-	-	105	83	87	88	94

## Surface topography

Surface roughness was measured for all the surfaces both as untreated and after each surface treatment method and power level. Neither plasma nor corona had an impact on the PPS roughness. In addition to PPS surface roughness measurements SEM and AFM images were acquired from the surfaces in order to provide more detailed information. The SEM images in figure 3 are taken at a 30 degree grazing angle along the coating direction. Figure 3A shows the surface of kaolin combined with 30 pph SA1 latex and figure 3 B the surface of kaolin combined with 30 pph SA2 latex. The high contrast in Figure 3A is probably due to the charging of mineral pigment edges. Differences in particle alignment can be observed, for kaolin combined with SA1 latex (figure 3 A and C) the particles seem not to be fully horizontal. The edges of the particles in the coating direction seem to be slightly higher in Z-direction. The PPS roughness decreased with an increasing amount of latex, the pure latex barrier resulting in the smoothest surface. The formulations containing only 10 pph latex (a level above CPVC), were generally uneven and roughness variations therefore large. No significant differences regarding roughness could be observed between the talc and kaolin coating layers. AFM topography images were also acquired from the surfaces and RMS roughness calculated from both  $20 \times 20 \mu\text{m}$  and  $100 \times 100 \mu\text{m}$  images. The trends were the same as for the PPS, higher amounts latex resulting in reduced roughness (Table 4).

## Barrier properties

Since different surface treatment methods in earlier studies [28] were reported to decrease barrier properties, WVTR measurements were performed to see whether this was the case in this study. The WVTR for all coatings was between 0-90  $\text{g}/\text{m}^2/\text{day}$  except when only 10 pph latex was used. The difference in barrier properties between talc and kaolin was minimal but between the latexes there was an obvious difference. The more hydrophobic SA1 latex gave significantly higher barrier properties than the less hydrophobic SA2 latex. The highest barrier properties with zero penetration were achieved with pure SA1 latex coating. Probably because of the thick barrier layers used, no changes in barrier properties could be observed as a result of any treatment method or power level.

## Coatability

### Visual evaluation

The barrier layers listed in table 4 were coated with the top coat formulation to see the influence of surface chemical and topographical properties of the barrier layer on coatability of the top coating. In addition, the surface tension of the top coat dispersion was investigated. The coatability was quantified on a scale from 1 (worst) to 4 (best) through visual evaluation of the topcoat quality as described in the Methods section. Figure 4 shows the coating result as a function of water contact angle on barrier layer and as a function of surface tension in top coat dispersion.

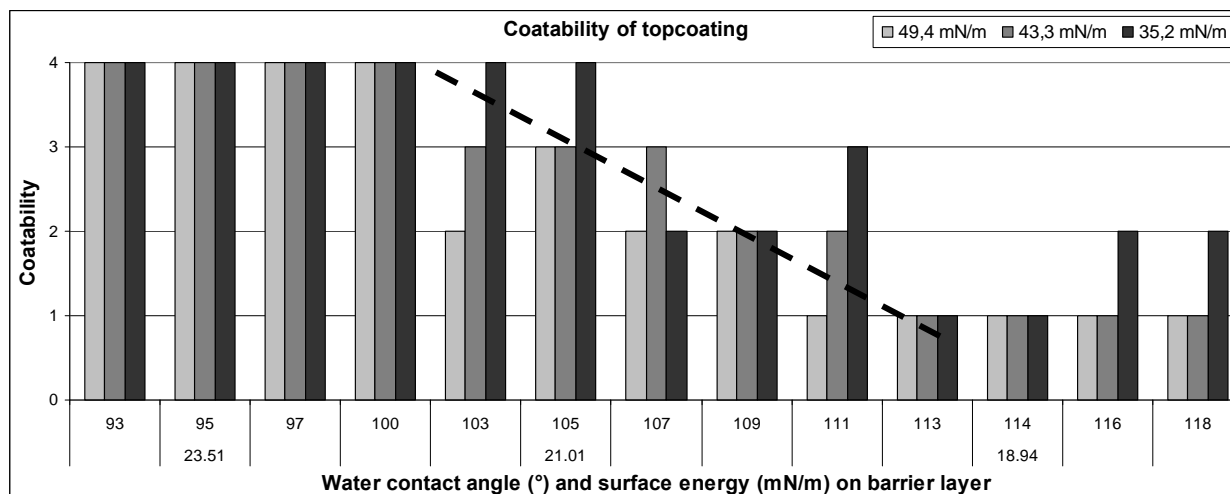


Figure 4. Top coat coatability versus contact angle for water on barrier layer. Three different surface tensions in top coat dispersion were used, 49,4 mN/m, 43,3 mN/m and 35,2 mN/m. Surface free energy was calculated at three points, 95°, 105° and 114° water contact angle.

The water contact angle of the barrier layer can be considered to be the most important factor predicting coatability. On barrier layers having water contact angles less than 100° the coating was possible with all the studied top coat dispersions. In the water contact angle range from 100° to 110° there is a variation in coatability and the effect of the reduced surface tension in top coat dispersion can be observed. When the water contact angle on barrier layer reaches 110° the coating of the top coat in an aqueous solution becomes impossible (Figure 4).

The surface tension in the top coat dispersion was adjusted by replacing water partially with iso-propanol. A lower surface tension in top coat solution increases wetting and improves therefore the coatability. Especially in the water contact angle range from 100° to 110° on the barrier layer a lower surface tension clearly improved coatability. A decrease of 10 to 15 mN/m in surface tension could counteract for ca. 5 to 10 degrees higher water contact angle on barrier layer.

The role of barrier layer surface roughness on coatability was also studied. A high surface roughness was expected to impact the coatability, since it can potentially pin the liquid that would otherwise dewet a surface. However, in the current study, the water contact angle seemed to play a more important role. Since coating of all top coat dispersions regardless of surface tension was possible on all the substrates having water contact angles less than 100° possible roughness effects can only be seen on substrates having contact angles higher than 100°. In Figure 5 the average coatability is plotted at different PPS roughness levels. The pure latex coatings give the smoothest surfaces while adding pigments increases roughness. As shown in Figure 3 particle edges are partly sticking out from the surfaces increasing the roughness. Talc particles are hydrophobic and the difference in hydrophobicity



between particle edges and latex filling up the voids between them is minimal, whereas kaolin particles are hydrophilic and a structure consisting of hydrophilic particle edges sticking out from a hydrophobic latex film is possible. This might explain the reason for a higher roughness slightly improving coatability although the contact angle for water is around  $110^\circ$ . The mechanism of forced wetting might also increase the effect by surface roughness. The high viscous dispersion is pressed and pulled onto the surface and the mechanism differs therefore from the dynamic wetting behavior. One should however note that, for functional printing, it is of interest to have the top layer surface in the multilayer structure as smooth as possible. Therefore, it is also favorable that the barrier layer has a low roughness, which enables coating of thin top coat layers.

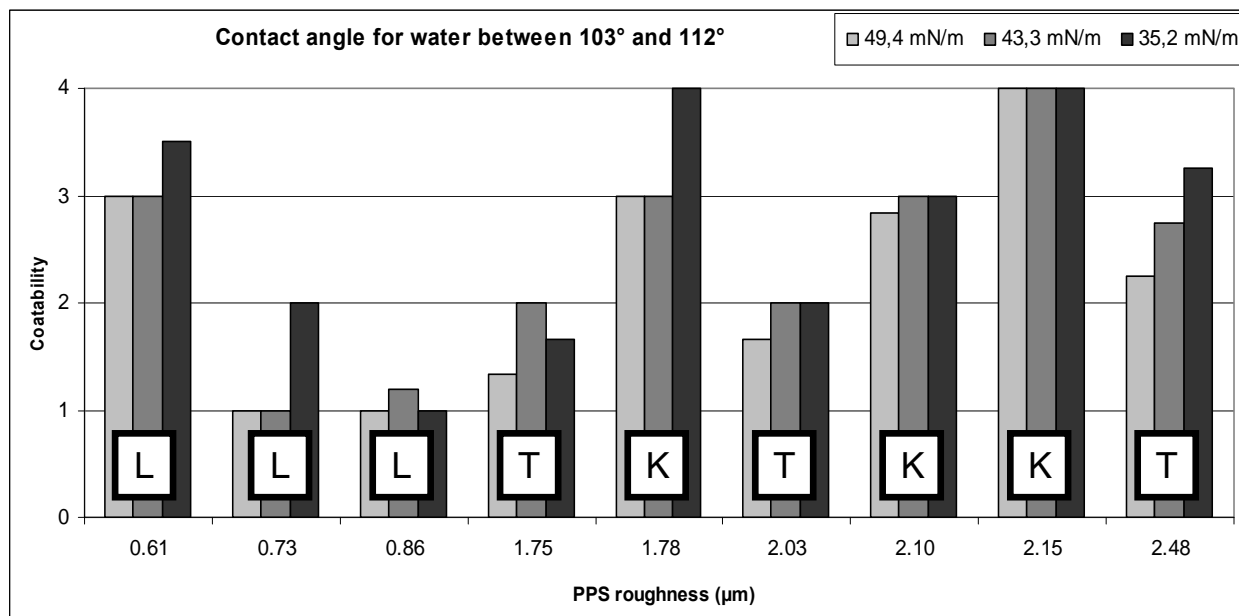


Figure 5. Coatability versus PPS roughness in the contact angle range  $103^\circ$  to  $112^\circ$  for water. The coatability is average of coatability on two to five surfaces at the same PPS roughness. L = 100 pph latex, T = Talc combined with 30 – 50 pph latex and K = Kaolin combined with 30 – 50 pph latex.

Earlier studies on flexographic printing suggest that surface energy of a substrate should be ca. 10 mN/m higher than the surface tension of an ink in order to make printing possible [17,28,38,39,40]. In this study such simple relation could not be observed. Although there is negligible nip pressure in the reverse gravure coating method, the coating process can still be considered forced wetting since the web is being pressed against the gravure roller by the web tension. This means that the coating color is forced to contact angles smaller than would be the case without external pressure. Since the coating color is a dispersion consisting of pigments in water at a relatively high viscosity of 500 mPas, the viscous resistance plays a role in wetting behavior. The dispersion does not have time to return to the contact angle set by the chemistry alone before water is removed by evaporation. The evaporation increases the viscosity rapidly and the pigments are immobilized where they have been pressed by the gravure roller. In flexography, the ink viscosity is usually in the range of 100 - 300 mPas and the viscous resistance therefore lower when compared to the 500 mPas coating color. Additional difference to the flexography is the applied ink amount, which in flexography is ca  $1\text{ g/m}^2$  compared to  $7\text{ g/m}^2$  (as dry) in the pigment coating process. The surface tension of flexographic inks is usually around 20 – 40 mN/m whereas the surface tension of the coating colors in this study varied between 35 and 50 mN/m. Taking into account the differences in density,  $1.3\text{ kg/dm}^3$  for the coating color and  $1\text{ kg/dm}^3$  for a flexographic ink, and dry solids content, 44% for the coating color and 40% for a flexographic ink, the applied volume per area is almost five times higher ( $1.22\text{ cm}^3/\text{m}^2$  vs.  $0.25\text{ cm}^3/\text{m}^2$ ) in the coating process. Furthermore, flexography is a patterned coating, meaning small defects can easily be induced when narrow lines consisting of dots are printed, compared to evenly covering large areas with uniform coating. This means that the coating process is not as sensitive to surface wetting as printing is.

## CONCLUDING REMARKS

In order to be able to coat on a dispersion coated barrier layer, the surface properties must be well understood and controlled. The most important aspect is the wetting properties, especially the water contact angle, which according to the coating results should be kept below 100°. The surface roughness measured by PPS could be seen to have only a minor effect on coatability. A higher roughness seemed to slightly improve coatability when the contact angles for water were around and above 110°. Different surface treatment methods such as plasma and corona can be used for improving the wettability. As shown in several earlier studies the wetting mechanism is a relationship between surface energy of barrier layer and the surface tension of the top coat dispersion. The surface tension can be decreased by addition of alcohol which improves the wettability to a certain extent. The high viscosity of the top coating formulation appears to improve coatability by delaying any dewetting prior to coating immobilization by drying. Since dewetting is a dynamic process, the process dynamics, e.g. delay in initiating drying and the speed of drying could potentially play a role. The process dynamics, and the influence of viscosity were, however, not investigated separately here. In earlier work by T. Schuman et al. barrier properties of dispersion coated substrates has been reported to decrease after corona treatment. This could not be seen in this study, probably because of the thick barrier layers used. The results of the current work can potentially also be helpful in understanding how barrier coated surfaces are wetted by aqueous based adhesives (glueability).

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