Tuning inkjet printability of hydroxypropylated-starch-based coatings by mineral selection

Ovaska Sami-Seppo, Mielonen Katriina, Lyytikäinen Johanna, Backfolk Kaj; Lappeenranta University of Technology, School of Energy Systems, Group of Packaging Technology, P.O. Box 20, FI-53851 Lappeenranta, Finland

Abstract

Coatings based on hydroxypropylated starch (HPS) provide extraordinary print density with dye-based inkjet inks, but their suitability for pigment-based inks can be limited due to inadequate carrier medium (water) absorption and colorant fixation. In the present work, HPS-based coatings were tailored for both a pigment-based and a dye-based ink by adding silicate minerals or acid clay (both Lewis and Brönstedt acid sites present in the acid clay). Substrate-ink interaction and colorant distribution were investigated via uncoated gloss and print quality indicators such as print density, ink bleeding tendency and delta gloss after printing with desktop printers. Three-dimensional z-stack CLSM images taken from printed samples revealed substantial differences in ink holdout and penetration characteristics between the studied materials. Pigment-filled HPS coatings showed up to 60% increase in print density compared to uncoated reference substrate and an increase of up to 35% compared to plain HPS coating with dye-based inks. In case of pigment-based ink, the increase was 17% between plain HPS coating and acid-clay-filled HPS coating. In addition, a combination of HPS and silicate mineral decreased ink bleeding substantially, but lower print gloss compared to substrate initial gloss was observed with the majority of experimental coatings. It was found that the pigment type affects substantially the unprinted gloss, which is naturally high in the case of pure HPS coating, but also the observed drastic difference in substrate permeability may explain the differences in print quality. The findings suggest that coating morphology, together with chemical interactions between starch and mineral and coated substrate and ink, has a key role in achieving high gloss and good print quality.

Introduction

Hydroxypropylated starch (HPS) has several useful features that make it an interesting raw material for coating paper or paperboard. HPS-based coatings have been shown to provide grease barrier properties [1] and to prevent the migration of both liquid and gaseous mineral oil [2]. Furthermore, HPS-based coatings have superior printability by means of print density, mottling behavior and dry rub resistance with dye-based inkjet inks, but problems with water fastness may occur [2, 3]. These features together suggest that especially food packaging industry might benefit from HPS-based coatings, since there is only a limited selection of cost-effective printable biodegradable coatings made from renewable resources that have barrier properties and prevent the migration of detrimental compounds to food.

Starch-based surface sizes have been reported to decrease paper opacity and brightness, but to increase paper gloss, print gloss, and print density with offset inks [4]. It has been suggested that the non-absorbing character of starch coatings limits their usability in inkjet printing [5]. Moreover, the film-forming tendency of starch causes a lack of absorbing pores, which are required for fast ink absorption. A large number of small pores is required for fast wetting in order to avoid problems related to lateral ink spreading such as bleeding, but this matter is not completely black-and-white, since too high absorbency leads to swelling of the coating layer, print-through problems and a loss of print density [6]. Generally the total efficiency of plain starch is considered only moderate in inkjet printing applications [7], although starch may provide good color gamut and reasonable print sharpness [8].

The physical characteristics and chemistry of other coating components such as pigments makes it possible to tailor the optical properties of a substrate, the ink-coating interaction and the achievable print quality. This suggests that the potential of starch-based coatings can be improved by adding inorganic minerals to the coating. For instance, a combination of cationic starch and silica has been reported to improve the paper surface properties and water fastness of prints, in which case the specific interaction between the anionic colorant and a cationic coating layer led to a better water fastness [9]. In addition, mixing certain minerals such as acid clay with starch leads to cross-linking and emergence of hydrogen bonds, which affect coating swelling behavior and its mechanical properties [10]. Cross-linking of starch has also been reported to increase paper gloss [11]. The excessive penetration of inkjet ink can be successfully controlled by e.g. starch/calcium silicate coatings, which also increases print density substantially [6]. HPS coatings can be filled with several minerals such as kaolin clay or synthetic silicate, which results in excellent print density and minimal bleeding in dye-based inkjet printing, but water fastness of such coatings is limited and the high viscosity of dispersed synthetic silicate limits its dosage in larger-scale coating process [3]. The limited water fastness of HPS-based coatings is probably due to solubility and to mildly anionic character of HPS that the cationic charges present on the edges of silicate particles are not capable to compensate. In addition, mixtures of calcium carbonates, clay and phyllosilicates can be used together with film forming film-forming polymers in order to achieve an ink-jet printable substrate [12].

The present study is part of a series of studies aimed at demonstrating the inkjet printability properties of substrates having an HPS-based coating. In our earlier studies, we have demonstrated the superior printability of mineral-filled HPS coatings with dye-based inkjet inks [3] and demonstrated other functional properties of such coatings [1, 2]. This part is focused on tailoring the HPS-based coatings for pigment-based inkjet inks, which was carried out by comparing the effects of three different minerals and determining substrate properties and print quality. The substrates were printed with both pigment-based and dye-based inkjet inks using desktop printers. Print quality and ink-substrate interactions were assessed by measuring print density, ink bleeding and wicking, and delta gloss. Confocal microscopy was used to clarify ink penetration characteristics on experimental coatings. We also shortly discuss the matters related to coating process scale-up from bench scale to full pilot scale.

Methodology

The coated paperboard samples were produced with a Pilot Coater (KCL Oy, Espoo, Finland) using roll applicator and rod doctoring at a machine speed of 100 m/min. The baseboard was a commercial solid bleached sulphate paperboard with a grammage of 190 g/m^2 (Trayforma^{TM} Natura; Stora Enso Oyj, Imatra, Finland). Backside of the paperboard was coated with hydroxypropylated-starch-based (HPS; Solcoat^{TM} P55; Solam GmbH, Emlichheim, Germany) coating colors having either 0 or 10 pph of dispersed synthetic silicate (S; Laponite^{TM} RDS, BYK-Chemie GmbH, Wesel, Germany), dispersed layered fluorosilicate (FS; Laponite^{TM} JS, BYK-Chemie GmbH, Wesel, Germany) or dispersed acid-leached phyllosilicate clay (AC; Fulacolor^{TM} XW, BYK-Chemie GmbH, Wesel, Germany). The proportion of pigment was limited to 10 pph due highly viscous nature of dispersed S that complicated the pilot-scale coating process and to avoid excessive pigment agglomeration [13]. The targeted coat weight was 4 g/m^2. The Brookfield viscosity of coating colors at approx. 40°C was determined at 50 and 100 rpm. In addition, dry solids content and pH were measured (Table 1). Both synthetic silicate and fluorosilicate increased the viscosity and pH, but such effect was not registered in the case of acid clay, which decreased the pH of the coating paste. Minor shear thinning was observed in case of pure HPS solution, HPS/FS mixture and HPS/AC mixture.

Table 1. Recipies and properties of coating colors.

Property	Coating			
	HPS 100 pph	**S 10 pph**	**FS 10 pph**	**AC 10 pph**
Brookfield viscosity, 50/100 rpm, mPas	57/79	275/265	90/106	60/77
pH	6.7	8.2	8.2	5.9
Solids content, %	19.4	20.3	20.0	19.5

The board samples were characterized by determining coat weight gravimetrically and measuring contact angle for water, Cobb60 test, air permeance (ISO 5636-3:2013), roughness, gloss, brightness and opacity. Apparent contact angle was determined with a Theta optical tensiometer (Biolin Scientific AB, Sweden). The drop volume was 3 µl and the value was read 0.5 s after dispensing the drop. Roughness was measured with a Parker Print-Surf instrument using a soft disc. Gloss was measured from both unprinted and printed (black, 100%) samples using a Zehntner ZLR-1050 device. Both unprinted gloss and delta gloss were reported. Opacity and brightness (D65/10°) were measured with a Lorentzen & Wettre ElRepho tester following standards SCAN-P 3:93 and SCAN-P 8:93.

Both uncoated and coated samples were printed with two desktop inkjet printers (HP OfficeJet Pro 8000 Enterprise, aqueous pigment-based inks and Memjet Lomond Evojet, aqueous dye-based inks). Print quality was evaluated by measuring print density with X-rite SpectroEye spectrophotometer in the 100% tone value areas for the black color. Wicking was measured from the raggedness of the black and red printed lines on the substrate, and bleeding was measured from the raggedness of the black and red lines printed on white with a yellow boundary using a digital pocket microscope (DPM 100, Fibro System AB, Sweden). In addition, confocal laser scanning microscope, (CLSM, Zeiss LSM 710, Carl Zeiss Ltd. Germany) images were captured from selected printed (magenta, 100%) surfaces.

Results

Substrate properties

The physical and optical properties of coated substrates were compared to uncoated base material (Table 2). Achieving accurately the targeted coat weight, 4 g/m^2, was found to be challenging at pilot coater due to low viscosity of certain coating solutions. The problem was emphasized especially with pure HPS solution and HPS/AC mixture. Both fluorosilicate and synthetic silicate pastes had a higher viscosity than pure HPS solution, which was due to the gelation tendency of such minerals that leads to a formation of three-dimensional structures when water is present [14]. Increased viscosity of HPS-pigment mixtures assisted in achieving the targeted coat weight, but it does not exclude the possibility that pigment agglomeration took place at some level. Pure HPS and pure synthetic silicate have both film forming properties, which decreased the air permeance substantially compared to base material and HPS/AC coating. The coating comprising synthetic silicate instead of fluorosilicate can be expected to have better integrity due to a larger specific surface area [15, 16]. The average particle size of acid-leached clay was visibly greater, which made the coating structure more open and could also explain the increase in roughness compared to nano-sized pigments such as the used synthetic silicate. Interestingly, none of the studied coating dispersions promoted surface smoothness, which was relatively low in the case of uncoated base substrate. However, an addition of synthetic silicate to HPS compensated the coating-induced surface roughening. Earlier Kenttä et al. [17] have reported increased surface roughness on polyvinyl alcohol-based coatings filled with certain types of silicas. In that case, the increased roughness was ascribed to cracks in the coating layer. Since no plasticizers were used in the present work, it is likely that there were cracks in the coatings that increased the surface roughness, but this should be confirmed e.g. by using scanning electron microscopy.

Plain HPS coating and HPS/S mixture both decreased the brightness of the substrate compared to uncoated base material, which was expected, since various types of starches have been reported to decrease brightness [4]. A loss of brightness, however, was not observed if the HPS solution had been filled with either fluorosilicate or acid-leached clay. As expected, the baseboard itself had initially good opacity, but a starch coating can decrease opacity due to starch penetration into the pore structure. However, the opacity remained somewhat unchanged after coating. The type of starch affects its penetration tendency because of electrostatic interaction [18] and viscosity, but in the present study, also the highly hydrophobic nature of the base board (contact angle was 114°) may have impeded the penetration of starch and affected its immobilization. However, all the coated samples had a hydrophilic character due to high starch concentration, which enables substrate wetting and thus improves printability. All the coatings were hydrophilic, and only moderate differences in water contact angles were observed between the studied coating compositions. HPS/AC coating was the most receptive for the water, which was ascribed to higher porosity and more open structure. Both spreading and absorption took place during the contact angle measurement regardless of the coating composition, although complete absorption took typically several seconds due to large droplet volume (3 µl). The Cobb test showed that all the coated boards were more absorptive than the uncoated, AKD-sized baseboard.

Table 2. Coat weight, air permeability, roughness, water contact angle (0.5 s after dispensing the drop), Cobb 60 value and optical properties of coated paperboard samples. Standard deviation is shown in parentheses.

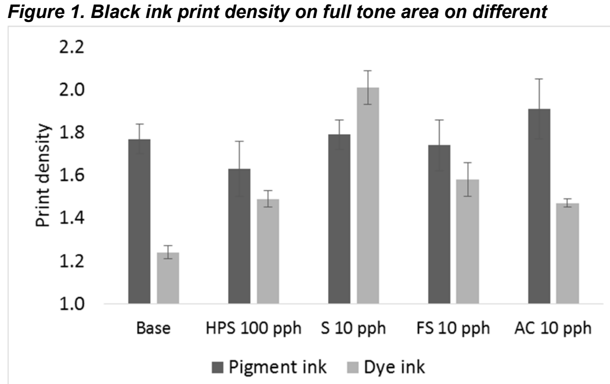
Property	Coating				
	Base	HPS 100 pph	S 10 pph	FS 10 pph	AC 10 pph
Coat weight, g/m^2	-	3.0	3.5	3.5	3.0
Air permeance, ml/min	582 (34)	52 (8)	51 (14)	102 (21)	458 (7)
Roughness, µm	5.1 (0.2)	6.0 (0.2)	5.8 (0.2)	7.5 (0.2)	7.4 (0.2)
Contact angle (water; 0.5 s), °	114 (3)	62 (7)	72 (5)	66 (5)	58 (3)
Cobb60, g/m^2	19 (1)	28 (1)	31 (0)	39 (1)	32 (0)
Brightness	84.3 (0.1)	83.4 (0.2)	83.0 (0.0)	84.2 (0.1)	84.4 (0.0)
Opacity	93.0 (0.1)	93.2 (0.2)	93.0 (0.2)	93.2 (0.2)	93.1 (0.1)
Unprinted gloss, %	12.5 (0.5)	20.4 (1.9)	19.5 (1.2)	9.8 (0.9)	8.1 (0.2)

Plain HPS coating and a mixture of HPS and synthetic silicate made the paperboard glossier. The ability of different types of starches to increase both paper gloss and delta gloss is widely known [4]. Based on the results, it seems that HPS increases the unprinted gloss only moderately compared e.g. to a more common cationic starch [4], although the difference was substantial compared to uncoated baseboard. A matt surface can be achieved by replacing synthetic silicate with acid-leached clay or fluorosilicate in the recipe, which is surprising, since these minerals can act as cross-linkers [10, 13], and cross-linking of starch should increase paper gloss [11]. The observed differences in gloss values were thus ascribed to different tendencies of pigments to orient and pack in the coating layer [17], induced by the different particle sizes of synthetic silicate and acid-leached clay.

Print quality

Figure 1 shows the print densities of studied paperboard samples. All the coatings increased the density of black ink if printing was carried out using dye-based ink. This result corresponds well with our earlier work in which the paperboard was coated with a bench-scale coater in sheet mode [2], although in the present study the coatings comprised only 10 pph of pigment. Synthetic silicate provided the highest print density in this connection, which suggests that the ink remained inside the coating layer. However, the other studied pigments performed poorer with the dye-based ink and they provided no additional value compared to plain HPS coating. Having in mind that the HPS/S coating was the least permeable, the results correspond relatively well with the work of Package Kenttä et al. [17], whose study indicated that the print density is at least partly affected by the coating layer porosity in dye-based inkjet printing. Pigment-based ink, however, behaved differently compared to dye-based ink and the earlier findings. A minor decrease in print density was observed if the substrate had an HPS coating. No major differences in print density was found between S and FS.

Figure 1. Black ink print density on full tone area on different



substrates.

Moderate differences in the evenness of ink layers were observed between the different coatings. The coatings comprising silicates had minor mottling problems with pigment-based inks (standard deviation for print density was 0.07-0.12), whereas the presence of acid clay resulted in less variation (standard deviation for optical density was only 0.03). Based on the results, the best coating composition for pigment-based inkjet printing was HPS/AC, which increased the print density approx. 10%, compared to the base material. Since a dense coating has been proposed to improve the print density with pigment-based inks and the HPS/AC coating was very permeable, there must be some other mechanism behind the improved print density. The contact angle measurement revealed that the baseboard was highly hydrophobic (Table 2), which might partly explain why the print density was so high with the pigment ink on baseboard and porous HPS/AC coating. The taken CLSM image (Fig. 4g) supports this interpretation, since the pigment ink had formed a clear layer inside the coating layer, close to the coating-paperboard interface.

Severe bleeding problems were found with pigment-based ink on uncoated, HPS-coated and HPS/FS-coated substrates (Figure 2). On uncoated sample, bleeding of pigment ink was substantial, but dye ink performed better. Uneven spreading and ink holdout problems occurring on the surface was probably due to high hydrophobicity (for water, CA was 114° and Cobb60 19 g/m^2) that led to inadequate and slow absorption of the ink solvent. Bleeding occurred on HPS-coated sample was probably induced by impermeable coating and ink spreading, suggesting limited immobilization of the colorant. Only minor bleeding was observed on HPS/S and HPS/AC coatings. In case of HPS/AC, the low bleeding tendency was probably caused by the more hydrophilic character compared to uncoated board and open and porous structure of the coating layer that resulted in fast wetting. Also the AC-induced cross-linking of starch may have had influence on the bleeding. Although the air permeance of sample having an HPS/S coating was somewhat similar to plain HPS coating, the small size of Laponite particles (lateral diameter can be as small as 25-35 nm in pure water) may have promoted the formation of a microporous structure that makes the penetration of ink carrier faster and prevents excessive ink spreading [6, 17]. With dye-based ink, less bleeding occurred compared to uncoated reference sample regardless of the coating composition. It thus seems that the improvement in print quality was induced at least partly by the surface hydrophilicity that made it possible for the ink to penetrate inside the coated sample fast and more evenly.

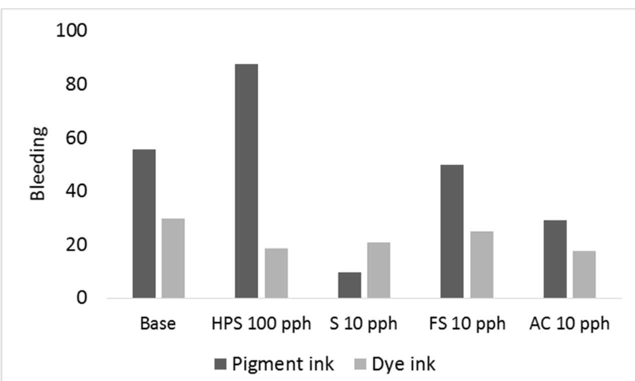


Figure 2. *Black in bleeding tendency on different substrates.*

Applying a thin hydrophilic coating on hydrophobic paperboard increased pigment ink wicking tendency regardless of the coating composition (Figure 3). The problem was emphasized on pure HPS coating that was not particularly permeable. If the coating comprised S, the increase in wicking was considerably smaller. This might be due to positive charges locating in the edges of Laponite particles that assisted in locking the ink pigments. No drastic changes in wicking was found with the dye ink, although HPS/S coating provided slightly better print quality in terms of wicking. This was also ascribed to cationic-anionic interaction between the ink and the positive charges on the edges of Laponite particles and faster penetration induced by microporous structure [14, 17].

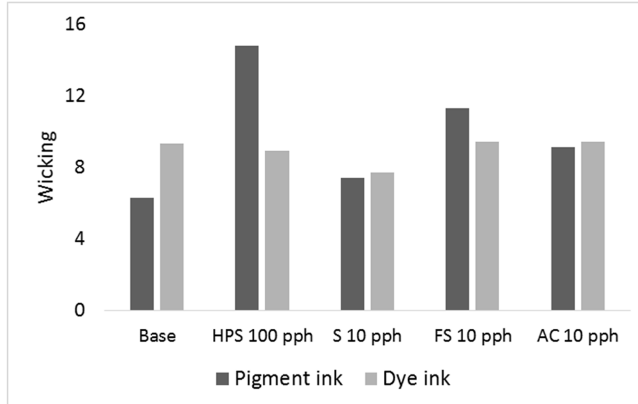


Figure 3. *Black ink wicking tendency on different substrates.*

Ink penetration and printed gloss

Three-dimensional z-stack CLSM images taken from printed paperboard samples revealed substantial differences in ink holdout and penetration characteristics between studied materials. In case of uncoated samples (Fig. 4a and 4b), wide vertical spreading was observed with both pigment and dye inks, which explains the lower print density compared to the best coated samples. Dye ink formed a thin layer inside the coating comprising 10 pph of S (Fig. 4d), indicating that a nanopigment in the coating on a hydrophobic substrate can be used for adjusting ink penetration depth and ink fixation. This leads to a higher print density when taking into account the transparency a starch coating, but the behavior was not as clear with the pigment ink (Fig. 4c), which suggests that the cationic edges of S particles assist in fixing the colorants. A formation of a thin pigment ink film was observed inside the HPS/AC coating (Fig. 4e), which also had the highest print density among the samples printed with the pigment ink. Having in mind the high air permeance, it seems that the coating layer was more porous, and the ink penetration probably stopped at coating-board interface due to baseboard hydrophobicity (Fig. 4g). As it can be seen from Fig. 1, the print density on an HPS/AC coating was close to uncoated base material with dye ink. When comparing Figs. 4b and 4f, it can be observed that extensive ink penetration occurred in both cases and no tightly packed ink layer was formed inside the coating. Based on this evaluation, an HPS/S coating suits for universal printing purposes, since it functions quite similarly regardless of the type of ink. Coatings comprising acid clay instead required a pigment-based ink in order to obtain high print quality, which was ascribed to excessive penetration of dye-based ink due to high porosity. Closing the surface with pigmented starch coating prevents excessive ink penetration [6], but in the present study the improvement in density was probably also affected by the hydrophobic baseboard that assisted the formation of a thin ink layer inside the coating layer.

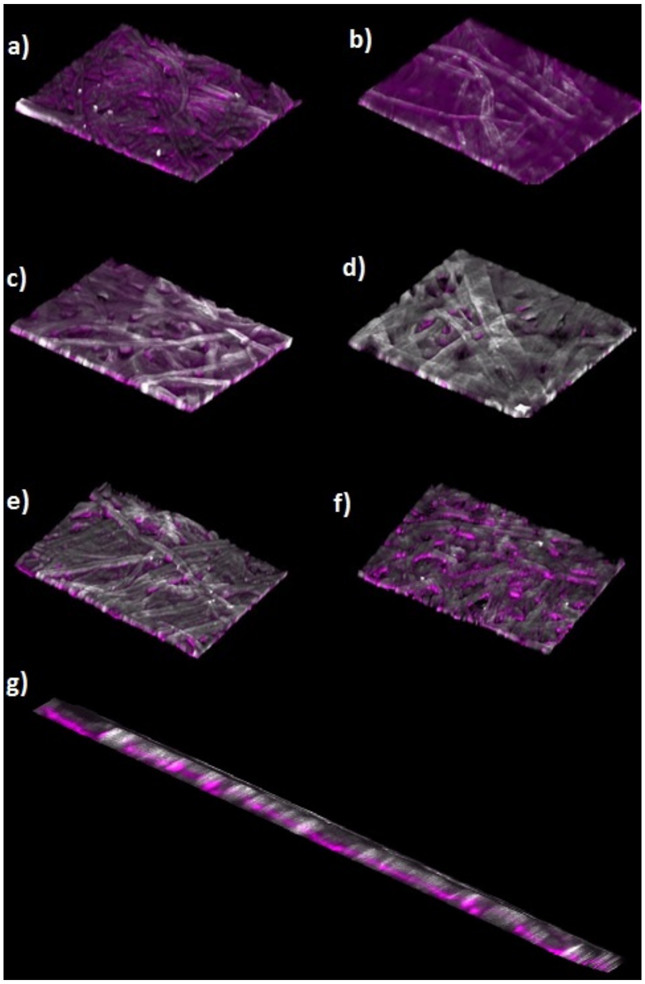


Figure 4. Confocal laser scanning micrographs of selected magenta-printed (100%) samples. The thickness of examined area is approx. 20-25 µm. (a. Uncoated board with pigment ink, b. uncoated board with dye ink, c. HPS/S coated board with pigment ink, d. HPS/S coated board with dye ink, e. HPS/AC coated board with pigment ink, f. HPS/AC coated board with dye ink, and g. tilted z-stack imaged area showing a thin ink layer inside HPS/AC coating)

The microstructure of a starch film becomes more mobile and open at moist conditions [19]. In addition, potato-based starches are highly hygroscopic that may cause swelling of the coating layer. Excessive swelling has been suggested to increase surface roughness that leads to a lower print gloss and further even negative delta gloss [20]. These features of starch-based coatings led to a hypothesis that it could be difficult to maintain the high gloss of HPS and HPS/S substrates after printing with water-based inks. To investigate this hypothesis, delta gloss was determined from each sample (Fig. 5). It was found that especially the use of pigment-based inks resulted in negative delta gloss. The problem was emphasized with pure starch coating, but an addition of pigment made the decrease smaller. Both S and AC performed relatively well, even though unprinted gloss was very different between these two coatings (Table 2). The decrease in delta gloss was substantially smaller with dye-based inks. This was seen most clearly on pure HPS coating, which indicates that its more impermeable nature assist in retaining the ink close to the surface and the lack of ink pigments makes the surface roughening smaller. Slightly positive delta gloss was observed on HPS/FS coating. However, by taking into account the standard deviations (unprinted gloss 0.2, printed gloss 0.3; not shown in Fig. 5), this result can be considered only indicative. It can be concluded that negative delta gloss values were not only due to morphological changes of the coating layer, and it is recommended to use dye-based inks if the substrate gloss is desired to be maintained.

	Base	HPS 100 pph	S 10 pph	FS 10 pph	AC 10 pph
Pigment ink	-8.9	-10.7	-2.2	-4.3	-3.3
Dye ink	-4.8	-0.3	-1.6	0.5	-2.6

Figure 5. Black ink delta gloss on different substrates (100% tone area).

Summary

The effects of various pigments on the inkjet print quality of HPS-based coatings were determined for both dye-based and pigment-based inks. The coatings were made using a pilot-scale coater that made it also possible to compare the material performance with our earlier findings [2, 3] after process scale-up. Optimal coating formulation for pigment-based inkjet inks was also searched. Compared to our earlier work, the print density value with dye-based ink for HPS/S coating was smaller, which was ascribed to a smaller pigment proportion (10 pph) due to high viscosity of S, but the density was still at adequate level for various printing applications. All the used pigments can cross-link starch [10, 13], which affects the coating solution and swelling behavior, and its absorption properties. In addition, keeping the pigment proportion at such level is required in order to avoid agglomeration [13]. The coating recipies were developed further for pigment-based inks and it was found that a combination of HPS and AC provides almost as high print density as HPS/S coating for dye-based inks. By taking into account the economic aspects, HPS/AC coatings are more competitive compared to HPS/S coatings, but this will lead to a compromise in delta gloss and ink bleeding. Based on this study, following guidance for coating selection is proposed in order to achieve the desired substrate properties:

Table 3. Optimal coating compositions for pigment-based and dye-based inkjet inks from the viewpoints of substrate properties and print quality.

Targeted property	Pigment ink	Dye ink
High opacity and brightness	Depends mostly on base material	
High unprinted gloss	HPS HPS/S	
Maintaining delta gloss	HPS/S	HPS/FS HPS
High print density	HPS/AC	HPS/S
Low bleeding	HPS/S	HPS HPS/AC HPS/S
Low wicking	HPS/S HPS/AC	HPS/S

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Author Biography

Sami-Seppo Ovaska received his doctoral degree in paper technology from Lappeenranta University of Technology (LUT) in 2016. He has worked particularly with dispersion coating, grease resistance and paperboard converting. During his studies, Dr. Ovaska was a member of the PaPSaT graduate school. He works now as a post-doctoral researcher at LUT in the Group of Packaging Technology whose scientific focus area is focused on renewable barrier and packaging materials.