Creating Functionality on Paper Using Novel Roll to Roll Surface Treatment Equipment: Case Studies

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Abstract

Two different case study applications were produced, which exploit a novel pilot scale surface treatment line in order to create intelligence on fibre based substrates. The first application was based on replacement of traditional pigment in coating colors by TPS (thermoplastic starch) to create smoother, better conducting surface for printed electronics. The second case study was based on addition of enzymes to create bioactive functionalities on the paper surface.

Introduction

The forest industry is going through many structural changes. Companies are looking for innovations to reduce the dependency on oil-based raw materials, enhance the usage of raw materials and achieve added value for their products.

Development of innovative products starts from the raw materials. The paper and board industry relies heavily on oil-based materials in certain applications. The replacement of the oil-based material with bio-based products might give a competitive advantage due to the need for more sustainable and greener image.

This paper presents two case studies in which a novel pilot-scale surface treatment equipment has been used to create functional fiber based materials. These functional fiber based products are sometimes referred to as printed intelligence.

Creating functionality and printed intelligence on paper by printing and coating is already possible and some simple demonstrative examples such as new detection methods, integration of electronics and optics onto large area substrates have been created already. These applications are aimed to replace more expensive products or are more sustainable.

What is printed intelligence? Printed intelligence means components and systems which extend the functions of printed matter beyond traditional visually interpreted text and graphics manufactured by continuously running i.e printing, roll-to-roll (R2R) methods, to enable cost efficient integration/embedding of simple intelligence everywhere.

First case study presents the development work and results acquired to create smoother base for printed electronics. Most obvious application being a simple RLC-circuit that requires surface smoothness less than 0.1 µm. Three different surface

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coating materials previously known to create as smooth coatings as possible were chosen in order to achieve this. Tradionally used coating pigment kaolin was an obvious pigment choice. Flexo-laquer used for protection of printed surface and to enhance appearance of magazines and quality packages was the second chosen coating. Third chosen surface coating was based on thermoplastic starch pigment developed, patented and successfully used as a coating color pigment. No pretreatments or multiple different coatings for the same trial point were used in order to create a smooth surface. After coating, the paper webs were calendered in a single, soft nip laboratory calendar. The calendar conditions were specified for each coating using high nip pressure and when possible high surface temperature of calendar rolls. Flexo-laquer could not tolerate high calendar roll surface temperatures.

The second case study presents development work and results to create bioactive functionalities on a fiber based web. Commercially available enzymes and substrate solutions were chosen for the case. Enzyme solution was added on to fiber web using spray equipment. The activity of enzymes on paper was determined by applying the enzyme's substrate solution on an enzyme coated web to create a color reaction on a pretreated web. The benefit of this technique is that small pieces from a large amount of fiber web can be cut out and each small piece can be used to create a biosensor.

Pilot equipment used for creation of these case studies is called SUTCO shortened from surface treatment concept. The name is mainly used internally within the company the authors are working for. It is manufactured by Coatema Coating Machinery GmbH. The line consists of 16 individual components that can be connected as desired and can be set up according to research needs. The main benefits of the surface treatment concept are low cost, low coating material demand and versatility of used substrates including plastics, paper and carton board.

Pilot coating equipment

The surface treatment concept can be used for coating and surface treatment of fiber based webs and plastics. It consists of separate one meter wide operational units built from aluminium frames. Each frame is on four wheels and all operational units are placed on rails to eliminate the need for levelling of units after changing the order of the units. Rails also eliminate the need for guidance and other roll alignment after rearranging the units. The units are attached together by a system called Click & Coat developed by manufacturer of equipment.

The concept includes several coating methods, pre-treatments and curing options. Coating methods include flexo-type roll coating, soft bar coating, kiss type coating and spray coating. The first three methods can be followed by spray coating to make wet on wet coatings. The sealable coating unit followed by exhaust air removal during drying makes it possible to test also coatings containing harmful volatile substances. Surface treatments can be cured using cold or hot air drying, IR-drying or UV-curing. Basic layout including all units of the surface treatment concept is shown below.



Picture 1. Basic layout of the surface treatment concept with configuration of 13 units. Unwinding is placed on the left hand side.



Picture 2. Drying section and rewinding.



Picture 3. Unwinding

During both case studies the surface treatment concept was developed in a way that it could be used for similar type cases in the future. The only coating method in the beginning of the project was a flexo-type roll coating. The surface treatment equipment is normally run at speeds below 20 m/min for easier operation.

Experimental

First case study: smoother surface for printed electronics

Materials

A precoated wood-free fine paper by UPM was used as base material before surface treatments. Toray Lumimirror polyester film was used as reference material.

Flexo-laquer used in trials was a Suncoat primer H9205 followed by top coat coated with Suncoat H6244 delivered by Sun Chemicals.

AP thermoplastic starch is a coating color pigment originally developed by VTT Technical Research Centre of Finland in co-operation with University of Helsinki and University of Joensuu. The pigment is a starch based thermoplastic, non-mineral pigment aimed to replace mineral pigments as fillers in base paper and as a pigment in coating colors. The starch pigment can be made for example from chemically cleaved potato starch. The non-water soluble starch derivatives are dissolved in organic solvent mixture. When non-solvent (e.g. water) is added to a mixture to dilute it a milky like particle dispersion is spontaneously formed. Particle size distribution can be varied by the dilution process, dilution speed and concentration of starch polymer solution. Organic solvent is removed after dilution by evaporation under controlled conditions depending on the solvents used.

The starch pigment can be made as a non-dried virgin quality pigment or as spray-dried pigment after precipitation. For practical storage and transportation reasons it is more feasible to use dried quality starch. Dispersion of the dried starch pigment requires slightly more attention than operation with traditional coating pigments. The dried pigment is light and therefore dusts easily. Mixing during disintegration should be done with slower than average rotation speed while the slurry is prone to foaming if too high mixing speed is used. The dried starch pigment was dispersed to 30% solids content in the trials.

Coating color formulation for thermoplastic starch pigment is shown in table I.

Table I.

Thermoplastic starch pigment	100
Latex	12
Thickener	0.8
Dispersing agent	0.1
pН	8
Dry solids content, %	30

Coating color formulation for kaolin is shown in table II.

Table II.

Kaolin	100
Latex	12
Thickener	0.8
Dispersing agent	0.1
рН	8
Dry solids content, %	45

In these trials no lubricant or optical brightening agent were added in the coating color in order to generate as simple as possible coating colors and also because optical properties were not the scope of the case studies.

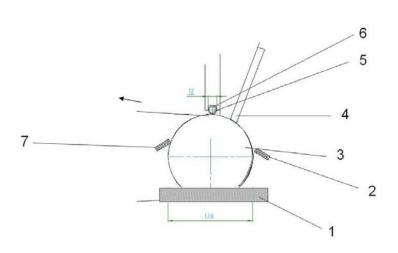
Pilot coating equipment modification

The soft bar coating was developed in order to achieve high coating amount in single coating pass and also to eliminate the problem of orange peel pattern occurring while coating with flexo-type roll coating. The coating color amount that can be applied on web using flexo-type roll coating becomes smaller with every pass and it may require excessive number of passes to reach coating amount desired.

During the case study only available coating method was flexo-type roll coating. The soft bar coating was created from existing equipment available in house due to lack of funding to purchase new equipment.

The soft bar coating is based on a grooved rod backed up with a flexible element. The grooved rod is loaded against the soft backing roll and volume of the grooves determines the coating amount on the web. The rod was placed on existing element on the coating equipment.

The coating module had a top feeding trough on top of the coating roller. The outer side trough wall had a micrometer adjustable plate which was created out of teflon and a groove was grinded in the bottom side of the wall to fit the grooved rod and to place the smoothening element under the rod. While coating, the rod was loaded against a running web before adding coating color to the coating color reservoir so that no visible gap was observed between the rod and paper and the paper was able to run without sticking between the grooved rod and the backing roll. The rod pressure had a minor effect for coat weight so no load measuring element was used during trials. The principle is shown in pictures 1-3.







- 1. Coating reservoir/tray
- 2. Flexible edge doctors; placed only on web edges
- 3. Counter roll
- 4. Fixed pre-doctor
- 5. Coating bar / loaded soft bar
- 6. Softening/cushioning element
- 7. Doctor cleaning of excess coating from counter roll

Pictures 1-3. Diagram showing different elements of the soft bar coating equipment (1). End of the grooved section of the rod. The black colored softening element is seen behind the bar (2). Grooved bar placed in the assembly groove (3).

The coating color was applied on to a web by guiding it through the coating reservoir backed up with a roll to eliminate the coating color transfer to back side of the web. After the coating color reservoir, a pre-doctor was used to take away the most of the excess coating color to prevent excessive overflow on the edges on the grooved bar and flowing of coating color on the back side of the web.

Modifications were made also for drying section. Due to the modular structure of surface treatment equipment tension for dried web it was possible to create only by bowing the drying section. The tension of the web cannot be created by turning the web after coating. Coated paper webs tend to curl from the edges when the edges are wet. It was observed that if the coated web passes the drying section with uncurled edges it will not curl during the rest of the machine. A simple solution was created to prevent edge curling. 100 mm long round pipes were placed over the leading bars on the drying section. The gap between the leading bars and pipes was adjusted to approximately 3 mm. The paper can run freely under the pipes even when the wet edges of the web will not curl. The solution is shown in pictures below.





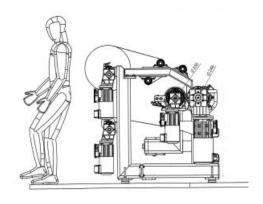


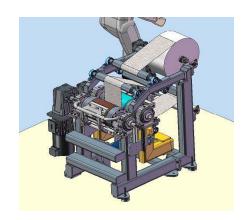
Pictures 4-6. Drying section bowed with modifications showing the solution to prevent edge curling during drying.

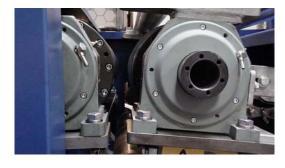
While using flexo-type roll coating there are usually two soft knives placed on the edges of the rubber covered backing roll. The purpose of these knives is to keep the edges of paper being coated drier and prevent curling issues during the drying stage. The edge knives can be made out of polyurethane for example. There is normally no circulation of coating color while the surface treatment equipment is operated. Coating color based on thermoplastic starch has a tendency to mix with air if no circulation of the coating color is employed in the coating color reservoir. After a while the thermoplastic starch started to accumulate on the back of the edge knives and eventually begins to bleed from the inner edges on to the running web onto the coated paper. If these knives are not used the edges of coated web will curl during drying on the horizontal drying section unless the round pipes are placed over the leading bars on the drying section. Thermoplastic starch containing coating color was therefore coated without the edge knives and the drying section pipe-solution presented earlier was developed to solve this issue.

Calendering

After coating, the paper web from each of the three individual trial points was calendered using hard-soft nip pilot-calender. The calender uses approximately 300 mm web width. The calendering occurs between polymer roll and metal roll (soft-hard nip) up to 350 kN/m of line load. The hard roll is heatable and surface temperatures up to 250 $^{\circ}$ C can be used and the web surface temperature measured from the running web. Web speeds range up to 90 m/min and 3 passes were used in this study. The calendar is shown below.









Pictures 7-10. Pilot-scale calendar used in the trials.

It was observed that after three passes the smoothness of the coated papers did not change significantly. The calendaring parameters are shown in table III.

Table III.

Coating based on	Thermoplastic starch pigment	Kaolin	Flexo-laquer
Nip pressure, kN/m	150	150	150
Roll surface temperature, °C	140-150	150	25
Number of passes	3	3	3

The flexo-laquer coating could not tolerate high temperatures so it was calendered using room temperature calendering rolls.

The reference point for smoothness measurements was chosen to be a product called Lumiflex. It had been previously used as printing substrate while printing a conductive ink in previously tested applications of printed intelligence. LumiFlex is a semi-gloss, one-side coated paper designed for heavier basis weight flexible packaging applications and had previously been shown to be suitable for this application.

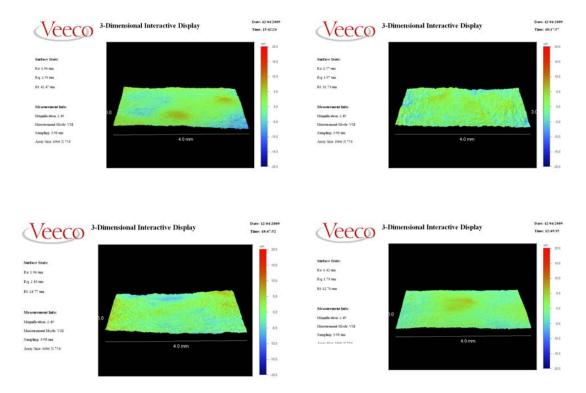
Thickness, Bentsen roughness and smoothness values were measured using Veeco Wyko NT9100 optical profilometer and are shown in table IV.

Table IV.

	Thickness, µm	Bendtsen roughness, ml/min	Roughness $R_{a,}\mu_{m}$	Roughness $R_{q,}\mu m$
Thermoplastic starch	64 ± 2	0	1,96	2,5
Kaolin	60 ± 2	0	1,42	1,78
Flexo-laquer	70 ± 1	10 ± 1	1,57	1,97
Lumiflex Ref	95 ± 2	13 ± 1	1,96	2,43

Papers with enhanced smoothness were achieved using the coating line compared to Lumiflex reference point. Paper coated with coating color based on kaolin produced the lowest surface smoothness.

The surface images of the different coatings are shown below.

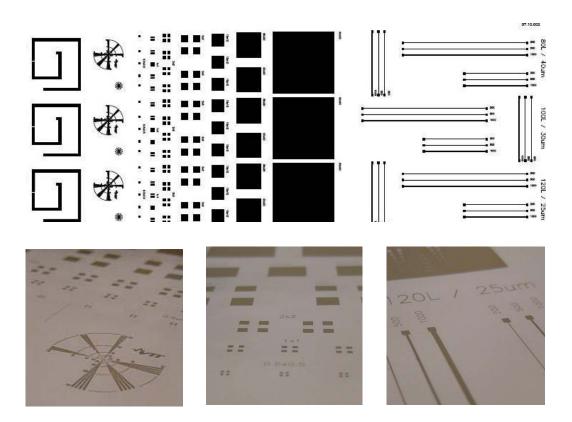


Pictures 11-14. Surface images of thermoplastic starch (top left), flexo-laquer (top right), Lumiflex (bottom left) and kaolin (bottom right).

Comparable measurement areas of the samples were selected for this Veeco analysis. It was observed that chosen measurement area had an influence on the measured roughness. Making comparisons between different measurement methods especially based on different areas can be therefore misleading.

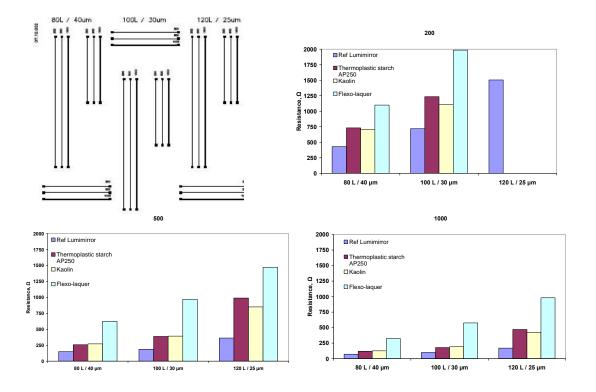
Printing with conductive ink

After coating, the webs were printed. The printing was done using a Pico gravure printing machine using conductive ink and a predetermined test pattern. Test pattern consisted of three similar pattern having different cell volumes. The printed test pattern and details from printed samples are shown below.



Pictures 15-18. The printed test pattern and details from printed samples.

For all of the coatings after calendering that were used it was possible to print conductive patterns. Uncalendered samples were not printed. The conductivity of the pattern is related to smoothness of the printed surface. Conductivity was measured as resistance, using a common multimeter, from different line widths. The smoother the surface the lower the resistance of the conductive pattern. As a reference point a plastic called Lumimirror was used which proven a valid base substrate while printing electronics. The conductivities of different trial points and print patterns showing are presented below.



Figures 1-4. The conductivities of different trial points and print pattern showing to which line the numbers in figures refer

The kaolin coated sample has the lowest electrical resistance of all fiber based trial points. Although it is still twice as large as that compared to the printed plastic reference point. The resistances for thermoplastic starch coated samples were slightly higher compared to kaolin coated samples. Flexo-laquer coated samples had clearly the highest resistances. The smoothness of these samples was lowest. After calendering some minor small scale cracking was observed in flexo-laquer coated samples which probably contributed to increased resistance. To minimize resistance as smooth as possible surface is desired.

Obviously also interprepted from the results is the fact that when ink amount on the coated surface is higher, the electrical resistance is lower.

Second case study: enzyme-based bioactive functionalities on paper web

Materials

Enzyme was sprayed using surface treatment concept on to the precoated base paper. The base paper was first coated with kaolin based coating color.

Enzyme and substrate base materials to make a solution were purchased from known suppliers. In this context substrate means the solution that is added on enzyme coated surface. The enzyme was prepared as an aquaous solution to enable it to be sprayed.

Modification of pilot coating equipment

Enzyme was added using the spray application. The spray-mist was applied using AVAC A4 high shear rate viscometer to provide the necessary pressure for the enzyme-solution to be added on to a moving web. A single spray-nozzle was placed in a box equipped with moderate fumes removal to prevent misting. The spray pattern of the nozzle was enough to cover the width of 500 mm when placed at an appropriate distance above the moving web. Enzyme solution was filtered through a smaller sized screen than the spray-nozzle opening size prior to spraying to prevent blockage of the nozzle.

During drying of the enzyme solution, moderate temperatures were used on the drying section to prevent the inactivation of enzyme during drying.

Results from enzyme case

A color reaction caused by application of substrate solution on enzyme coated web is shown in pictures below.



Picture 19. Immediately after application of substrate solution on enzyme coated surface



Picture 20. 5 seconds after application of substrate solution on enzyme coated surface



Picture 21. 10 seconds after application of substrate solution on enzyme coated surface

Application of substrate on enzyme coated web created a color reaction on pigment coated web successfully, which proves the enzyme stayed active during the process of spraying and drying. Based on these trials, the application technique developed during this case study has later been used in other similar application for slightly modified purposes.

Discussion

In the first case study a new coating method was developed and implemented successfully. Coating trials made using it followed by calendaring produced even enough surface to print conductive patterns. The surface roughness was not as low as the surface roughness of plastic. Trials attempting to create smoother base for printed electronics will continue and various variables can be trialled.

The second case study proved a simple concept of relative cheap solution to create bioactive surfaces. By combining knowledge from biotechnology a simple biosensor was successfully implemented on pilot surface treatment environment.

Surface treatment concept was successfully used to create demonstrative products of printed intelligence in the applications described in this paper. The modifications necessary were implemented and used in planned purposes. The versatility of the surface treatment line is seen well in both case studies. The surface treatment concept line opens new possibilities for new products and processes for forest industry and converters.

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