

12TH TAPPI
EUROPEAN PLACE CONFERENCE



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Aqueous polyolefin dispersion – for low energy polyolefin melt application

Presented by:

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DOW Europe GmbH, Switzerland**

Abstract:

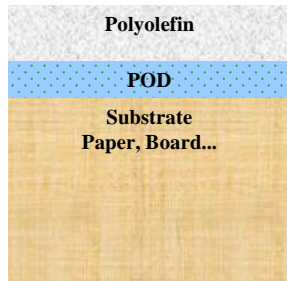
Extrusion coatings onto paper and board are today primarily based on polyethylene. One main purpose to apply polyethylene coatings is to manage paper and board properties with improved water vapor barrier combined with other functionalities.

Adhesion of non-polar polyethylene extrusion coatings can be an issue when coated on polar paper or paperboard. In addition to the surface chemistry, surface roughness and porosity of the paper has a key role in anchoring polyethylene coatings on paper or paperboard surface. Adhesion of polyethylene to paper can be decreased by sizing agents, surfactants, silicones or other chemicals and additives present in papermaking.

This work introduces novel aqueous polyolefin dispersion coating as an adhesion primer for paper and paperboard. These aqueous high solids dispersion consist of high molecular weight polyolefins with functional groups allowing adhesion on both polar and non-polar surfaces. The coatings can be applied with standard coating or printing techniques designed for aqueous coatings.

Melt application of a range of polyolefin's were studied considering processing temperatures, polyolefin type and substrate pre-treatment. Various polyolefin's demonstrate significant improvement in adhesion to polyolefin dispersion primed board.

What we describe



We Demonstrate

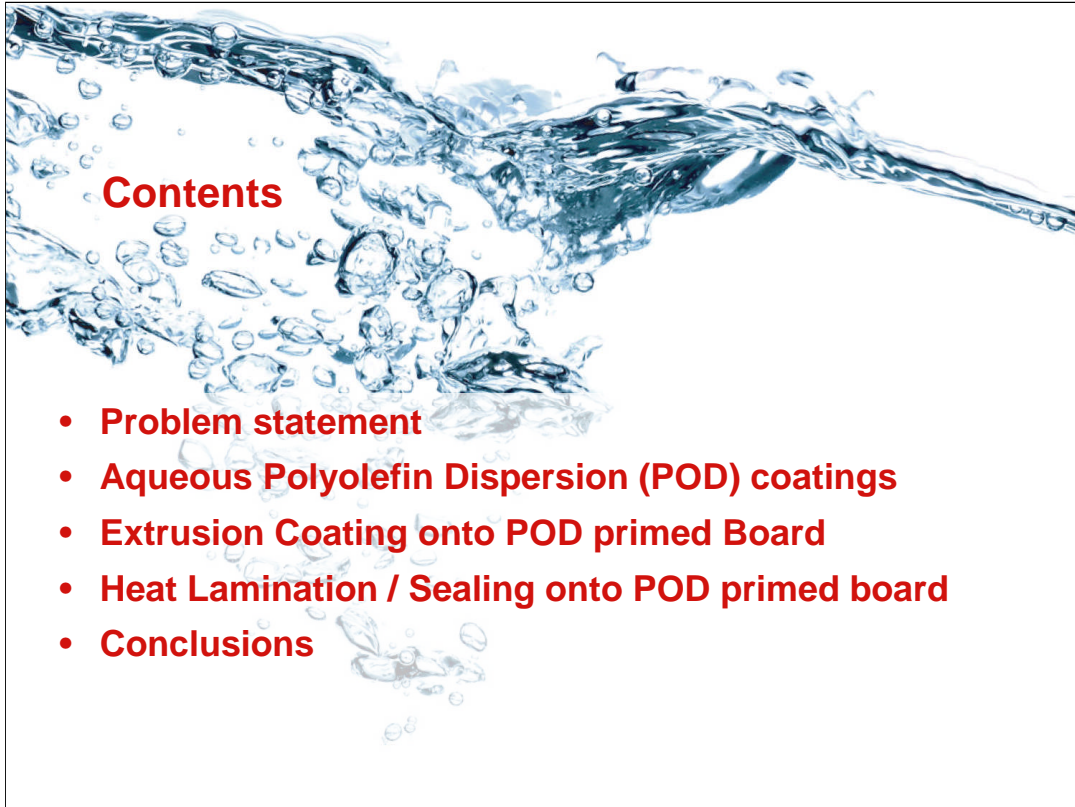
- Polyolefin dispersion (POD) application
- Extrusion Coating
- Heat Lamination / Heat Sealing

Novel Adhesion Solutions for:

- Paper and board manufacturer
- Extrusion coating
- Heat lamination

This presentation is divided in two parts. The first part discusses the polyolefin dispersion (POD) application on paperboard. The second part will discuss extrusion coating and heat lamination / sealing on POD primed paperboard.

The results will explain new adhesion solutions for paperboard manufacturer, extrusion coater and for heat laminator.



The presentation will discuss first the issues related to polyolefin adhesion to paper products.

POD dispersions and aqueous coating technique will be presented with properties measured for the primed paperboard.

The second part will focus in further converting operations and new coating and heat lamination / sealing options with POD primed paperboard

Problem Statement:

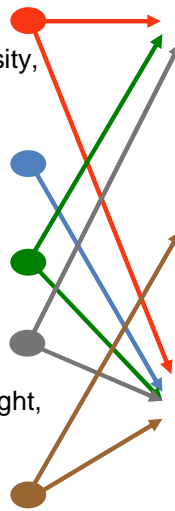
Adhesion of polyolefin to paper

Adhesion Tool Box

- **Paper surface properties**
 - fiber mix, roughness, porosity, strength, chemicals, treatments
- **Surface modifications**
 - corona, flame, ozone or plasma treatment
- **Polymer**
 - viscosity, type
- **Extrusion conditions**
 - melt temperature, coat weight, line speed, air gap
 - coextrusion
- **Primers**

Adhesion mechanisms

- A. Mechanical interlocking**
- B. Molecular inter-diffusion**
- C. Chemical interactions / attractions**



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ADHESION MECHANISMS

Interfacial adhesion and inter-layer cohesion determines the mechanical integrity of a multilayer structure. We focus only on adhesion as cohesion is predetermined by the polymer or paper and board properties. To simplify the adhesion mechanisms there are basically three contributors to adhesion – mechanical interlocking, molecular inter-diffusion and chemical interactions.

ADHESION TOOL BOX

Perhaps for the extrusion coater the paper is the biggest challenge as adhesion depends not only on roughness and porosity but also on chemical treatments or coatings that may be negative for adhesion – and all these are given with the paper and the same grade may have variations.

Converter has more freedom to increase the adhesion in the next steps (surface modification, polymer properties and extrusion coating settings and primers). All these steps are connected to either mechanical interlocking or chemical interactions.

In our solution with POD primer we can address the chemical interactions and also the molecular inter-diffusion area due to the compatibility / solubility of the systems. Our dispersion process enables to tailor made the POD for given extrusion grade polyolefin.

How are PODs different from other dispersions?

Enabled by mechanical dispersion technology

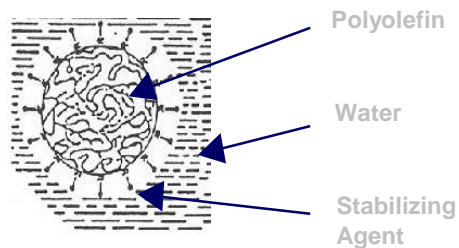
- Dispersion of polymers which are not self-dispersing
- High molecular weight polymers
- Thermoplastic and elastomer formulations
- Low viscosity
- High solids
- Solvent-free solution



The PODs are different from normal dispersions in several ways. We can manufacture dispersions of high molecular weight polymers that are not self dispersing. We can produce thermoplastic and elastomeric formulations. The utilized dispersion process allow us to deliver low viscosity and high solids dispersions. Solvents are not used in the process and thus the offered dispersions are solvent-free.

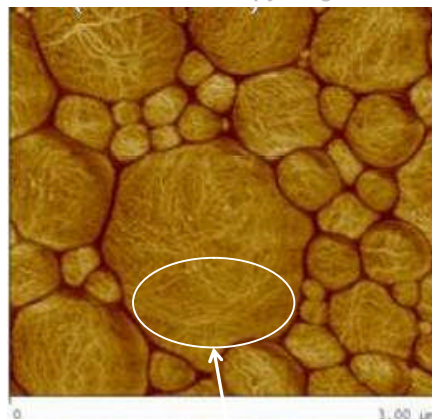
An Inside Look at the Chemistry

Polyolefin Dispersion Characteristics



- Avg. particle size: ~ 1 μm
- Solids content: 40 - 55%
- pH: 8.0 - 10.5
- Viscosity (Brookfield @ 25°C) < 500 cps

Atomic force microscopy image of POD

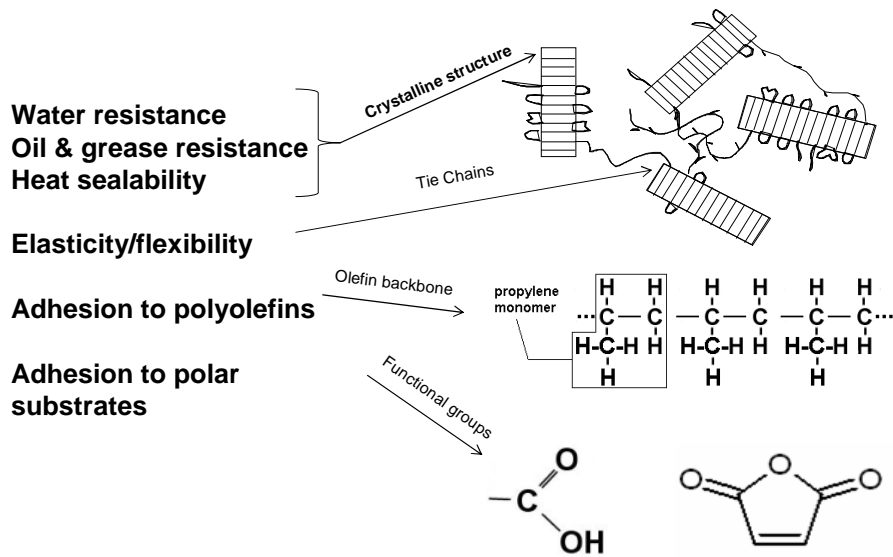


Semi-crystalline polymers

The polyolefin dispersions consist of three basic building blocks; polyolefin, stabilizing agent and water. The particle size is around 1 μm and solids content is typically 40-55%.

An Inside Look at the Chemistry

Properties of Polyolefin Dispersions



The polyolefin dispersion consist of polyolefins that are semi-crystalline. The key for adhesion is the olefin backbone giving adhesion to polyolefins and functional groups giving adhesion to polar substrate like paper. POD thus provides a link that can combine polar paper and non-polar polyolefins.

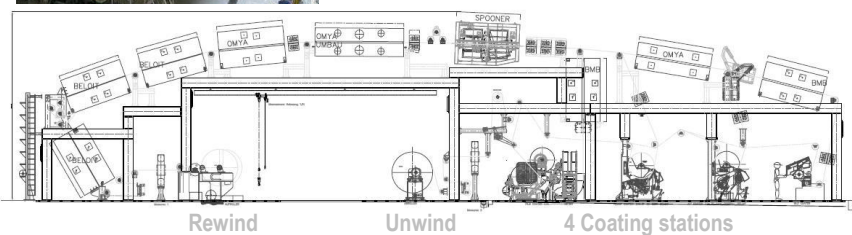
Pilot Coater for Aqueous Coatings

Samstagern, Switzerland



- Speed: 300 m/min (max : 2500 m/min)
- Coat weight: 4-6 g/m²
- Coating solids: 38-50%
- Board: uncoated, 255 g/m²
- Formulations:
 - F1: functionalized PP copolymer 1
 - F2: functionalized PP copolymer 2
 - F3: functionalized PE copolymer 3
 - F4: functionalized PE copolymer 4

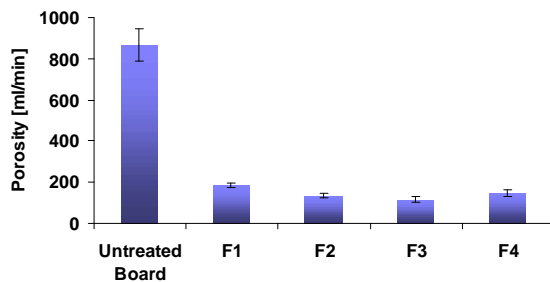
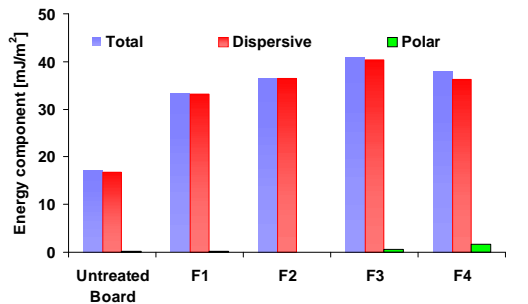
Length: 40m



The paperboard priming with POD was performed in our pilot coater facility for aqueous coatings in Switzerland. The coating speed was 300 m/min and we applied 4-6 g/m² coat weight with blade coating technology. The solids of the dispersions were 38-50%.

Four different POD formulations were applied on uncoated board. Two of the dispersions were based on polypropylene-copolymer and two were based on ethylene-copolymers.

Chemical vs. Mechanical Interlocking?



- Untreated board was hard sized indicated by low surface energy
→ can cause weak boundary layer / difficult to adhere surface
- POD coated board can increase surface energy
→ wetting improves
- POD coated board at applied coat weights demonstrate lower porosity
→ low porosity does not improve mechanical locking

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Surface energy was determined by analyzing contact angle of three liquids (de-ionized water, ethylene glycol and diiodomehtane) and calculating the energy components by using Fowkes method.

The results clearly show higher surface energy for POD primed paperboard that suggest improved wetting with extruded polyolefin. The fact that the paperboard has very low surface energy indicates hard sizing (high content of hydrophobizing agent) that can cause low surface energy but also a weak boundary layer leading to decreased adhesion.

The porosity was measured with Parker Print-Surf (PPS) instrument to evaluate the differences in porosity and hence understand the potential change in for mechanical interlocking. The data indicates that at these coat weights we are disadvantaged in mechanical interlocking.

Summary

Aqueous polyolefin dispersion application

- Allow high molecular weight polyolefin application as aqueous dispersion
- Standard liquid coating application systems can be utilized
- Functionalized ethylene / propylene based dispersions are targeted to interface with polar substrate like paper and board
- POD coatings deliver:
 - Polyolefin compatible layer for further converting
 - Surface energy modification
 - Reduce contamination / incompatibility related issues

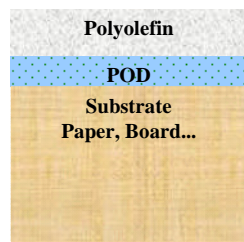
Polyolefin dispersions allow application of high molecular weight polyolefins with standard aqueous coating systems.

It is possible to disperse functionalized ethylene / propylene or other olefins allowing to match with the polyolefins used in next steps of converting.

POD coatings give compatible layer for further converting and acts as a link between non-polar polyolefin and polar paper surface, which otherwise are incompatible. We can modify the surface energy to improve wetting of paper and it is also possible to reduce contamination / incompatibility related issues by coating with POD

Extrusion coating and Heat Lamination on POD Primed Board

Low Energy Melt Application



Reducing Energy in melt application for

- Extrusion Coating / Extrusion Lamination
- Heat Lamination
- Heat Sealing

Melt Application - Heat Management

Heat transfer

- Heat conductivity
- Polyolefin melting behavior
- Structural considerations

Mass involved (dictated by process)

- Extrusion coating / extrusion lamination
- Heat Lamination
- Heat Sealing

Mass involved (How much polyolefin needs to be molten)

Extrusion coating/ extrusion lamination

- Melting by friction and heat
- Full mass of polymer molten

Heat Lamination

- Partial melting of polymer mass - restricted to surface layer
- Melting by heat radiation – IR heaters
- Melting by heat conduction – heated rollers
- Upstream process heat input

Heat Sealing

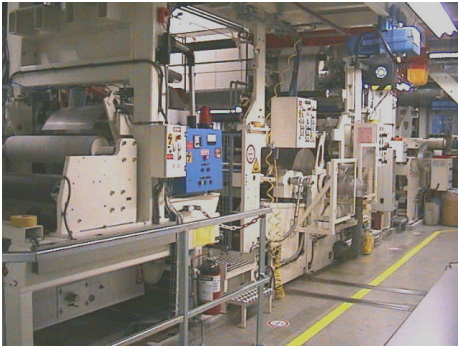
- Mostly requires full melting of the polyolefin layer to be sealed
- Heat Source defined by structural considerations (induction vs. impulse vs. electrical heaters vs. ultrasonic)

Extrusion Coating onto POD Primed Board

- Experiments - High speed coating
- Adhesion classification
- Energy considerations in extrusion

Experiments

High Speed Board Extrusion Coating



Enhanced Polyethylene (EPE) coatings

- Coat weight: 15 g/m²
- Line Speed: 500 mpm
- Set temperatures: 300°C & 330°C

Laminator settings

- Air-gap = 250 mm
- Nip off = -15 mm

Substrate pre-treatment

- With / without Corona

Resin Processing:

- Resin used : EPE (I2=11, D=0.911)

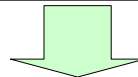
Extrusion Coating Temperature Settings:

- 3.5" x 32D Extruder - General Purpose Screw
- Profile 300C : Feed – water cooled
 - Barrel – Zones – 200/ 250/ 280/ 300/ 300°C
 - Adapter/ Pipes – 8x 300°C
 - Die – 10x 300°C
- Profile 330C : Feed – water cooled
 - Barrel – Zones – 200/ 250/ 300/ 330/ 330°C
 - Adapter/ Pipes – 8x 330°C
 - Die – 10x 330°C
- Corona pre-treatment – 10 kW

Manual Adhesion Classification

EPE extrusion coatings

Average Ratings	300 °C		330 °C	
	No Corona	With Corona	No Corona	With Corona
Substrates				
Ref Board	<2	<2	<2	<2
Board + F3	>3 to 4	>6 to 7	>4 to 5	7.4
Board + F2	>4 to 5	>6 to 7	>5 to 6	7.7
Board + F1	>6 to 7	7.6	7.6	8.2
Board + F4	>3 to 4	>4 to 5	>3 to 4	>4 to 5



Manual Adhesion Rating

- Scale: 1 (worst) - 10 (best)
- Good correlation to tensile peel strength ($R^2=0.73$)

- **High EPE adhesion ratings**
 - At low and high melt temperature
 - Preferably with Corona pre-treatment
- **POD coatings forming integral layer of board**

Manual Adhesion Classification

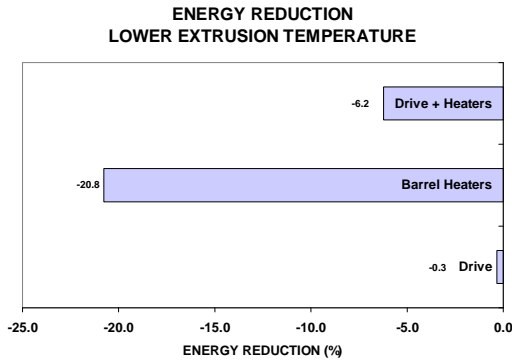
- Minimum 5 test persons
- Ratings are averages of multiple classifications

Observations of delamination failure included

- Delamination of coating from substrate
- Fiber Tear
- Coating Break
- Board Tear (internal strength)

Energy Savings in Extrusion

330°C vs. 300°C Set Temperature



6 % Total Extruder Energy Savings

- extruder drive and barrel heaters
- No significant contributions from extruder drive
- Energy savings on other heated hardware components not included.

21 % Energy Savings in Barrel Heaters

- 60% of this contribution is occurring in heater zones 3 and 4

Future Energy Savings ?

- Direct drive extruders (est. 5%)
- High speed extruders (est. 5%)

Process data acquisition:

- Davis Standard "Blitz" data download combined with internally developed trial data acquisition tool
- 12 data points collecting energy consumption by heater zone and drive motor collected

Resin processed:

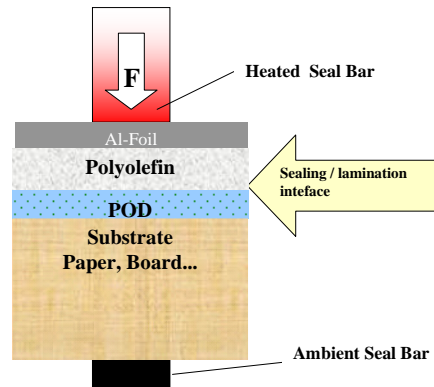
- EPE (I2=11, D=0.911)

Heat Lamination / Heat Sealing

POD Primed Board or Paper

- Heat Sealing / Heat Lamination
- Experiments
- Results

Heat Sealing / Heat Lamination



Heat Sealing of thin Al-foil coatings to web based materials represent an efficient model to judge

- Polyolefin heat sealing performance
- Estimate polyolefin heat lamination conditions

Heat Sealing Conditions:

- Lab Heat Sealer: Kopp model: SGPE 20
- Top Sealing Bar – Temperature varied to max 160C
- Bottom Seal Bar – Ambient temperature
- Seal bar width = 5mm
- Sealing Pressure = 5N/mm²
- Sealing time = 0.5sec

Heat Seal Strength Measurements:

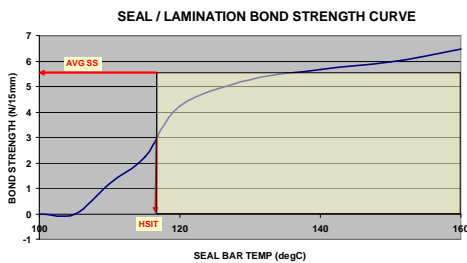
- Lloyd Instruments Ltd, Model LR5K
- Measured on tensile tester
- Crosshead speed = 100mm/min
- Sample width = 15mm

Coated Al-foil used as excellent heat conductor. Other potential heat sources can be

- Heated rollers
- Induction heating
- Hot air
- Flame
- IR heating

Heat Sealing / Heat Lamination

POD Primed vs. Reference Board



Increased seal / lamination strength

- Package integrity
- Broad application range

Reduced seal / lamination temperature

- Converting speed
- Wide operating range

Al-Sealant to Board with vs. without POD	Reduction Bond Initiation Sealing + Lamination	Increase Average Bond Strength Sealing + Lamination
	[°C]	[%]
LDPE 290°C	-12	325
LDPE 320°C	-13	170
EPE 1 290°C	-13	740
EPE 2 320°C	-9	500
POP 290°C	-21	525
POP 320°C	-6	320
PBP290°C	-14	345
EAA (7-9%AA) 290°C	-12	265
EAA(290°C)	-12	415

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Heat sealing of coated Al-foil to substrates with and without POD surface architecture

➤ POD coated board sample F1

➤ Sealant layers coated or coextrusion coated at indicated process set temperatures in °C.

Definition of Heat Seal strength / Lamination strength and Bond Initiation temperature

➤ Obtained with overlay of heat sealing data with POD primed vs. unprimed board substrate

➤ Measurements above 3N/15mm considered of practical value

➤ Average of seal strength measurements over temperature range to max 160°C

➤ Heat seal initiation temperature defined as temperature at which measurable results were achieved (generally higher than 0.5N/15mm)

Nomenclature:

Avg SS : Average Seal Strength

HSIT: Heat Seal Initiation Temperature

Conclusions

New adhesion solutions



Dow Europe GmbH
Centers of Excellence
Paper and Board Coating
Extrusion coating

- **Paper and board manufacturers can**
 - Conveniently integrate polyolefin components
- **Extrusion coaters can**
 - Save energy by reduced processing temperatures
 - Reduce emissions to the air due to lower oxidation
 - Increase line speed
- **Heat laminators can**
 - Directly laminate polyolefins to POD coated paper or board
- **Converters can**
 - Directly heat seal polyolefins to paper and board
 - Take advantage of high laminate integrity



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Paperboard priming with aqueous polyolefin dispersion offer paperboard manufacturer or extrusion coater a convenient way to integrate polyolefin components on paperboard surface enabling improved downstream processing.

Low temperature extrusion coating process allow marked energy savings and reduced oxidation leading to reduced emissions, improved odor/taste and possibilities to increase line speed.

In heat lamination step POD allow completely new way to integrate polyolefins to paper or board.

In later converting step POD coated board or paper allow direct heat sealing and in case of laminated structure improved integrity can be observed.

HYPOD™ polyolefin dispersion
primed paper and board solutions enable

Heat lamination & Heat sealing

to a wide range of polyolefin types including

DOW LDPE Low density Polyethylene **DOW HDPE** High density Polyethylene

DOW PP Polypropylene

INSPIRE™ Performance Polymers

DOWLEX™ Linear Low Density Polyethylene

ELITE™ Enhanced Polyethylene

ATTANE™ ULDPE Polyethylene

AFFINITY™ Polyolefin Plastomers

AMPLIFY™ Functional Polymers

PRIMACOR™ Copolymers

VERSIFY™ Plastomers and Elastomers

INFUSE™ Olefin Block Copolymers

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Substrate priming of paper or board with aqueous polyolefin dispersion enables

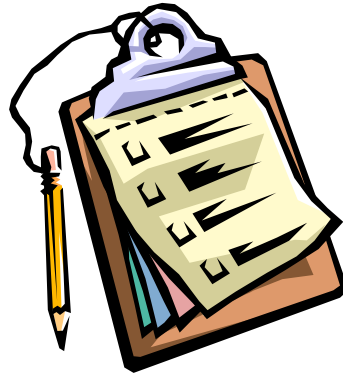
- Direct heat induced sealing to a wide range of polyolefins
 - With significantly improved heat induced sealing or lamination of polyolefin's
 - Reduced temperature and wider sealing range

- Predicted heat induced lamination to a wide range of polyolefins
 - With solid lamination strength
 - Favorable processing conditions

Thank You – Merci

PRESENTED BY
Jouko Vyorykka and Charly Zuercher

DOW Europe GmbH



***Please remember to turn
in your evaluation sheet...***