THE EFFECT OF PAPERBOARD COATING TREATMENTS ON POLYETHYLENE ADHESION DURING EXTRUSION COATING

Don Ventresca
Trinseo LLC, Petrolia, Ontario, N0N 1R0, Canada

Greg Welsch
Trinseo LLC, Midland, Michigan 48674, USA

Kelly Frey
Chevron Phillips Chemical Company LLC, Bartlesville, Oklahoma 74003-6670, USA

ABSTRACT

This paper presents results from a study evaluating the effect of changes to the composition, structure, and application of paperboard coating on the adhesion of extruded low density polyethylene (LDPE) and also the extrusion coating conditions required to achieve proper LDPE adhesion to these coatings. The main reasons for applying a polyolefin extrusion coating to paperboard are: to provide moisture barrier in a liquid packaging application; to increase tear, scuff and puncture resistance; to provide grease, oil and chemical resistance; to provide adhesion between multiple substrates; and to provide a heat sealable package or carton.[1] The main components of a paperboard coating include calcium carbonate, kaolin clay and a variety of binders. Latex binders used in paperboard coatings are typically styrene-butadiene (SB), styrene-acrylate (SA), vinyl acrylic (VA) and polyvinyl acetate (PVAc) based compositions. Latex binders are used in the coatings to provide adhesion to the basesheet and cohesion between the pigments.[2] Additionally for paperboard coatings, the binder must also provide the appropriate dry and wet strength as this is important for the printing, finishing and converting of the paperboard as well as for final carton use.

In this study, coating composition variables included three binder systems. SA, SB and a novel Starch Containing Emulsion (SCE) each evaluated at two different binder levels in both coating layers. Additionally three different calcium carbonate/clay ratios in the top-coat were studied. Other variables included the coat weight split between the pre-coat and top-coat and also the impact of calendering load.

In the extrusion coating process, the variables studied were time in air gap, presence of a primer, LDPE thickness, and line speed. This broad range of conditions would help determine which of the paperboard coating treatments would most likely experience LDPE adhesion failures or conversely satisfactory LDPE adhesion.

INTRODUCTION

This paper presents results from a study evaluating the effect of changes to the composition, structure and application of paperboard coating on the adhesion of extruded low density polyethylene (LDPE) film and also the extrusion coating conditions required to achieve proper LDPE adhesion to these coatings. The main reasons of applying a polyolefin extrusion coating to paperboard include: to provide moisture barrier in a liquid packaging application; to increase tear, scuff and puncture resistance; to provide grease, oil and chemical resistance; to provide adhesion between multiple substrates; and to provide a heat sealable package or carton. The main components of a paperboard coating include calcium carbonate, kaolin clay and a variety of binders. Latex binders used in paperboard coatings are typically styrene-butadiene (SB), styrene-acrylate (SA), vinyl acrylic (VA) and polyvinyl acetate (PVAc) based compositions. Latex binders are used in the coatings to provide adhesion to the basesheet and cohesion between the pigments. Additionally for paperboard coatings, the binder must also

1

provide the appropriate dry and wet strength as this is important for the printing, finishing and converting of the paperboard as well as for final carton use.

In this study, coating composition variables included three binder systems. SA, SB and a novel Starch Containing Emulsion (SCE), each were evaluated at two different levels in both coating layers. Additionally, three different calcium carbonate/clay ratios in the top-coat were studied. Other variables included the coat weight split between the pre-coat and top-coat and also the impact of calendering load.

The extrusion coating process involves the application of a molten thermoplastic coating onto a moving substrate web (paperboard, paper, plastic films, etc.). Adhesion of the thermoplastic coating to the chosen substrate is achieved by several means through various combinations of physical and chemical pre-treatments. These include flame or corona pre-treatment of the substrate web, the use of adhesion promoting primers, and most importantly oxidation of the thermoplastic LDPE melt film.

Oxidation is achieved by extrusion of LDPE at elevated temperatures. For example extrusion coating LDPE grades are typically extruded at approximately 310 to 332 $^{\circ}$C, creating functional groups via this reaction[3]:

$(CH_2-CH_2)_n$ $\xrightarrow[\Delta]{O_2 \ or}$ Oxidized Polyethylene

Polyethylene
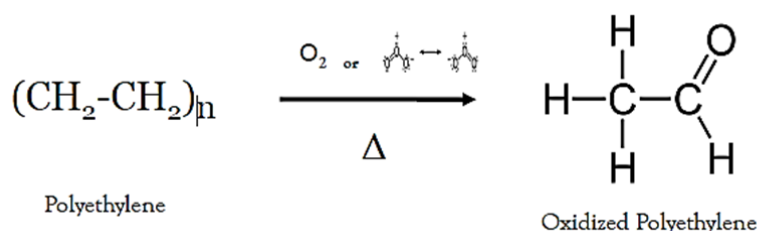
The oxidation process can be further optimized by adjusting the time in the air gap. Time in the air gap can be adjusted by adjusting the distance between the die lips and the LDPE-substrate nip point, or by adjusting line speed, or a combination of these two. Longer time in the air gap will result in a longer exposure of the molten LDPE to oxygen in the air, resulting in more oxidation.[3]

A common primer used in LDPE extrusion on coated paperboard is polyethylenimine (PEI) which has primary and secondary amine reactive groups (R'NH$_2$). The functional groups on the PEI will bond with the functional groups on the oxidized polyethylene, via hydrogen and covalent bonding, forming amide linkages.[4, 5]

Oxidized Polyethylene Melt   +   Crosslinked PEI Primer

Bonded Product

Amide Linkage
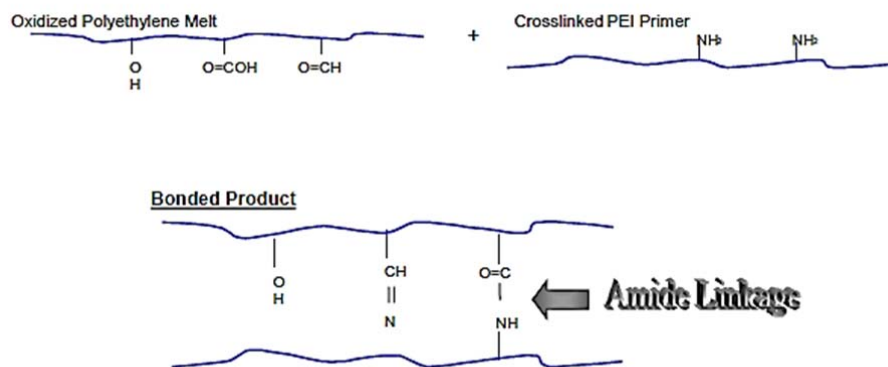
It is also hypothesized that the PEI amine groups will bond with the carboxylic functional groups on the SA or SB binders that are in the paperboard coating, resulting in improved adhesion of the LDPE.[4, 5]

$$RCOOH + R'NH_2 \rightarrow RCONHR' \ (H_2O)$$

2
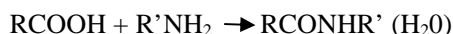
In this study, the extrusion coating process variables studied were time in air gap, substrate pre-treated with/without primer, and LDPE coating thickness. This broad range of conditions would help determine which of the paperboard coating treatments would most likely experience LDPE adhesion failures or conversely satisfactory LDPE adhesion.

There are three modes of adhesion failures of the LDPE layer with the paperboard coating.[6] One mode is insufficient mechanical interlocking of the LDPE with the surface of the pigmented coating. This can be affected by the roughness and surface porosity of the paperboard coating and also the rheological and thermal properties of the LDPE; which in turn can be impacted by the extrusion conditions and primer used in the extrusion coating process. A second mode, cohesive failure, occurs when the coating strength is insufficient or when there is a weak layer or interface. A third mode of failure occurs if the wettability of the coating layer by the LDPE layer melt is insufficient.

EXPERIMENTAL

Formulations Evaluated

The coating formulations used in this study are described in Table 1 (see also Appendix A). These formulations were chosen as a general representation for a bleached board coating suitable for a liquid packaging application.

Table 1. Coating formulations.

Formulations	Pre-Coat	Top-Coat A	Top-Coat B	Top-Coat C
	Parts	Parts	Parts	Parts
Coarse Calcium Carbonate	100			
Fine Calcium Carbonate		60	50	40
Fine Clay		40	50	60
Latex Level - SA or SB or SCE	16 or 14	15 or 13	16	17
Thickener (polyacrylate)	0.3	0.2	0.2	0.2
Solids, %	66	65	65	65
pH	8.5	8.5	8.5	8.5

The binder system was one of the variables evaluated to determine its effect on polyethylene adhesion. In addition to a styrene acrylate (SA) binder (Tg 20 $^{\circ}$C, particle size 120 nm), we also evaluated a typical styrene butadiene (SB) binder (Tg 5 $^{\circ}$C, particle size 130 nm) used in paperboard coatings, and a novel starch containing emulsion (SCE) (SA based, Tg 20 $^{\circ}$C, particle size 120 nm). Each binder was evaluated at the standard concentration and 2 parts lower in both the pre-coat and top-coat layers. An additional condition was included where the SA binder was also evaluated with approximately 0.5% additional surfactant (diester sulfosuccinate type) to determine if the standard flame treating or primer used during extrusion coating would be enough to overcome the change in surface energy as a result of the additional surfactant. These conditions were all run with a 60:40 split of fine calcium carbonate to fine clay (Top-Coat A).

Another variable evaluated was the effect of the top-coat pigment system on the polyethylene adhesion, also shown in Table 1. In addition to the 60:40 split (Top-Coat A) of fine calcium carbonate to fine clay, we evaluated a 50:50 (Top-Coat B) and 40:60 (Top-Coat C) split, with the styrene acrylate binder increased from 15 to 16 and 17 parts respectively. Binder level was increased to account for the higher binder demand typically required as the clay level was increased.

3

One of the process variables evaluated was the coat weight split between the pre-coat and top-coat. This was included to simulate "out of control" conditions that may occur on a paperboard machine during process upsets. These conditions are shown in Table 2. It is suspected that a low pre-coat coat and low top-coat coat weight will result in delamination failures in extrusion coated paperboard. The following table shows the coat weight study that was conducted with the SA based pre-coat and top-coat (Top-coat A) formulations.

Table 2. Coat Weight Study

Coat Weights	Pre-Coat	Top-Coat	Total
	g/m^2	g/m^2	g/m^2
Control	11.8	10.3	22.1
Low Pre-Coat	7.3	10.3	17.6
High Pre-Coat	16.2	10.3	26.5
Low Top-Coat	11.8	5.7	17.5
High Top-Coat	11.8	15.8	27.6

Paperboard Coating Process

The formulations were applied onto 12 point bleached board at the Trinseo Pilot Coater in Midland, MI (Figure 1). The pre-coats were applied using an applicator roll coater and a rigid blade (0.38 mm/35°) configuration. The standard pre-coat weight was 11.8 gsm. The top-coats were applied using the applicator roll coater and a bent blade (0.38mm square tip) configuration at a standard top-coat weight of 10.3 gsm.

All the double coated board conditions were hot soft calendered off-line at equivalent conditions (1 nip, 350 m/min, 90 °C, 87 kN/m). One control roll was calendered at a higher load to produce a smoother surface as an additional trial point (1 nip, 150 m/min, steel roll temperature 107 °C, nip load 87 kN/m).

Figure 1 – Trinseo Pilot Coater – Midland, MI USA
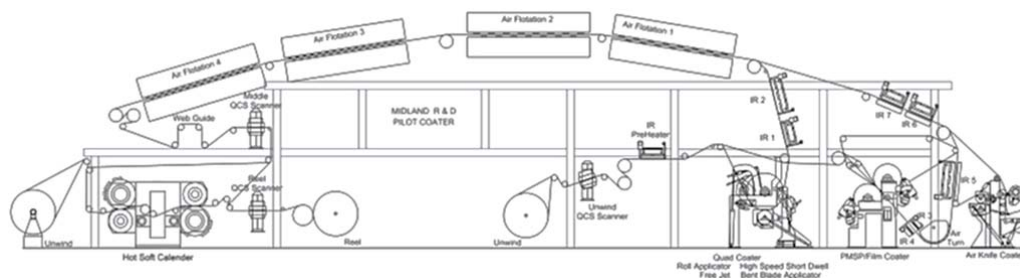
Extrusion Coating Process

The rolls of 12 point coated bleached paperboard (16 double coated conditions) were utilized for extrusion trials with the objective to determine how different coating treatments processed during extrusion coating, paying particular attention to the bond between the LDPE extruded coating and the coated paperboard.

The sample rolls were evaluated on the Chevron Phillips Guardian Packaging extrusion coating pilot line, located in Bartlesville, OK. This pilot extrusion coating line is only slightly smaller than some extrusion coating production lines. It is considered representative of production lines used in the industry with similar capabilities, throughputs, and line speeds. The size of this line is also such that results typically "scale-up" very well to real world manufacturing.

4

Specifications of the extrusion coating pilot line are shown in Table 3. A schematic of the extrusion coating pilot line is shown in Figure 2.

Table 3. Chevron Phillips Extrusion Coating Pilot Line Specifications.

Extruders	Laminator
A = 4.5" Egan	100 - 1800 FPM
B = 2.5" Egan	Substrates: 0.5 Mil to 27 Pt. Board
C = 2.5" Egan	Core Sizes: 3", 6" & 12" - Primary
D = 2.5" Egan	Core Sizes: 3" & 6" - Secondary
	Primary Unwind - Maximum 54" O.D.
Feedblock and Die	Secondary Unwind - Maximum 24" O.D.
Nordson 40" Die with Auto Gauge Control	NDC Beta and IR Gauging System with APC
Nordson Coextrusion 5 Layer Feedblock	Minimum Web Width = 24"
	Maximum Web Width = 32"
	Minimum Die Opening = 24"
Direct Gravure Priming Station	**Three Chill Rolls**
Dryer Length = 16.5 Feet	Gloss = 0.5 rms
Enclosed Doctor Blade System	Mirror Pocket = 10-13 rms
Ceramic Coated Applicator Roll (60 rms)	Matte A = 75 rms
Other Pretreatment	
Bare Roll and Conventional Corona Pretreater	**In-line Drying Capacity**
Flynn Burner Flame Pretreatment	Gas Fired Drum Dryer
	Digital Controlled Maxon Burner

Figure 2 - Chevron Phillips Pilot Line – Bartlesville, OK USA
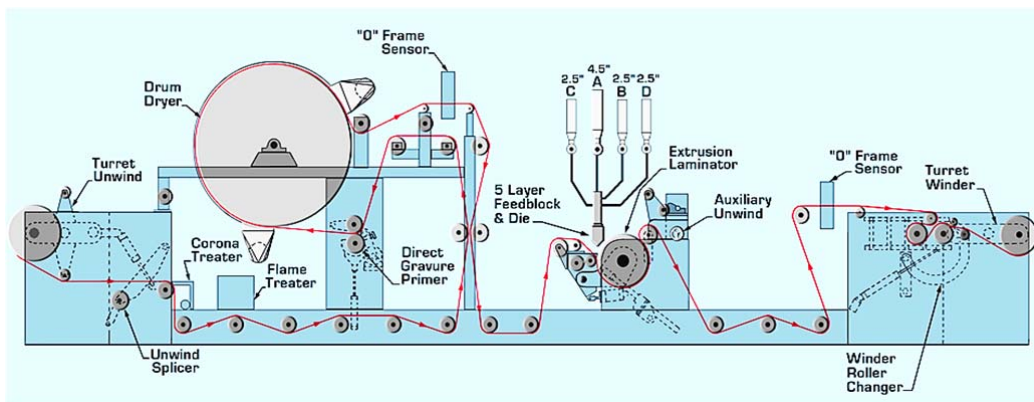
For this study the extrusion coating pilot line was configured with a matt chill roll (75 rms); monolayer configuration (11.4 cm extruder); flame plasma pre-treatment (12,000 btu); primer pre-treatment of 1.0 gsm wet of Mica H760 PEI (Polyethylenimine) primer diluted with 1 parts primer and 1 parts water; primer drying at 93 ^{o}C; 24.5 kN/m nip pressure; and a target process melt temperature of 310 ^{o}C.

For the start of this experiment the extrusion coating pilot line was lined-out using 59 gsm natural kraft paper, using Marlex® 4517 LDPE (5.1MI, 0.923 g/cc density extrusion coating grade), until a uniform coating weight of 25.4 micrometers +/-5% was achieved, at a line speed of 198 mpm. Once lined-out then the rolls of 12 point bleached coated paperboard were evaluated on the pilot line and extrusion coated with Marlex® 4517 LDPE under the 6 treatment conditions shown in Table 4.

5

Table 4. Extrusion Coating conditions.

	LDPE Coating Wt. (microns)	Line Speed (mpm)	Air Gap Distance (cm)	Time in Air Gap (milli-sec)	Substrate Pretreated w/ Primer
Condition 1	25.4	198	17.8	100	Primer
Condition 2	25.4	198	17.8	100	No Primer
Condition 3	25.4	198	25.4	150	No Primer
Condition 4	25.4	198	25.4	150	Primer
Condition 5	25.4	99	25.4	300	Primer
Condition 6	63.5	99	25.4	300	Primer

These conditions were selected in order to provide various levels of adhesion performance, with better adhesion typically obtained with longer time in the air gap, with the addition of primer, and with higher LDPE coating weights. In order to measure the adhesion level of the LDPE extrudate to the pigmented coated substrate and to make comparisons between the various rolls, it was important to select a set of conditions where adhesion was different amongst the various conditions. It was also imperative to select a set of conditions where all of the samples did not have excellent bond in order to differentiate between the coating treatments selected. The coated board rolls were extruded in random order.

Immediately after extrusion coating the six different conditions of each of the sixteen coated roll treatments, samples were evaluated for adhesion by performing a T-peel test on each condition for each sample. The T-peel test measures the amount of force required to remove the LDPE extrudate coating from the substrate. The T-peel test is done with a 2.54 cm by 15 cm sample specimen on a Friction Peel Tester. The separated LDPE film tab and the coated board tab are each clamped into separated grips of the tester and then separated at a rate of 127 mm/min. The average peel force is recorded in grams.   All samples were also again tested for T-Peel one week later to look at any effects of aged bond. It was also noted if the delamination occurred within the pigmented coating, or between pigmented coating interfaces, instead of at the LDPE coating / pigmented coating interface. T-Peel test is shown below in Figure 3.

Figure 3 – Example of T-Peel Strength test.
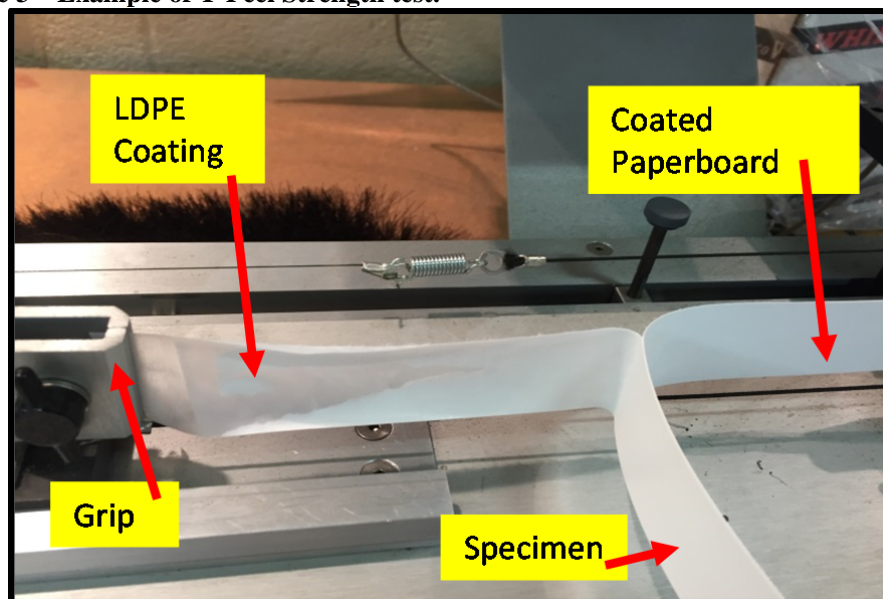
RESULTS AND DISCUSSION

Effect of Binders

The coated board characterization data for the binder experiment can be found in Table 5. Samples that are statistically different than the control SA condition are highlighted in bold text. The SA binder at level 1 was chosen as the Control for the study as this represents a typical condition for coated bleached board.

Table 5. Characterization Data for Binder Type Experiment.

	PPS Roughness (micron)	Hagerty Porosity (s/10cc)	Surface Energy Total (nN/m)	IGT Dry Pick (cm/s)	Hot Melt Glue (% Fiber Tear)	Aqueous Glue (% Fiber Tear)
SA Level 1 Control	1.56	364	45.1	326	82.5	100
SA Level 2	**1.24**	**213**	44.7	331	**97.5**	100
SB Level 1	**1.30**	**1437**	**53.9**	375	**47.5**	100
SB Level 2	**1.06**	**815**	**56.4**	342	**5.0**	100
SCE Level 1	**1.43**	327	**46.5**	354	**100**	100
SCE Level 2	**1.10**	**174**	**47.9**	360	**100**	100
SA w Surfactant	**1.05**	**468**	**42.9**	349	**100**	100

Changing binder type and reducing binder levels can change smoothness (PPS) significantly. We observed better smoothness with lower binder levels, most likely due to less surface disruption (pigment locking) as the coating dries or as a result of the increased porosity making the coating more compressible during calendering. The SB system gives a lower smoothness, most likely due to a lower film formation temperature. The SA condition with additional surfactant gave a lower PPS compared to the control. This is surprising but it is possible that the surfactant could have improved leveling of the coating during application and drying.

Binder changes also had significant effects on Hagerty Porosity. The SB binder gave a significantly less porous (higher value) coating. The SCE gave a slightly more porous coating than the control. Reducing the binder level resulted in a more porous coating (lower value).

The surface energy data shows that the SB produced a coating with significantly higher surface energy than the SA and SCE systems. The SCE also has a slightly higher surface energy than the SA condition at equal binder level. Adding surfactant to the SA binder resulted in a reduction in surface energy as expected.

The IGT Dry Pick data trends, although not statistically significant, indicates that the SB, SCE, and the SA with added surfactant systems had slightly higher IGT dry pick than the SA samples. Only the SB binder showed a reduction in dry pick with the decrease in binder level. The SA and SCE samples did not show a significant difference with the binder reduction but these also started at lower cohesive strength than the SB systems tested.

The hot melt glue data showed that the SB system was significantly lower in fiber tear than the other systems. This was surprising since good dry pick strength was observed. This observation may be related to the reduced coating porosity and lower mechanical adhesion due to higher smoothness observed for these conditions. All samples had 100% fiber tear in the aqueous glue testing.

The results of the T-Peel testing can be found in Figure 4.

7

Figure 4. T-Peel Strength on Extrusion Coated Samples – Binder Experiment.

The observations on the T-Peel strength data are summarized below by each extrusion condition.

25.4 micron LDPE coating; 198 mpm; 17.8 cm air gap, 100 ms in air gap; with primer:
With the lowest time in the air gap, the low peel strength indicates poor adhesion of the LDPE to the coating for all coating samples. No coating pick was observed on the peeled LDPE coating, so the failure mechanism was adhesive failure.

25.4 micron LDPE coating; 198 mpm; 17.8 cm air gap, 100 ms in air gap; without primer:
Without primer, the adhesion was poor as well. No coating pick was observed on the peeled LDPE coating, so the failure mechanism was adhesive failure.

25.4 micron LDPE coating; 198 mpm; 25.4 cm air gap, 150 ms in air gap; without primer:
With increased time in the air gap, more oxidation of the LDPE is achieved, and we observed improved adhesion with coating cohesive failure (coating removed). We observed little difference in peel strength when the binder level was reduced for the SA and SCE systems. For SB, there was a drop in peel strength at the reduced level similar as to what was observed with IGT dry pick. The Control SA with added surfactant gave lower peel strength and even with the flame pre-treatment, the added surfactant still appeared to interfere with LDPE coating adhesion.

25.4 micron LDPE coating; 198 mpm; 25.4 cm air gap, 150 ms in air gap; with primer:
With primer the peel strength was increased significantly for most samples. All samples experienced cohesive coating failure. The SA Level 1 Control failure mechanism was a combination of coating cohesive failure but also the 25.4 micron thick LDPE coating broke during testing due to the adhesion bond (peel) strength being greater than the LDPE tensile strength. The SB Level 1 sample had two failure mechanisms, a combination of cohesive coating failure but also fiber tear. Reduction of SB level did reduce the peel strength significantly. SCE had a higher peel strength on the reduced level sample. The SA with added surfactant had a high peel strength but the LDPE-coating bond strength was lower than the Control which experienced the LDPE coating break during testing. So even with the flame pre-treatment, added surfactant can adversely affect LDPE adhesion.

8

25.4 micron LDPE coating; 99 mpm; 25.4 cm air gap, 300 ms in air gap; with primer:
This extrusion condition increased oxidation of the LDPE significantly and will increase adhesion strength. All samples experienced "lock up" during the peel testing. This is where the LDPE coating breaks almost immediately at the start of the peel test, due to the adhesion strength being greater than the LDPE tensile strength at 25.4 micron thickness. This indicates that the LDPE-coating bond strength, coating cohesive strength and the coating-fiber adhesion are greater than the LDPE tensile strength. So with extrusion conditions producing the greatest oxidation of the LDPE, coating changes such as those evaluated here, may not affect the LDPE adhesion adversely.

63.5 micron LDPE coating; 99 mpm; 25.4 cm air gap, 300 ms in air gap; with primer:
In an attempt to obtain meaningful T-peel data, at this condition with the highest LDPE oxidation, the LDPE coating thickness was increased by 2.5 times to 63.5 microns. It is expected that higher strengths are obtained due to the slower quench time of the LDPE and greater penetration of the molten LDPE.[7] All samples experienced cohesive coating failure. The SCE samples gave significantly higher peel strength than the SA and SB samples. The SCE Level 1 sample also had fiber tear. It is proposed that under this high oxidation extrusion condition, the PEI primer creates a greater network of amide linkages with the coating due to bonding to the –OH groups in the starch component of the SCE. It is also possible that the higher porosity of these SCE coatings could have allowed better penetration of the primer and anchorage of the LDPE, resulting in higher peel strength. The Control SA with additional surfactant also has a higher peel strength than the Control SA sample.

After one week of aging, the T-Peel strength was re-tested on these samples. The % change relative to the "off the line" data, reported above, is shown in Table 6. There was no change to the extrusion condition 5 samples – after aging these still had "lock-up" performance. For the samples with 25.4 microns of LDPE, the conditions with primer pre-treatment had the biggest loss in T-peel strength, indicating that the amide linkages can deteriorate with time. In condition 4, the samples with lower binder levels had a smaller decrease in strength. In extrusion condition 6, the SA and SCE binder had an increase in strength while the other samples all experienced a decrease.

Table 6. Aged T-Peel Strength Data.

% Change in T-Peel Strength after 1 week	Cond 1 25.4 microns film 198 mpm; 17.8 cm air gap; 100 ms; Primer	Cond 2 25.4 microns film 198 mpm; 17.8 cm air gap; 100 ms; No Primer	Cond 3 25.4 microns film 198 mpm; 25.4 cm air gap; 150 ms; No Primer	Cond 4 25.4 microns film 198 mpm; 25.4 cm air gap; 150 ms; Primer	Cond 5 25.4 microns film 99 mpm; 25.4 cm air gap; 300 ms; Primer	Cond 6 63.5 microns film 99 mpm; 25.4 cm air gap; 300 ms; Primer
SA Level 1 Control	**-39**	-11	-1	**-35**	n/a	**33**
SA Level 2	**-26**	13	-27	12	n/a	**24**
SB Level 1	5	39	-18	**-27**	n/a	-13
SB Level 2	**-52**	53	-26	-8	n/a	-2
SCE Level 1	**-67**	-36	36	**-57**	n/a	27
SCE Level 2	**-52**	8	39	-3	n/a	-7
SA w Surfactant	**-87**	-31	69	**-72**	n/a	-9

Effect of Top-Coat Pigment

The coated board characterization data for the top-coat pigment experiment can be found in Table 7. Samples that are statistically different than the control are highlighted in bold text.

9

Table 7. Characterization Data for Top-Coat Pigment Experiment.

	PPS Roughness (micron)	Hagerty Porosity (s/10cc)	Surface Energy Total (nN/m)	IGT Dry Pick (cm/s)	Hot Melt Glue (% Fiber Tear)	Aqueous Glue (% Fiber Tear)
Top-coat A – 60/40	1.56	364	45.1	326	82.5	100
Top-coat B - 50/50	**1.18**	**474**	43.1	364	**100**	100
Top-coat C - 40/60	**1.44**	**721**	43.6	304	**100**	100

There was a reduction in PPS with the 50/50 carbonate/clay system but the PPS of the 40/60 carbonate/clay system was only slightly lower than the control. We expected a smoother surface with higher clay concentration but the higher binder level may have offset this expected change.

Higher Hagerty values (less porous coatings) were seen with increasing clay concentration in the top-coat layer. The increased binder level that was used with the increasing clay concentration would also contribute to the lower coating porosity.

Increasing clay concentration, caused a reduction in surface energy.

We saw an increase in Dry Pick with the 50/50 system but the 40/60 system is lower than the Control. The increased binder level that was used with the increasing clay concentration typically contribute to higher IGT values.

In the Hot Melt Glue testing, we see that both the 50/50 and 40/60 samples are slightly higher than the Control. The increased binder level that was used with the increasing clay concentration would also contribute to the higher glue strength. In the Aqueous Glue testing, all samples had 100% fiber tear.

The results of the T-Peel testing can be found in Figure 5.

Figure 5. T-Peel Strength on Extrusion Coated Samples - TC Pigment Experiment.
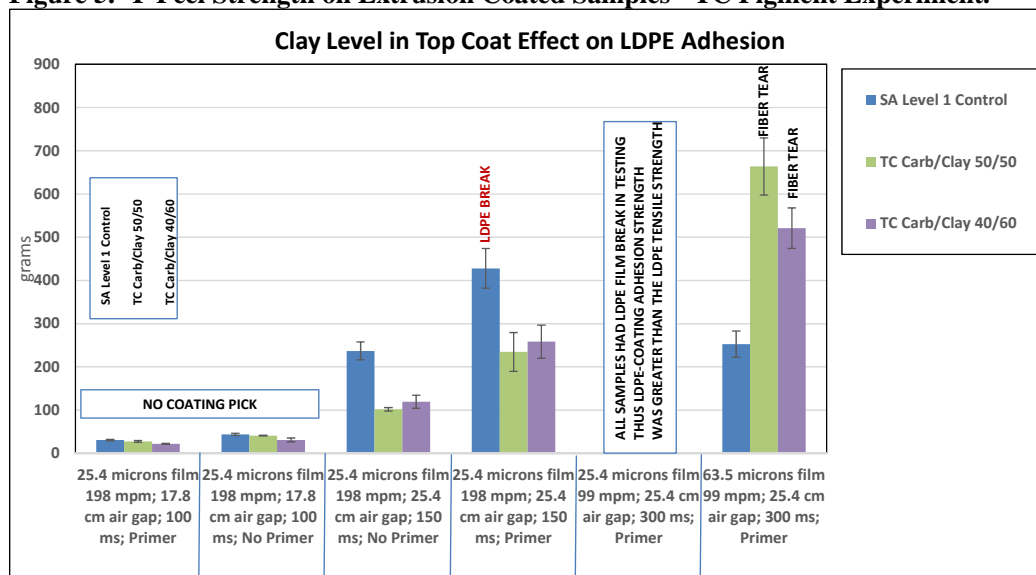
The observations on the T-Peel strength data are summarized below by each extrusion condition.

10

25.4 micron LDPE coating; 198 mpm; 17.8 cm air gap, 100 ms in air gap; with primer:
With the lowest time in the air gap, the low peel strength indicates poor adhesion of the LDPE to the coating for all coating samples. No coating pick was observed on the peeled LDPE coating, so the failure mechanism was adhesive failure.

25.4 micron LDPE coating; 198 mpm; 17.8 cm air gap, 100 ms in air gap; without primer:
Without primer, the adhesion was poor as well. No coating pick was observed on the peeled LDPE coating, so the failure mechanism was adhesive failure.LDPE

25.4 micron LDPE coating; 198 mpm; 25.4 cm air gap, 150 ms in air gap; without primer:
With the longer air gap, resulting in more oxidation of the LDPE, we saw improved adhesion and coating cohesive failure as there was coating pulled from the coating layer. The peel strength of the samples with higher clay content in the top-coat are significantly lower than the Control.

25.4 micron LDPE coating; 198 mpm; 25.4 cm air gap, 150 ms in air gap; with primer:
With primer, the peel strength was increased significantly. All samples experienced coating cohesive failure. The Control SA failure mechanism was a combination of coating cohesive failure but also the 25.4 micron thick LDPE coating broke during testing due to the adhesion bond strength being greater than the LDPE tensile strength. The Control SA had significantly higher peel strength than the samples with the higher clay content.

25.4 micron LDPE coating; 99 mpm; 25.4 cm air gap, 300 ms in air gap; with primer:
This extrusion condition increased oxidation of the LDPE significantly and will increase adhesion strength. All samples experienced "lock up" during the peel testing.

63.5 micron LDPE coating; 99 mpm; 25.4 cm air gap, 300 ms in air gap; with primer:
In an attempt to obtain meaningful T-peel data, at this condition with the highest LDPE oxidation, the LDPE coating thickness was increased by 2.5 times to 63.5 microns. All samples experienced cohesive coating failure. The samples with higher clay content in the top-coat had higher peel strength than the Control, plus these samples both experienced fiber tear. There is no obvious explanation for this improved performance but it confirms that under the highest oxidation condition and slowest primer drying, coatings could experience improved performance.

After one week of aging, the T-Peel strength was re-tested on these samples. The % change relative to the "off the line" data, reported above, is shown in Table 8. There was no change to the extrusion condition 5 samples – after aging these still had "lock-up" performance. For the samples with 25.4 microns of LDPE, the conditions with primer pre-treatment had the biggest loss in T-peel strength, especially in condition 4. In condition 4, the samples with lower binder levels had a smaller decrease in strength. Top-coats B and C, with the higher clay levels, overall had the biggest loss in T-peel strength compared to the control Top-coat A.

Table 8. Aged T-Peel Strength Data.

% Change in T-Peel Strength after 1 week	Cond 1 25.4 microns film 198 mpm; 17.8 cm air gap; 100 ms; Primer	Cond 2 25.4 microns film 198 mpm; 17.8 cm air gap; 100 ms; No Primer	Cond 3 25.4 microns film 198 mpm; 25.4 cm air gap; 150 ms; No Primer	Cond 4 25.4 microns film 198 mpm; 25.4 cm air gap; 150 ms; Primer	Cond 5 25.4 microns film 99 mpm; 25.4 cm air gap; 300 ms; Primer	Cond 6 63.5 microns film 99 mpm; 25.4 cm air gap; 300 ms; Primer
Top-coat A - 60/40	**-39**	-11	-1	**-35**	n/a	33
Top-coat B - 50/50	-14	**-78**	-17	**-73**	n/a	0

11

	PPS Roughness (micron)	Hagerty Porosity (s/10cc)	Surface Energy Total (nN/m)	IGT Dry Pick (cm/s)	Hot Melt Glue (% Fiber Tear)	Aqueous Glue (% Fiber Tear)
Top-coat C - 40/60	**-73**	**-39**	11	**-85**	n/a	**-39**

Effect of Calendering

The coated board characterization data for the calendering experiment can be found in Table 9. Samples that are statistically different than the control are highlighted in bold text.

Table 9. Characterization Data for Calendering Experiment.

	PPS Roughness (micron)	Hagerty Porosity (s/10cc)	Surface Energy Total (nN/m)	IGT Dry Pick (cm/s)	Hot Melt Glue (% Fiber Tear)	Aqueous Glue (% Fiber Tear)
Standard Calendering	1.56	364	45.1	326	82.5	100
Heavy Calendering	**1.18**	**650**	45.9	**370**	90.0	100

As expected, there was a significant reduction in PPS with calendering at a higher calender temperature and slower line speed.   Heavier calendering conditions also produced lower porosity (higher Hagerty value), and slightly higher IGT Dry Pick.   The slightly higher strength may be due to higher compression resulting in improved contact of the binder particles with the pigment particles or further coalescence of the binder under these conditions, producing a stronger coating. There are no significant differences in surface energy, hot melt, or aqueous glue strength.

The results of the T-Peel testing can be found in Figure 6.

Figure 6. T-Peel Strength on Extrusion Coated Samples - Calendering Experiment.

The observations on the T-Peel strength data are summarized below by each extrusion condition.

25.4 micron LDPE coating; 198 mpm; 17.8 cm air gap, 100 ms in air gap; with primer:
With the lowest time in the air gap, the low peel strength indicates poor adhesion of the LDPE to the coating for all coating samples. No coating pick was observed on the peeled LDPE coating, so the failure mechanism was adhesive failure.

12

25.4 micron LDPE coating; 198 mpm; 17.8 cm air gap, 100 ms in air gap; without primer:
Without primer, the adhesion was poor as well. No coating pick was observed on the peeled LDPE coating, so the failure mechanism was adhesive failure.

25.4 micron LDPE coating; 198 mpm; 25.4 cm air gap, 150 ms in air gap; without primer:
With the longer air gap, resulting in more oxidation of the LDPE, we see improved adhesion and coating cohesive failure as there was coating pulled off. The peel strength of the Control is higher than the heavy calendered sample for this condition.

25.4 micron LDPE coating; 198 mpm; 25.4 cm air gap, 150 ms in air gap; with primer:
With primer, the peel strength is increased significantly for most samples. All samples experienced cohesive coating failure. The Control failure mechanism was a combination of coating cohesive failure and also the 25.4 micron thick LDPE coating broke during testing due to the adhesion bond strength being greater than the LDPE tensile strength. The peel strength of the heavy calendered sample is similar to the Control and has the same failure mechanisms.

25.4 micron LDPE coating; 99 mpm; 25.4 cm air gap, 300 ms in air gap; with primer:
This extrusion condition increases oxidation of the LDPE significantly and will increase adhesion strength. All samples experienced "lock up" during the peel testing.

63.5 micron LDPE coating; 99 mpm; 25.4 cm air gap, 300 ms in air gap; with primer:
In an attempt to obtain meaningful T-peel data, at this condition with the highest LDPE oxidation, the LDPE coating thickness was increased by 2.5 times to 63.5 microns. All samples experienced cohesive coating failure. The heavier calendered sample had higher peel strength than the Control. There is no obvious explanation for this improved performance but it confirms that under the highest oxidation condition and slowest primer drying, coatings could experience improved performance.

After one week of aging, the T-Peel strength was re-tested on these samples. The % change relative to the "off the line" data, reported above, is shown in Table 10. There was no change to the extrusion condition 5 samples – after aging these still had "lock-up" performance. For the samples with 25.4 microns of LDPE, the conditions with primer pre-treatment had the biggest loss in T-peel strength, especially in condition 4. In conditions 4 and 6, the heavy calendered sample had the biggest loss in T-peel strength compared to the Control (standard calendaring).

Table 10. Aged T-Peel Strength Data.

% Change in T-Peel Strength after 1 week	Cond 1 25.4 microns film 198 mpm; 17.8 cm air gap; 100 ms; Primer	Cond 2 25.4 microns film 198 mpm; 17.8 cm air gap; 100 ms; No Primer	Cond 3 25.4 microns film 198 mpm; 25.4 cm air gap; 150 ms; No Primer	Cond 4 25.4 microns film 198 mpm; 25.4 cm air gap; 150 ms; Primer	Cond 5 25.4 microns film 99 mpm; 25.4 cm air gap; 300 ms; Primer	Cond 6 63.5 microns film 99 mpm; 25.4 cm air gap; 300 ms; Primer
Standard Calendering	**-39**	-11	-1	**-35**	n/a	33
Heavy Calendering	**-38**	-17	59	**-57**	n/a	**-12**

Effect of Coat Weight Split

The coated board testing data for the coat weight experiment can be found in Table 11.

13

Table 11. Characterization Data for Coat Weight Experiment.

Coat Weight Variation	PPS Roughness (micron)	Hagerty Porosity (s/10cc)	Surface Energy Total (nN/m)	IGT Dry Pick (cm/s)	Hot Melt Glue (% Fiber Tear)	Aqueous Glue (% Fiber Tear)
SA Level 1 Control	1.56	364	45.1	326	82.5	100
Low Pre-Coat	**2.65**	367	45.1	331	**35.0**	100
High Pre-Coat	1.62	**402**	46.1	**400**	97.5	100
Low Top-Coat	1.73	363	45.4	310	77.5	100
High Top-Coat	1.57	**392**	45.9	344	92.5	100

The greatest change in PPS was with the low pre-coat coat weight, which resulted in an increase of more than 1 micron compared to the Control. With the other coat weight splits, there was no significant difference compared to the Control.

The highest Hagerty porosity (lowest value) was seen with the high pre-coat and the high top-coat samples, both of which are higher in total coat weight. Both of these samples had the highest total coat weight, so this result is not surprising. With the two other coat weight splits, there was no difference compared to the Control.

The coat weight split changes did not significantly affect the surface energy.

Only two conditions gave different IGT Dry Pick results compared to the Control. Significantly higher Dry Pick value was seen with the high pre-coat coat weight sample. Lower Dry Pick was seen with the low top-coat sample.

The hot melt glue fiber tear data shows that with more coat weight there was a slightly higher fiber tear compared to the Control. The lowest fiber tear was seen with the low pre-coat coat weight. In the Aqueous Glue testing, all samples had 100% fiber tear.

The results of the T-Peel testing can be found in Figure 7.

Figure 7. T-Peel Strength on Extrusion Coated Samples – Coat Weight Experiment.
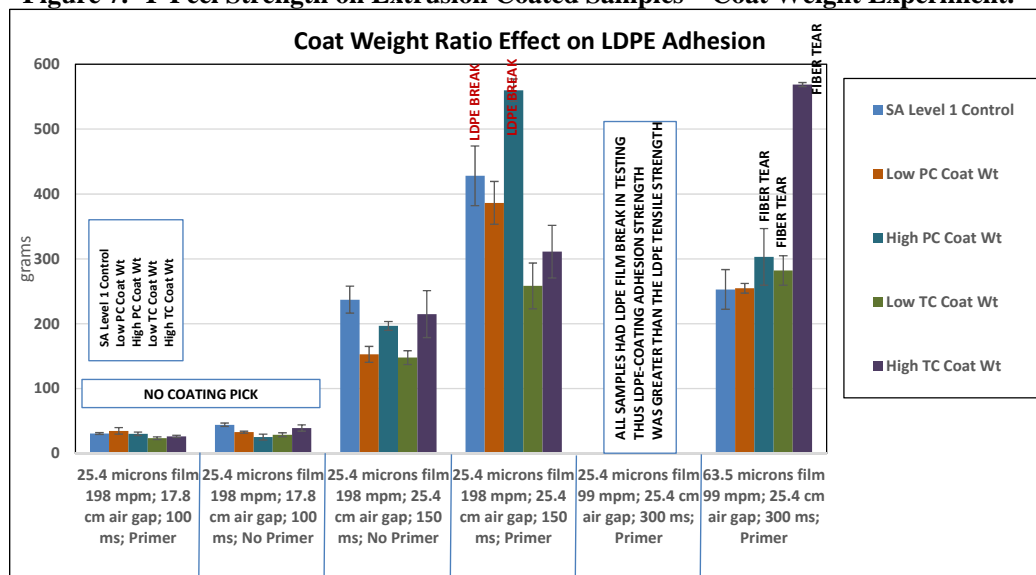
The observations on the T-Peel strength data are summarized below by each extrusion condition.

14

25.4 micron LDPE coating; 198 mpm; 17.8 cm air gap, 100 ms in air gap; with primer:
With the lowest time in the air gap, the low peel strength indicates poor adhesion of the LDPE to the coating for all coating samples. No coating pick was observed on the peeled LDPE coating, so the failure mechanism was adhesive failure.

25.4 micron LDPE coating; 198 mpm; 17.8 cm air gap, 100 ms in air gap; without primer:
Without, the adhesion was poor as well. No coating pick was observed on the peeled LDPE coating, so the failure mechanism was adhesive failure.

25.4 micron LDPE coating; 198 mpm; 25.4 cm air gap, 150 ms in air gap; without primer:
With the longer air gap, resulting in more oxidation of the LDPE, we see improved adhesion and coating cohesive failure as there was coating pulled off. The peel strength of the Control was similar to the samples with the higher coat weight in each layer. The samples with the low pre-coat coat weight and the low top-coat coat weight have the lowest peel strength.

25.4 micron LDPE coating; 198 mpm; 25.4 cm air gap, 150 ms in air gap; with primer:
With primer, the peel strength was increased significantly for most samples. All samples experienced coating cohesive failure. The Control failure mechanism was a combination of cohesive coating failure but also the 25.4 micron thick LDPE coating broke during testing due to the adhesion bond strength being greater than the LDPE tensile strength. Low pre-coat coat weight gave lower peel strength than high pre-coat coat weight. The low top-coat coat weight gave lower peel strength than high top-coat coat weight. The high pre-coat coat weight had similar failure mechanisms as the Control and the peel strength was higher. The Control was higher than the other samples.

25.4 micron LDPE coating; 99 mpm; 25.4 cm air gap, 300 ms in air gap; with primer:
This extrusion condition increases oxidation of the LDPE significantly and will increase adhesion strength. All samples experienced "lock up" during the peel testing.

63.5 micron LDPE coating; 99 mpm; 25.4 cm air gap, 300 ms in air gap; with primer:
In an attempt to obtain meaningful T-peel data, at this condition with the highest LDPE oxidation, the LDPE coating thickness was increased by 2.5 times to 63.5 microns. All samples experienced cohesive coating failure. Low pre-coat coat weight gave lower peel strength than high pre-coat coat weight and low top-coat coat weight gave lower peel strength than high top-coat coat weight. The Control was similar to the low pre-coat coat weight sample, but lower than the other three samples. Those three experienced fiber tear. The highest value was with the high top-coat coat weight sample.

After one week of aging, the T-Peel strength was re-tested on these samples. The % change relative to the "off the line" data is shown in Table 12. There was no change to the extrusion condition 5 samples – after aging these still had "lock-up" performance. For the samples with 25.4 microns of LDPE, the conditions with primer pre-treatment had the biggest loss in T-peel strength, especially in condition 4. There is no clear trend of the % change with the coat weight ratios.

15

Table 12. Aged T-Peel Strength Data.

% Change in T-Peel Strength after 1 week	Cond 1 25.4 microns film 198 mpm; 17.8 cm air gap; 100 ms; Primer	Cond 2 25.4 microns film 198 mpm; 17.8 cm air gap; 100 ms; No Primer	Cond 3 25.4 microns film 198 mpm; 25.4 cm air gap; 150 ms; No Primer	Cond 4 25.4 microns film 198 mpm; 25.4 cm air gap; 150 ms; Primer	Cond 5 25.4 microns film 99 mpm; 25.4 cm air gap; 300 ms; Primer	Cond 6 63.5 microns film 99 mpm; 25.4 cm air gap; 300 ms; Primer
SA Level 1 Control	**-39**	-11	-1	**-35**	n/a	33
Low Pre-Coat	**-32**	24	55	**-59**	n/a	15
High Pre-Coat	10	64	-4	**-18**	n/a	**-45**
Low Top-Coat	**-39**	39	19	**-66**	n/a	**-12**
High Top-Coat	**-27**	-8	10	**-59**	n/a	**-44**

Statistical Analysis of T-Peel Data

Although the experiment was not constructed as a designed experiment, statistical analysis was conducted with the entire T-peel strength data set from extrusion conditions 3, 4 and 6 where differences were observed. These results are summarized by extrusion condition below.

25.4 micron LDPE coating; 198 mpm; 25.4 cm air gap, 150 ms in air gap; without primer:
Regression analysis of T-Peel strength data and the coated board test properties resulted in a significant model with the coefficient of multiple determination (R^2) of 0.82 and a p-value of 0.04. The significant parameters were PPS and Hagerty Porosity where more roughness and higher porosity gave higher peel strength.[7]

Regression analysis of T-Peel strength data and the coating treatment variables resulted in a significant model with the coefficient of multiple determination (R^2) of 0.65 and a p-value of 0.03. The significant parameters were binder type and binder level.

25.4 micron LDPE coating; 198 mpm; 25.4 cm air gap, 150 ms in air gap; with primer:
No correlations could be achieved with either the coated board properties or the coating variables.

63.5 micron LDPE coating; 99 mpm; 25.4 cm air gap, 300 ms in air gap; with primer:
Regression analysis of T-Peel strength data and the coated board properties did not generate any correlations. Regression analysis of T-Peel strength data and the coating variables resulted in a significant model with the coefficient of multiple determination (R^2) of 0.78 and a p-value of 0.02. The significant parameter was binder type, with the SCE system giving the highest peel strength.

LDPE Extrusion at Higher Temperatures

The LDPE extrusion for this study was done at 310 $^{\circ}$C, which is on the lower end of the typical extrusion temperature range, in order to force failure. Selected samples were also evaluated with the LDPE coating thicknesses of 25.4 mm at higher extrusion temperatures, resulting in melt temperatures of 321 and 332 $^{\circ}$C, run at a speed of 198 mpm. It is expected that the higher melt temperatures will result in higher bond strength due to the higher level of oxidation of the LDPE and due to a slower quench time of the LDPE, allowing for greater penetration of the molten LDPE into the substrate.[7] The data is summarized below in Table 13.

For the 25.4 mm thick LDPE coatings, both of the higher melt temperatures resulted in significantly higher adhesion strength of the LDPE to the pigmented coating. Most of the samples experienced "lock-up". This proves that significant pigmented coating formulation

16

and treatment changes could still perform well in extrusion coating applications if the melt temperature is optimized or high enough to generate the appropriate level of oxidation.

Table 13. T-Peel Strength Data on Higher Melt Temperature Samples at 25.4 mm.

	T-Peel Strength (grams) 25.4 microns; 198 mpm; 25.4 cm air gap; 150 milli-sec					
Melt Temp. (^{o}C)	310		321		332	
Primer Pre-Treatment	No Primer	Primer	No Primer	Primer	No Primer	Primer
SA Level 1 Control	237	428	Poly Break	Poly Break	Poly Break	Poly Break
SA w Surfactant	110	439	Poly Break	Poly Break	Poly Break	Poly Break
Low TC Coat Wt	148	258	Poly Break	Poly Break	Poly Break	Poly Break
Low PC Coat Wt	153	386	Poly Break	Poly Break	351	Poly Break
Top-coat B - 50/50	102	235	Poly Break	Poly Break	Poly Break	Poly Break
Top-coat C - 40/60	119	258	672	Poly Break	Poly Break	Poly Break

CONCLUSIONS

In this study, coating composition variables included three binder systems; SA, SB and a novel Starch Containing Emulsion (SCE). Each binder chemistry was evaluated at two different levels in both coating layers.   Three different calcium carbonate/clay splits in the top-coat, the split of the pre-coat/top-coat coat weight, and calendering load were other variables studied.   The conclusions are summarized below:

- At low levels of LDPE oxidation, poor LDPE adhesion was experienced, with no coating pick observed for any conditions.
- Increasing oxidation resulted in improved LDPE adhesion and the failure mechanisms observed included coating cohesive failure and when primer was used fiber tear was observed.
- At the highest oxidation level, we observed LDPE "lock-up" situation, for all samples, where the LDPE-coating bond strength, coating cohesive strength and the coating-fiber adhesion are greater than the LDPE tensile strength. The highest oxidation level overcame all of the formulation and coating changes studied.
- At the highest oxidation level and higher LDPE film thickness, the SCE systems had higher peel strength. This could be due to greater interaction between the PEI primer and the –OH groups of the starch component, resulting in a greater amide interaction and linkage.
- For the SA and SB systems, a reduction in binder level resulted in lower peel strength. For the SCE system, the binder reduction resulted in higher peel strength – this may be due to a combination of the OH groups of the starch and more coating porosity.
- Addition of surfactant to the SA binder resulted in lower peel strength, despite flame pre-treatment of the board, at the 25 micron LDPE coating thickness. At 63.5 microns, the peel strength was higher than the Control.
- Increasing clay concentration in the top-coat resulted in reduced peel strength, at the 25 micron LDPE coating thickness. At 63.5 microns, higher clay concentration had higher peel strength.
- Increased calendering produced a smoother board and had slightly higher peel strength than the Control.
- Low pre-coat coat weight gave lower peel strength than high pre-coat weight and low top-coat coat weight gave lower peel strength than high top-coat coat weight.
- Statistical analysis of extrusion condition 3 showed increasing T-peel strength with increasing roughness and increased coating porosity.
- Re-testing of aged LDPE-coated coated paperboard resulted in reduced T-peel strength, especially in conditions using primer pre-treatment. SCE without primer pre-treatment exhibited an increase in aged T-peel strength.

17

- Higher extrusion melt temperatures significantly improved LDPE adhesion to pigmented coating and resulted in good performance even when significant pigmented coating formulation and treatment changes were made.

ACKNOWLEDGEMENTS

REFERENCES

1. Kellogg, T., "Coated Board", Chapter 3, Section VII from "The Coating Process", prepared by the Coating Process Committee of the Coating and Graphic Arts Division, 1993.

2. Andersson, L.G., "Typical Coating Components", Chapter 2, Section I from "The Coating Process", prepared by the Coating Process Committee of the Coating and Graphic Arts Division, 1993.

3. Frey, K., "An Overview of Paperboard Extrusion Coating & Laminating", proceedings from the TAPPI PaperCon 2015 Conference, Atlanta, Georgia.

4. Hammond, D. R., "Primer Coatings on Paperboard for Extrusion Coating and Heat Sealing Applications", proceedings from the TAPPI PaperCon 2015 Conference, Atlanta, Georgia.

5. Cushing, G., "Chemical Primers As Surface Modifiers For Film Substrates", proceedings from the TAPPI PLACE 2005 Extrusion Coating Short Course.

6. Salminen, P.J., Ahtinen, M.E., Zurcher, K., Baserga, A., "Fundamental Mechanisms for Polyethylene Adhesion on Pigment Coated Paper and Board", proceedings from the 13th European PLACE Conference 2011, Bregenz, Austria.

7. Morris, B.A., "Understanding Why Adhesion in Extrusion Coating Decreases With Diminishing Coating Thickness, Part 1: Penetration of Porous Substrates", proceedings from the TAPPI PLACE 2005 Conference, Las Vegas, Nevada.

APPENDIX A – Pilot Coater Run Data – Formulations

	Base Coat							Top Coat									
FORMULATION	A	B	C	D	E	F	P	G	H	I	J	K	L	M	N	O	Q
PIGMENTS																	
Hydrocarb 60	100	100	100	100	100	100	100										
Hydrocarb 90								60	60	50	40	60	60	60	60	60	60
Hydrafine 90								40	40	50	60	40	40	40	40	40	40
Dispex N40	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
ADDITIVES																	
SA Latex	16	14					16	15	13	16	17					15	
SB Latex			16	14								15	13				
SCE Latex					16	14								15	13		
SA Latex + 0.5% Surfactant																	15
Sterocol FS Thickener	0.3	0.3	0.3	0.3	0.3	0.4	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.4	0.2
Eka RC 5550 glyoxal insolubilizer					0.15	0.13								0.14	0.12		
BEFORE RUN FORMULATION	A	B	C	D	E	F	P	G	H	I	J	K	L	M	N	O	Q
% Solids	66.0	66.0	66.1	66.0	66.0	65.8	65.8	65.1	65.1	65.2	65.1	65.0	65.1	65.0	64.9	44.0	64.8
pH w/NH4OH	8.3	8.5	8.4	8.7	8.3	8.2	8.4	8.6	8.5	8.4	8.4	8.5	8.5	8.2	8.3	8.5	8.5
Viscosity @ 20 rpm, w/#4	2590	3050	3740	3380	2910	2640	2860	3670	3390	3960	3740	4550	3340	3420	3090	10	4320
Viscosity @ 50 rpm, w/#4	1372	1604	1932	1756	1548	1400	1512	1836	1704	1984	1884	2288	1804	1720	1572	32	2204
Viscosity @ 100 rpm, w/#4	864	1012	1204	1102	980	898	958	1116	1030	1198	1122	1386	1110	1044	964	40	1350
Hercules max rpm viscosity	36.9	36.4	37.2	35.2	37.8	39.0	34.6	33.3	30.7	33.7	38.4	50.3	53.8	36.0	32.9	7.3	40.8

APPENDIX B – Pilot Coater Run Data – Base Coat Rigid Blade

RUN NUMBER	1	2	3	4	5	6	7	9	10	11	12	13	14	15	16	17	18	39
ROLL DESIGNATION	90086C	90100C	90090Z	90084A	90088E	90104Z	90114C	90102E	90103F	90116E	90122D	90115D	90117	90113B	90123E	90125Z	90085B	90101D
FORMULATION	A	A	A	A	A	A	A	A	A	A	B	C	C	D	E	E	F	P
MACHINE SETTINGS																		
Blade (B=Bent)	15-45	15-35	15-35	15-35	15-35	15-35	15-35	15-35	15-35	15-35	15-35	15-35	15-35	15-35	15-35	15-35	15-35	15-35
Rod (G=Grooved)																		
Head Angle	49.7	39.7	39.7	39.7	39.7	39.7	39.7	39.7	39.7	39.7	39.7	39.7	39.7	39.7	39.7	39.7	39.7	39.7
Head Stops (in.)																		
Applicator Roll Speed (%)	28	28	28	28	28	28	28	28	28	28	28	28	28	28	28	28	28	28
Applicator Roll Gap (in.)	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+
Coater Speed (FPM)	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150
Coated Side	Print	Print	Print	Print	Print	Print	Print	Print	Print	Print	Print	Print	Print	Print	Print	Print	Print	Print
RUN VARIABLES																		
Basestock Moisture (%)	4.5	5.0	4.1	4.2	4.6	4.3	3.8	4.8	4.6	3.6	4.9	3.9	3.4	3.5	4.3	4.0	4.4	5.2
Coat Weight (lbs./3300 SF)	8.0	8.0	7.9	8.0	8.1	7.9	8.2	8.1	4.9	11.0	8.0	8.1	8.1	8.1	8.2	8.1	8.1	8.1
Moisture (%)	3.6	3.9	3.4	3.7	4.0	3.9	3.3	3.9	3.7	3.6	3.8	3.5	3.2	3.6	3.6	3.8	3.8	3.6
Tube Load (PSI)	6.8	12.4	11.0	9.7	9.7	8.8	9.8	8.3	45.6	1.8	10.6	9.3	7.8	7.8	8.4	7.4	6.7	9.7
DRYER SETTINGS																		
IR Preheat (%)	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
IR Zone 1 (%)	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
TEC 1 Temperature (°F)	400	400	400	400	400	400	400	400	400	400	400	400	400	400	400	400	400	350
Pressure (in. of water)	0.5	0.7	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	1
TEC 2 Temperature (°F)	400	400	400	400	400	400	400	400	400	400	400	400	400	400	400	400	400	350
Pressure (in. of water)	0.5	0.7	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	1
TEC 3 Temperature (°F)	400	400	400	400	400	400	400	400	400	400	400	400	400	400	400	400	400	350
Pressure (in. of water)	0.5	0.7	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	1
TEC 4 Temperature (°F)	400	400	400	400	400	400	400	400	400	400	400	400	400	400	400	400	400	350
Pressure (in. of water)	0.5	0.7	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	1
SHEET TEMPERATURE																		
IR Preheat (°F)	115	117	119	119	118	120	119	117	118	120	115	119	122	122	121	122	121	115
Before TEC 1 (°F)	113	114	116	116	115	116	115	113	115	116	112	116	117	117	116	117	117	112
After TEC 1 (°F)	135	135	136	133	132	133	134	132	138	125	131	134	135	135	135	133	131	134
After TEC 2 (°F)	157	155	158	152	152	152	156	155	159	147	155	153	155	155	156	153	150	156
After TEC 3 (°F)	175	173	174	168	167	168	175	171	173	165	174	170	172	172	172	170	167	174
After TEC 4 (°F)	189	186	189	181	183	182	189	187	187	180	187	183	187	187	187	184	182	187
Reel (°F)	133	133	137	134	130	132	134	132	134	135	130	135	134	136	135	134	135	131

19

APPENDIX C – Pilot Coater Run Data – Top Coat Bent Blade

RUN NUMBER		19	20	21	22	23	24	25	26	29	30	31	32	33	34	35	36	37	40
ROLL DESIGNATION		RUN 7	RUN 1	RUN 5	RUN 4	RUN 2	RUN 2	RUN 11	RUN 10	RUN 12	RUN 6	RUN 9	RUN 14	RUN 13	RUN 15	RUN 16	RUN 17	RUN 18	RUN 39
FORMULATION		G	G	G	G	G	G	G	G	H	I	J	K	K	L	M	M	N	Q
MACHINE SETTINGS																			
Blade	(B=Bent)	15 B	15 B	15 B	15 B	15 B	15 B	15 B	15 B	15 B	15 B	15 B	15 B	15 B	15 B	15 B	15 B	15 B	15 B
Rod	(G=Grooved)																		
Head Angle		29.4	29	29	29	33.2	25	29	29.1	31.9	27.5	29.7	31.7	30.2	30.2	31.1	31.7	34.3	30.6
Head Stops	(in.)																		
Applicator Roll Speed	(%)	28	28	28	28	28	28	28	28	28	28	28	28	28	28	28	28	28	28
Applicator Roll Gap	(in.)	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+	.012+
Coater Speed	(FPM)	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150	1150
Coated Side		Print	Print	Print	Print	Print	Print	Print	Print	Print	Print	Print	Print	Print	Print	Print	Print	Print	Print
RUN VARIABLES																			
Basestock Moisture	(%)	3.2	3.6	3.9	3.6	3.7	4.0	3.6	3.5	3.7	3.7	3.8	3.2	3.5	3.2	3.5	3.5	3.7	3.8
Coat Weight	(lbs./3300 SF)	6.2	6.6	6.6	6.6	3.9	10.8	6.5	6.5	6.8	6.5	6.6	6.5	6.5	6.4	6.7	7.1	6.7	6.8
Moisture	(%)	2.5	3.0	3.1	2.9	2.7	3.4	2.7	2.9	2.9	2.9	3.0	2.5	2.8	2.4	2.8	2.8	2.9	2.7
Tube Load	(PSI)	14.7	16.7	14.8	17.9	12.2	17.4	19.1	19.3	15.2	12.4	13.5	18.0	16.0	13.2	15.1	16.7	16.7	15.5
DRYER SETTINGS																			
IR Preheat	(%)	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
IR Zone 1	(%)	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
TEC 1 Temperature	(°F)	350	350	350	350	350	350	350	350	350	350	350	350	350	350	350	350	350	350
Pressure	(in. of water)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
TEC 2 Temperature	(°F)	350	350	350	350	350	350	350	350	350	350	350	350	350	350	350	350	350	350
Pressure	(in. of water)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
TEC 3 Temperature	(°F)	350	350	350	350	350	350	350	350	350	350	350	350	350	350	350	350	350	350
Pressure	(in. of water)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
TEC 4 Temperature	(°F)	350	350	350	350	350	350	350	350	350	350	350	350	350	350	350	350	350	350
Pressure	(in. of water)	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
SHEET TEMPERATURE																			
IR Preheat	(°F)	140	135	137	139	141	135	136	139	160	148	150	136	137	148	140	142	139	159
Before TEC 1	(°F)	124	122	123	124	125	122	123	124	133	128	129	121	122	125	123	125	123	130
After TEC 1	(°F)	140	133	133	135	151	123	133	134	149	142	144	134	135	143	137	138	136	148
After TEC 2	(°F)	161	153	152	155	167	140	154	155	163	159	158	158	157	164	157	158	154	165
After TEC 3	(°F)	176	167	166	170	179	163	171	171	175	174	173	176	173	180	171	172	169	175
After TEC 4	(°F)	189	181	179	182	190	180	184	184	185	183	183	188	186	191	186	184	182	186
Reel	(°F)	138	134	135	135	139	133	136	136	138	136	139	136	135	136	137	136	135	136

APPENDIX D – Paperboard Testing Data

Top Coat Formulation	Hagerty Permeability (sec per 10cc)	PPS	Sheet Gloss	Surface Energy Total	Surface Energy Polar	Surface Energy Dispersive	Dry Pick - Ink ft per min	Hot Melt Glue (%Tear)	Aqueous Glue (% Fiber Tear)
SA Level 1 Control	364	1.56	47.3	45.1	10.7	34.4	658	83	100
SA Level 2	213	1.24	52.7	44.7	9.8	34.9	650	98	100
SB Level 1	1437	1.30	46.6	53.9	15.2	38.7	736	48	100
SB Level 2	815	1.06	53.2	56.4	17.3	39.1	671	5	100
SCE Level 1	327	1.43	46.8	46.5	11.7	34.9	695	100	100
SCE Level 2	174	1.10	51.2	47.9	12.2	35.7	707	100	100
SA w Surfactant	468	1.05	39.4	42.9	11.6	31.3	685	100	100
Heavy Calendering	650	1.18	60.0	45.9	11.8	34.1	726	90	100
Low TC Coat Wt	363	1.73	44.3	45.4	10.7	34.7	608	78	100
High TC Coat Wt	392	1.57	49.8	45.9	12.0	33.9	675	93	100
High PC Coat Wt	402	1.62	47.4	46.1	12.1	34.1	783	98	100
Low PC Coat Wt	367	2.65	40.6	45.1	10.7	34.4	650	35	100
TC Carb/Clay 50/50	474	1.18	43.3	43.1	9.5	33.6	715	100	100
TC Carb/Clay 40/60	721	1.44	49.1	43.6	8.6	35.0	596	100	100

20

APPENDIX E – Extrusion Coated Board Testing Data

Sample	T-Peel Strength (grams)					
	25.4 microns film 198 mpm; 17.8 cm air gap; 100 ms; Primer	25.4 microns film 198 mpm; 17.8 cm air gap; 100 ms; No Primer	25.4 microns film 198 mpm; 25.4 cm air gap; 150 ms; No Primer	25.4 microns film 198 mpm; 25.4 cm air gap; 150 ms; Primer	25.4 microns film 99 mpm; 25.4 cm air gap; 300 ms; Primer	63.5 microns film 99 mpm; 25.4 cm air gap; 300 ms; Primer
SA Level 1 Control	31	44	237	428	Film break	253
SA Level 2	47	98	283	479	Film break	270
SB Level 1	22	54	269	438	Film break	205
SB Level 2	66	70	139	346	Film break	148
SCE Level 1	30	33	177	150	Film break	720
SCE Level 2	27	51	217	442	Film break	869
SA w Surfactant	15	29	110	439	Film break	593
Heavy Calendering	13	46	147	511	Film break	423
Low TC Coat Wt	23	29	148	258	Film break	282
High TC Coat Wt	26	39	215	311	Film break	569
High PC Coat Wt	30	25	197	560	Film break	303
Low PC Coat Wt	35	33	153	386	Film break	255
TC Carb/Clay 50/50	28	41	102	235	Film break	664
TC Carb/Clay 40/60	22	31	119	258	Film break	521

APPENDIX F – Aged Extrusion Coated Board Testing Data

Sample	T-Peel Strength (grams)					
	25.4 microns film 198 mpm; 17.8 cm air gap; 100 ms; Primer	25.4 microns film 198 mpm; 17.8 cm air gap; 100 ms; No Primer	25.4 microns film 198 mpm; 25.4 cm air gap; 150 ms; No Primer	25.4 microns film 198 mpm; 25.4 cm air gap; 150 ms; Primer	25.4 microns film 99 mpm; 25.4 cm air gap; 300 ms; Primer	63.5 microns film 99 mpm; 25.4 cm air gap; 300 ms; Primer
SA Level 1 Control	19	39	234	279	Film Break	337
SA Level 2	35	111	206	536	Film Break	334
SB Level 1	23	75	221	319	Film Break	178
SB Level 2	32	107	103	319	Film Break	145
SCE Level 1	10	21	240	65	Film Break	939
SCE Level 2	13	55	301	430	Film Break	792
SA w Surfactant	2	20	186	123	Film Break	538
Heavy Calendering	8	38	234	246	Film Break	371
Low TC Coat Wt	14	40	176	88	Film Break	249
High TC Coat Wt	19	36	236	126	Film Break	317
High PC Coat Wt	33	41	189	450	Film Break	168
Low PC Coat Wt	23	41	237	157	Film Break	292
TC Carb/Clay 50/50	24	9	85	63	Film Break	666
TC Carb/Clay 40/60	6	19	132	39	Film Break	319