Optimizing Application Properties of EB Curable Laminating Adhesives for Flexible Packaging

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Introduction

The advantages and performance properties of Electron Beam (EB) curable laminating adhesives have been previously described.\textsuperscript{1-10} The most significant advantage is that full bonding is achieved instantly upon EB irradiation. This allows immediate slitting, shipping, heat sealing, and filling of the package. One challenge with instant bonding is that the adhesive appearance is “frozen” immediately upon cure. This is in contrast to isocyanate/polyol based adhesives where the adhesive can continue to flow-out in the rewind roll as the curing process continues. Of course the risk with isocyanate/polyol adhesives is that the flow out may not meet expectations and that a significant amount of scrap may result. At least with EB curable adhesives the final optical appearance of the laminate can be evaluated immediately upon production.

It is known that the most effective way to improve the optical properties of an adhesive laminated structure is to improve the lay-down of the adhesive on the primary web. Some additional improvements may also be possible by adjustment of the nip conditions. The goal of this study is to investigate the factors that affect the lay-down properties of EB laminating adhesives. The factors studied were: 1) web speed, 2) adhesive flow additives, 3) adhesive temperature, 4) coater configuration, and 5) corona treatment.

Experimental

Four different adhesive compositions were studied; a base adhesive designated 51345 along with three modified versions “A, B, and C”. The properties of these adhesives are summarized in Table 1 below. The liquid surface tensions of the adhesives were measured with a DuNouy ring type tensiometer. The relative foaming properties were evaluated by checking the appearance in the adhesive reservoir on the coater during the run.

<table>
<thead>
<tr>
<th>Product No.</th>
<th>Viscosity (cps @ 25°C)</th>
<th>Liquid Surface Tension (dynes/cm)</th>
<th>Foaming Characteristics</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>51345</td>
<td>600</td>
<td>44.6</td>
<td>Low</td>
<td>Base adhesive</td>
</tr>
<tr>
<td>51345A</td>
<td>600</td>
<td>36.7</td>
<td>Moderate</td>
<td>Surfactant type A</td>
</tr>
<tr>
<td>51345B</td>
<td>600</td>
<td>38.0</td>
<td>High</td>
<td>Surfactant type B</td>
</tr>
<tr>
<td>51345C</td>
<td>250</td>
<td>33.8</td>
<td>Very low</td>
<td>Low surface energy diluent</td>
</tr>
</tbody>
</table>

Unprinted chemically treated PET (Melinex 813) was used. The surface energy of this film without corona treatment was about 38 dynes/cm.

Adhesives were applied on the pilot line at the Faustel Technical Center.\textsuperscript{11} A three roll offset gravure coater was used consisting of a 150 line/inch chrome gravure cylinder with an enclosed doctor blade chamber, a rubber application roll, and a smooth chrome impression roll (see Figure 1). Adhesive was pumped and circulated to the doctor blade chamber. The roll direction and speed of the gravure cylinder and application roll are reported relative to the web direction. For example, .4R/.9F indicates the gravure cylinder is turning in reverse at 40% of web speed while the application roll is turning forward at 90% of web speed.

The adhesive weight was determined by the weight change upon removing the adhesive from a measured sample of PET film. The weight remained relatively constant with all four adhesives. It was also fairly constant at 100 and 500 ft/min. The main factor affecting the adhesive weight was the coater set-up. The average weights are shown in Figure 1.
1F/1F  2.0 lbs/3000 ft² (3.2 g/m²)
0.3R/1F  1.1 lbs/3000 ft² (1.8 g/m²)
0.4R/0.9F  1.3 lbs/3000 ft² (2.1 g/m²)

Figure 1. Offset Gravure Coater Configuration

All four adhesives were run at room temperature.  51345A and 51345C were also tested at 44 ºC and 35 ºC respectively by heating the adhesive reservoir as well as the gravure cylinder.

The adhesives were EB cured at 3.0 Mrads under a nitrogen atmosphere with an Energy Sciences Electocure unit. Coated and cured adhesive samples on the PET film were collected at the rewind station. They were evaluated by microscopic examination and optical light transmission. It was found that the best evaluation came from a visual ranking of the samples. The coated samples were mounted on a glass window and were then sorted and ranked by appearance on a scale of 1 to 5 (1 = highest clarity, 5 = lowest clarity)

Results and Discussion

Figure 2 shows the effect of web speed and adhesive composition on appearance. All of the modified adhesives gave better appearance at 100 fpm compared to 500 fpm. The base adhesive had poor appearance and was not affected by the web speed. This was likely due to the high liquid surface tension of the unmodified adhesive (see Table 1) relative to the surface energy of the film (38 dynes/cm), which prevented flow-out of the adhesive on the film. Overall, the modified adhesives gave improved appearance relative to the base adhesive.

Figure 3 shows the effect of the coater set-up on the appearance. Operating the gravure roll in the reverse direction appears to help the lay down of the adhesive. This is likely due to the wiping action that occurs as adhesive is transferred to the application roll. Some additional improvement seems to also occur when the application roll is run slightly below the speed of the web (.4R/.9F set-up). The 51345C adhesive gave improved appearance under all conditions. This is likely due to the lower viscosity, lower surface energy, and lower foaming of this adhesive (see Table 1).

The results shown in Figure 4 indicate that corona treatment does not improve the appearance of the adhesive. In some cases a slight negative effect was observed.

The results shown in Figure 5 indicate that heating the adhesive does not help the appearance. If appearance is related to adhesive flow-out on the web, heating the adhesive may not be effective due to rapid cooling given the high mass of the web relative to the adhesive.
Figure 2. Effect of Web Speed/Adhesive
no corona treatment

Figure 3. Effect of Coater Set Up/Adhesive
500 fpm, no corona treatment
The four adhesives used in this study were used to prepare laminations in order to check the effect of the adhesive modifications on the bonding properties. Adhesive was applied to 25 micron Aluminum foil with a flexo hand-proofer. Melinex 813 was applied to the wet adhesive followed by curing at 3.0 Mrads at 165 kV. T-peel tests were performed dry and also after soaking for 1 hour in water. The results are shown in Table 2. Film tear of the
PET was achieved in all cases under dry conditions. The maximum strength at tear is reported. The “A” and “B” modifications appeared to have a slight negative effect on the maximum dry bond strength while “C” had improved wet bond strength.

Table 2. Adhesive Bond Performance

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Average Dry Bond</th>
<th>Maximum Dry Bond (g/in)</th>
<th>Average Wet Bond (g/in)</th>
</tr>
</thead>
<tbody>
<tr>
<td>51345</td>
<td>Film Tear</td>
<td>546</td>
<td>234</td>
</tr>
<tr>
<td>51345A</td>
<td>Film Tear</td>
<td>484</td>
<td>209</td>
</tr>
<tr>
<td>51345B</td>
<td>Film Tear</td>
<td>476</td>
<td>213</td>
</tr>
<tr>
<td>51345C</td>
<td>Film Tear</td>
<td>543</td>
<td>260</td>
</tr>
</tbody>
</table>

Conclusions

The main factors that appear to improve the flow out of EB laminating adhesives are modification of the adhesive and the set-up of the coater. These improvements along with other process improvement should make it possible to produce laminates with excellent optical properties while maintaining the benefits of the instant bonding properties of EB laminating technology.

Acknowledgement

We are please to acknowledge Joseph Marotz and Larry Venable of the Faustel Technical Center staff for their assistance in the EB pilot line testing.

References

11. Faustel, W194 N11301 McCormick Dr., Germantown, WI 53022 (www.faustel.com)
Optimizing Application Properties of EB Curable Laminating Adhesives for Flexible Packaging

Presented by:
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Vice President, Technical Director
Northwest Coatings LLC

Outline
I. EB Laminating Background
II. Laminate Appearance Study
III. Conclusions
IV. Future Work

Laminating Technologies for Flexible Packaging

- Extrusion lamination
- Solvent base adhesives
- One-component water base
- Two-component water base
- Two-component solventless adhesives
- UV Curable adhesives
- EB Curable adhesives
Electron Beam Laminating Process

Adhesive Laminating Technology Comparison

<table>
<thead>
<tr>
<th>2-Component Solventless Laminating Adhesives</th>
<th>EB Curable Laminating Adhesives</th>
</tr>
</thead>
<tbody>
<tr>
<td>Requires accurate mixing of two components</td>
<td>One component, no mixing, remains unchanged until EB exposure</td>
</tr>
<tr>
<td>Heated application equipment</td>
<td>Room temperature application</td>
</tr>
<tr>
<td>Typical application weight 1.0 lb/3000 ft² (1.6 g/m²)</td>
<td>Typical application weight 1.0 lb/3000 ft² (1.6 g/m²)</td>
</tr>
<tr>
<td>Sensitive to some ink solvents and chemistry</td>
<td>Relatively insensitive to ink solvents and chemistry</td>
</tr>
</tbody>
</table>

Adhesive Laminating Technology Comparison (continued)

<table>
<thead>
<tr>
<th>2-Component Solventless Laminating Adhesives</th>
<th>EB Curable Laminating Adhesives</th>
</tr>
</thead>
<tbody>
<tr>
<td>Toxicity concerns with residual aromatic amines</td>
<td>No aromatic amines; high conversions minimize uncured material</td>
</tr>
<tr>
<td>Requires several days to cure; delayed QC, slitting, shipping, and filing</td>
<td>Instant cure; immediate QC, slitting, shipping, and filing</td>
</tr>
<tr>
<td>Multilayer structures require multiple lamination steps</td>
<td>In-line multilayer lamination possible</td>
</tr>
<tr>
<td>Laminate appearance may continue to improve in the roll</td>
<td>Laminate appearance is “frozen” immediately upon cure</td>
</tr>
</tbody>
</table>
Commercial Status of EB Laminating Technology

- Fully commercial with at least 7 different converters in 8 locations
- Largest application – film to paperboard lamination
- Established commercial non-food flexible packaging applications
- Commercial food-packaging application with recognized barrier materials
- Line speeds running up to 750 ft/min with acceptable laminate appearance

Keys to Broad Commercial Acceptance of EB Laminating Technology

- Meet required bond performance with suitable substrates
- Compatibility with available ink systems
- Suitable for food packaging – FDA compliance, no taint or odor
- Applied cost competitive with other adhesive technologies
- Consistent good quality laminate appearance
- No adverse effects of EB on substrates

EB Curable Adhesive Properties

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Viscosity (cps @ 25 ºC)</th>
<th>Relative Hydrophobicity</th>
<th>Cured Tg (ºC)</th>
</tr>
</thead>
<tbody>
<tr>
<td>51325</td>
<td>355</td>
<td>1 least hydrophobic</td>
<td>21.5</td>
</tr>
<tr>
<td>51335</td>
<td>473</td>
<td>2</td>
<td>22.4</td>
</tr>
<tr>
<td>51345</td>
<td>593</td>
<td>3</td>
<td>27.6</td>
</tr>
<tr>
<td>51355</td>
<td>852</td>
<td>4 most hydrophobic</td>
<td>29.2</td>
</tr>
</tbody>
</table>
**Bond Performance Summary**

- Excellent film destruct bonding properties achieved with multiple substrates
- The laminates exhibit good water and food product resistance
- The properties of the cured adhesive affects the maximum bond strength at tear
- The optimum adhesive composition depends on the substrates that are used
Optimizing EB Adhesive Laminate Appearance

• Challenge
  – The appearance of two-component solventless adhesive laminations can improve as the adhesive continues to flow out in the roll
  – The appearance of EB adhesive laminations are "frozen" immediately upon cure
• Approach – Conduct study to optimize EB laminate appearance

EB Adhesive Lay-Down and Appearance Study

• Study Conditions
  – Faustel Technical Center pilot line
  – Optimize adhesive lay-down (more critical than nip conditions)
  – Offset gravure application (preferred over direct gravure and multi-roll coating application)
  – Coat and EB cure adhesives on PET without lamination
  – Rate the optical appearance of the applied adhesive:
    • 1 = best, 5 = worst
• Factors Studied
  – Gravure and application roll direction and speed
  – Web speed
  – Corona treatment
  – Adhesive temperature
  – Adhesive flow additives (modifications A, B, C)

Faustel Technical Center Pilot Line
EB Coating Web Path
Faustel Technical Center Pilot Line

EB Laminating Web Path
Faustel Technical Center Pilot Line

EB Laminating Adhesive Variables

<table>
<thead>
<tr>
<th>Adhesive</th>
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### Offset Gravure Application

**Web Speed = 1.0**

<table>
<thead>
<tr>
<th>Gravure</th>
<th>Application</th>
<th>Adhesive Wt. (lbs/3000 ft²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 F</td>
<td>1 F</td>
<td>2.0</td>
</tr>
<tr>
<td>0.3 R</td>
<td>1 F</td>
<td>1.1</td>
</tr>
<tr>
<td>0.4 R</td>
<td>0.9 F</td>
<td>1.3</td>
</tr>
</tbody>
</table>

### Effect of Web Speed/Adhesive

*(room temperature, no corona)*

- Appearance
- Adhesive
- 1F/1F
- 3R/1F
- 4R/3F

### Effect of Coater Set Up and Adhesive Modifications

*(500 fpm, no corona treatment)*

- Appearance
- Adhesive
- 51345
- 51345A
- 51345B
- 51345C
Effect of Corona Treatment
(500 ft/min, .3R/1F)

Effect of Adhesive Temperature
(no corona treatment)

Conclusions

- Web speed, coater set up, and adhesive modifications have the largest impact on adhesive appearance
- Corona treatment and adhesive temperature have little effect on adhesive appearance
- Excellent high speed EB adhesive laminate appearance may be expected under optimized conditions
- Optimum application conditions for EB adhesives will likely be different than solvent based, water based, or solventless adhesives
**Future Work:**

*Keys to Broad Commercial Acceptance of EB Laminating Technology*

- Meet required bond performance with suitable substrates
- Compatibility with available ink systems
- Suitable for food packaging – FDA compliance, no taint or odor
- Applied cost competitive with other adhesive technologies
- Consistent good quality laminate appearance
- No adverse effects of EB on substrates

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**Ink Bond Performance**

- Nearly all flexible packaging applications involve bonding to printing inks
- It is desirable for EB adhesives to perform with current commercial laminating inks
- Challenge
  - Two-component solventless adhesive extended cure time may allow ink/adhesive interaction which improves bond strength
  - Instant cure of EB adhesives may limit ink/adhesive interaction
- Status
  - Printed films are being successfully EB laminated at up to 750 ft/min with commercial solvent and water based inks
  - Successful trials have been run with polyurethane, polyamide, PVB, and acrylic ink systems
- Future Work
  - Systematic high speed EB lamination study is in progress with multiple known commercial ink systems

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**Four Ways To Establish FDA Compliance**

1. No migration/no food additive position – below 50 ppb detection limit for most materials and applications – Current EB food applications
2. Threshold of Regulation Listing – requires non-toxic/non-carcinogen materials with dietary intake less than 0.5 ppb
3. Use materials cleared under existing regulations (Prior Sanction letter, GRAS position, prior Food Additive Petition) – few UV/EB materials have appropriate FDA status
4. Register new materials with FDA through a Food Additive Petition or Food Contact Notification (FCN) application – in progress
EB Laminating Adhesive Migration
Initial Results
(single side test cell, 40ºC/10 days)

<table>
<thead>
<tr>
<th>Material</th>
<th>10% Ethanol A Migration (ppb)</th>
<th>95% Ethanol B Migration (ppb)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 mil LLDPE</td>
<td>ND</td>
<td>178/155 C</td>
</tr>
<tr>
<td>0.75 mil oPP</td>
<td>ND</td>
<td>ND</td>
</tr>
</tbody>
</table>

A – Aqueous and alcoholic food simulant
B – Fatty food simulant
C – Two adhesive components
ND – Non-detect, ~20 ppb detection limit

Thank You

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Please remember to turn in your evaluation sheet...