The hottest trends in electron beam curable laminating adhesives for flexible packaging.

Technology Flexes Its Muscles

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RECENTLY, there has been significant interest in electron beam (EB)-curable laminating adhesives for flexible packaging [1]. EB curing offers a number of potential advantages over solvent-based, water-based and solventless adhesive systems. These advantages include:

- full bond strength achieved immediately upon cure;
- one-component systems;
- adhesive remains unchanged until cured;
- no viscosity adjustments;
- shelf-life of at least six months;
- 100 percent reactive systems offer near zero VOC's;
- potential for application at room temperature;
- easy clean-up;
- potential for very low misting;
- compatible with multiple ink systems including water-base, solvent-base and UV/EB curable inks;
- no isocyanates;
- and no aromatic amines.

The interest in EB-curable laminating adhesives has been driven in part by the availability of new EB-curing equipment. This equipment requires a much lower capital investment compared to the previous generation of equipment. It also offers lower voltage, which allows penetration of outer films while limiting energy applied to the inner heat-seal surface. This is important for preventing EB-induced degradation of heat seal properties of certain films [2].

Although EB laminating of flexible packing materials is a new technology, EB curable inks and topcoats for other types of packaging have been in commercial use more than 20 years [3]. In addition, UV laminating has been well established for more than 10 years. The primary use of UV adhesives has been the in-line lamination of clear films to printed labels [4]. EB laminating offers several advantages over UV laminating for flexible packaging:

- EB will penetrate printed and opaque films, while UV is...
limited to clear films, which allow light penetration;
• EB will typically produce higher conversion compared to UV;
• and most EB materials cure without photoinitiators.
These advantages are important in order to achieve low odor and low migration to allow use in food packaging applications.
One of the challenges associated with any new adhesive technology is the ability to achieve adequate bonding to the wide variety of substrates used in the flexible packaging industry. It is often very desirable to have a single adhesive that will bond to multiple substrates used within a manufacturing facility. A goal of this study was to optimize the adhesive compositions for specific packaging film structures. A further goal was to identify adhesive compositions that are suitable for bonding to multiple substrates.

Experimental
A design of experiments (DOE) was used to examine the effect of the adhesive components. Design-Expert 6.0 (Stat-Ease) software was used for the design and the analysis. A D-Optimal design was selected which chooses a subset of runs from a rich candidate set. The adhesive components studied included a base formulation (A) as well as polymer (B), monomer (C) and additive (D). The resulting design included 13 different adhesive formulations along with two replicates. Films common to the packing industry were used. These include oriented polypropylene (oPP), metalized polypropylene, polyester (PET) and linear low-density polypropylene (LLDPE). The following laminate structures that are commonly used in flexible packaging were prepared:
• oPP/oPP (Mobil 90BSR1/90BSR1)
• Modified oPP/modified oPP (Mobil 90SPW/90SPW)
• oPP/metalized oPP (Mobil 90BRS1/Mobil 70MET)
• PET/LLDPE (DuPont 48LBT2/200Huntsman)
• Modified PET/LLDPE (Melinex 813/200Huntsman)
• PET/heat seal PET (DuPont 48LBT2/DuPont 500LT).
The films were pretreated (corona or flame) by the manufacturers. The heat seal PET (DuPont OLT) was the only film that gave improved bonding upon inline corona treatment.
The other films were used as received. The 15 adhesives and six films resulted in a total of 80 different test conditions. The adhesives were tested in random order.
The adhesives were applied using an offset gravure coater at room temperature. Differential roll speeds were adjusted to maintain a coat weight of 1.95 + 0.32 g/m2 (1.2 + 0.2 pounds/3000 ft.2). The top film was nipped to the wet adhesive immediately after application of the adhesive on the base film. An EB dose of 3 M rads was used. This dosage was based on screening studies, which showed good curing for the range of formulations in this design. The EB voltage was maintained at 165 kV.
Slip-sheets were used to facilitate the preparation of samples for T-peel testing. Test samples were prepared by cutting 25 mm wide strips in the machine direction. Initial testing was conducted within 48 hours of lamination. The T-peel testing was repeated after three weeks to determine the effect of aging on the bond strength. Three replicate peel tests were run for each adhesive/film combination. The average and maximum peel force was recorded.

Results and Discussion
The DOE software was used to analyze the T-peel data. The software selected either a linear or quadratic model to provide the best fit. With film structures 2 and 5, film tear occurred with nearly all of the adhesive combinations. This prevented a detailed analysis of the effects of the adhesive components.

The modified oPP film (Mobil SPW) is designed with a high-energy surface layer to enhance bonding [5]. The modified PET (DuPont Melinex 813) has an acrylic chemical treatment. The destruction of the film upon peel testing shows the superior bonding characteristics of these films.
A typical contour plot is shown in Figure 1. This plot examines the effect of three adhesive components on the bond strength of film structure 1 (oPP/oPP). The base formula (A) is kept at a constant level for this plot. Other families of plots may be generated to examine the effect of all adhesive components anywhere within the design.

The most significant result seen in Figure 1 was the decrease in bond strength with increasing levels of polymer component B. The bond strength was relatively insensitive to the amount of monomer (C) and additive (D) present in the formulation. Figure 2 shows the effect of three weeks of aging on the oPP/oPP bond strength. By comparing Figure 1 to Figure 2, it can be seen that the additive (D) has a significant effect on bond strength. No additional curing of the adhesive is expected after the initial EB cure. The increase in bond strength is likely due to the interaction with the film surface or a physical relaxation of the cured adhesive [6].

Figures 3 and 4 show the effect of the adhesive components on the oPP/met oPP and PET/LLDPE structures. The contour plots for these films show the positive effect of the additive (D) and the negative effect of the polymer (B). Very little change was observed in the bonding properties of these films after three weeks of aging.

Figure 5 shows the effect of the adhesive components on the PET/heat seal PET structures. This plot has a much different appearance compared to Figures 1 to 4. Here, the polymer component (B) has a positive rather than a negative effect on the bonding properties of these films. These films were also insensitive to the presence of additive (D).

The DOE analysis allows a search of the design space for the best adhesive compositions. The highest average and maximum peel strengths were identified for each film structure.

The resulting predicted bond strengths are shown in Table 1. The DOE was also optimized simultaneously to give the best adhesive compositions for all of the film structures. The composition with the best overall result was designated 17900LA. The predicted properties of 17900LA are shown in Table 1.

The results predict that 17900LA will give bonding performance nearly equal to the individually optimized formulas for film structures 1, 3, and 4. 17900LA is not desirable for structure 6 (PET/heat seal PET) where the individual specialized composition gave much stronger bonds. The actual 17900LA composition was produced and has given excellent results in a number of adhesive trials with commercial flexible packaging structures. Typical properties of 17900LA are shown in Table 2. 17900LA has very desirable low viscosity and low odor properties.

### Table 1: DOE Predicted Optimized Bond Strengths

<table>
<thead>
<tr>
<th>Film Structure</th>
<th>Predicted Avg. Bond (g/25 mm)</th>
<th>Predicted Max Bond (g/25 mm)</th>
<th>17900 LA Predicted Avg (g/25 mm)</th>
<th>179000 LA Predicted Max (g/25 mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>OPP/oPP</td>
<td>231</td>
<td>315</td>
<td>198</td>
<td>327</td>
</tr>
<tr>
<td>OPP/met oPP</td>
<td>249</td>
<td>277</td>
<td>249</td>
<td>277</td>
</tr>
<tr>
<td>PET/LLDPE</td>
<td>236</td>
<td>273</td>
<td>228</td>
<td>226</td>
</tr>
<tr>
<td>PET/heat seal PET</td>
<td>168</td>
<td>240</td>
<td>64</td>
<td>102</td>
</tr>
</tbody>
</table>

### Table 2: 17900 LA Adhesive Properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Viscosity</td>
<td>250 cps @ 25˚</td>
</tr>
<tr>
<td>Appearance</td>
<td>clear, light red</td>
</tr>
<tr>
<td>Liquid odor</td>
<td>mild</td>
</tr>
<tr>
<td>Application method(s)</td>
<td>offset gravure or roll</td>
</tr>
<tr>
<td>Recommended application weight</td>
<td>1.3 — 2.4 g/m² (0.8 — 1.5 lbs/3,000 ft²)</td>
</tr>
<tr>
<td>Recommended cure</td>
<td>3. Mrads</td>
</tr>
<tr>
<td>Cured odor</td>
<td>very low</td>
</tr>
</tbody>
</table>

### Conclusion

DOE techniques were very useful for the development of EB curable laminating adhesives. Contour plots and trend analysis were used to elucidate the effect of each adhesive component on bond strengths. Optimized compositions were identified for individual film structures. Simultaneous optimization was also used to identify the best overall adhesive for a range of film structures. The resulting adhesive appears to have properties that are attractive for many flexible packaging applications.

### References

1. a) A. Mykytiuk, Flexible Packaging, February 2000, p. 20.
   b) S. Friedman, packagePRINTING, October 2000, p. 42.
   c) A. Mykytiuk, Flexible Packaging, August 2000, p. 16.

2. I. Rangwalla and E. Maguire, RadTech Report, May/June 2000, p. 27.


### Acknowledgment

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