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UV Curable Laminating Adhesives

By

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ABSTRACT

The popularity of UV curable laminating adhesives has rapidly been increasing. Therefore, a better understanding of the cure mechanism based on photoinitiator and lamps was investigated for two new oligomers for laminating adhesives. UV curable laminating adhesive formulations were prepared using a series of designed experiments with various substrates, including PE, PET, OPP, metallized PET, paper and foil. Clear film laminations were studied based on a series of photoinitiators and lamps for UV cure. The film was corona treated and failure was determined as the point at which the film could be separated and not torn.

INTRODUCTION

UV curable laminating adhesives are currently being used to bond clear films to various films, papers, and foil substrates. Typically, the adhesive is applied to the clear film and then nipped onto the second substrate surface. The adhesive is then cured using UV irradiation through the clear film. Similar technology is also being used with EB curable adhesives (does not contain the photoinitiator) to laminate together opaque films, metallized films, paper, foil and, clear films.

Since most of the work has been based on polypropylene (OPP and BOPP) and polyester (PET), this study was done using polyester (PET). It tends to be more difficult to cure through, since the PET absorbs UV light below 340 nm. The PET used was 0.5 mil film, and film tearing was used to determine sufficient cure. All films were corona treated prior to lamination.

DISCUSSION AND RESULTS

Two new low viscosity oligomers for laminating adhesives were used for this study (Table 1). The formulations contain either CN135 or CN137. These are low viscosity oligomers that help promote adhesion for laminating adhesives. CN135 is a lower functionality oligomer, whereas CN137 has a higher cross-link density. CN966H90 and CN962 are aliphatic urethane diacrylate oligomers, which improve elongation, flexibility, and adhesion. CN112C60 is an epoxy novolak acrylate, which has high temperature resistance. CN301 is a polybutadiene dimethacrylate, which has great flexibility and improves the SAFT (Shear Adhesion Failure Temperature) of most adhesives[1].

Table 1

Laminating Adhesive Formulations

Ingredients	A	B
CN135	95.7	-
CN137	-	46.5
CN966H90	4.3	3.9
CN962	-	25.0
CN112C60	-	11.6
CN301	-	10.0
Irganox1076	-	3.0

For the designed experiment, each of the new low viscosity oligomers was tested using five different photoinitiators at three different concentrations. Each of these oligomers was cured using three different light sources. Then the maximum line speed to fully cure the adhesive was found for each. This was done by determining the point at which film separation could be achieved. The photoinitiators are not designed to be effective over each of the bulbs tested. This test can show the effectiveness of certain photoinitiators in the laminating adhesive on PET.

Photoinitiators in the Study

Five different photoinitiators were used in this study to understand the importance of photoinitiator and light source. The photoinitiators chosen were Irgacure 369, Irgacure 819, Darocure 4265, Esacure KTO46, and Esacure KIP150.

Irgacure 369 is an aminoalkylphenone. The maximum absorption is at 325-335 nm. This photoinitiator will work with most of the bulbs tested, but it will work better with those in the desired output range.

Irgacure 819 is a more recent photoinitiator, which uses the BAPO (bis acyl phosphine oxide) technology. It is Bis (2,4,6-trimethylbenzoyl)phenylphospineoxide. Its maximum absorption occurs at 360-390 nm. This area of absorption is strong for both the H-bulb and the D-bulb and right on the edge of the strong portion of the V-bulb.

Darocure 4265 is another BAPO blend, which is 50% TMPO (acyl phosphine oxide) and 50% Darocure 1173 (2-hydroxy-2-methyl-1-phenyl-1-propanone). This photoinitiator has absorption maxima of 270-290 nm and 365-380 nm, which result from the two photoinitiators.

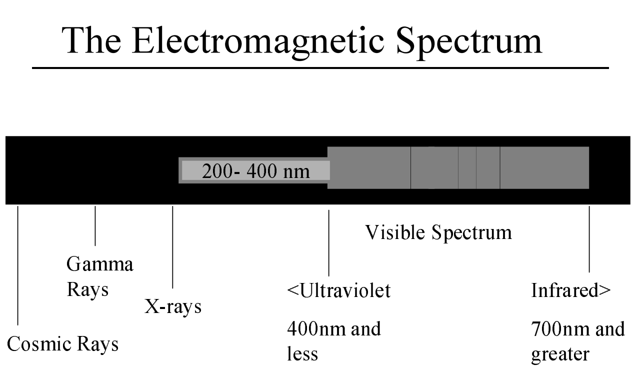
Esacure KTO46 is a liquid blend of 2,4,6-trimethyl benzoyldiphenyl phosphine oxide, oligo 2-hydroxy-methyl-1-[4-(1-methylvinyl)phenyl]propanone and methylbenzophenone derivatives. Its maximum absorption occurs at 245, 363-378, and 400 nm, because of the blend of the three photoinitiators.

Esacure KIP150 is oligo [2-hydroxy-2-methyl-1-[4-(1-methylvinyl)phenyl]propanone]. It has a maximum absorption at 240-320 nm. This type of photoinitiator is typically used with a H-bulb type cure system.

Light Sources in the Study

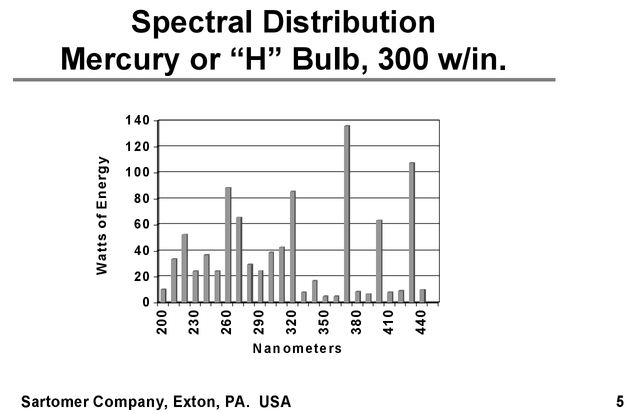
Three different light sources were used to test the adhesive systems - H-bulb, V-bulb and D-bulb. Various bulbs emit different wavelengths of light, and therefore, affect the photoinitiators differently. The ultraviolet wavelength is 200-400 nm, as shown in the electromagnetic spectrum in Figure 1.

Figure 1



The most common system used for curing in the UV industry consists of a mercury lamp or H-Bulb on a Fusion UV processor. The spectra emitted from the H-bulb is shown in Figure 2.

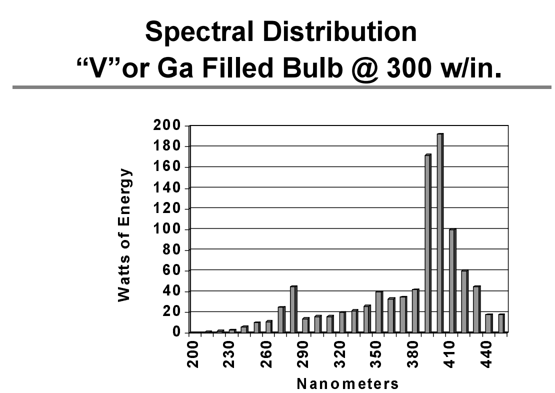
Figure 2



As you can see, the bulk of the spectral output is in the 200-320 nm range.

Another common bulb is the gallium filled bulb or V-bulb. The spectral output of this bulb is shown in Figure 3 below.

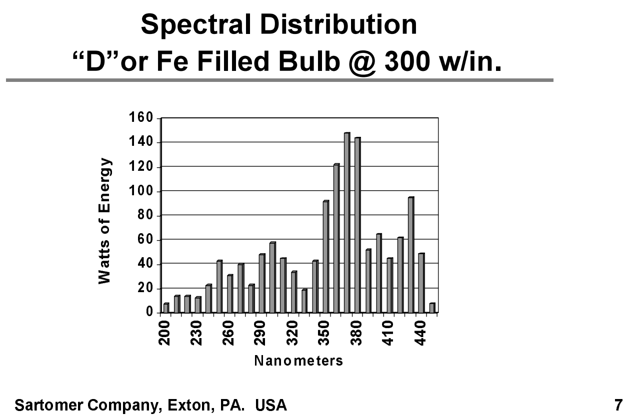
Figure 3



The spectral output is quite different than the H-bulb. Where the H-bulb is near the lower end of the spectrum, the V-bulb is near the upper end of the spectrum (390-420 nm.). It is very intense over a small spectral output. As such, most of the photoinitiators used would respond better with this bulb.

Another common bulb is the iron filled bulb or D-bulb. The spectral output of the D-bulb is shown in Figure 4, below.

Figure 4

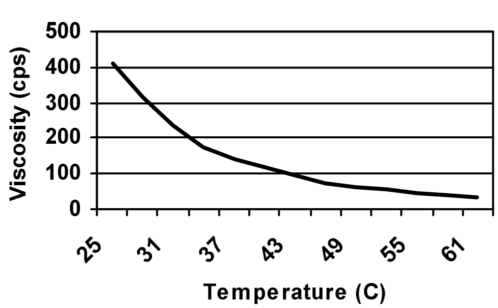


The D-bulb has a much wider range of spectral output, 350-440 nm, and also produces a very intense light. Even at the lower end of the spectrum, a fairly intense light is observed and yields an output suitable for mercury lamp photoinitiators. Therefore, the D-bulb should yield curing for most types of photoinitiators.

Standard Laminating Adhesive Formulation (A)

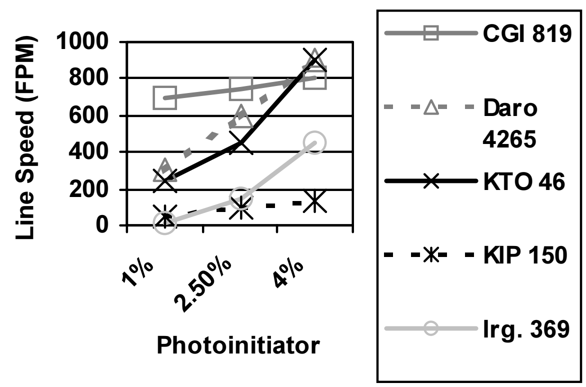
Formula A, shown in Table 1, is considered to be a starting point laminating adhesive formulation. A masterbatch was prepared. The formulation was cured using 5% Esacure KIP150 and a mercury vapor lamp at 25 FPM. The resultant 5 mil cured film yielded an elongation of 87%, a tensile stress of 400 psi, and a Tg via DSC of 8.9 °C. A temperature versus viscosity profile was run on the masterbatch. The results are shown in Figure 5. As shown in Figure 5, the room temperature viscosity of Formula A is fairly low (about 400 cps).

Figure 5
Formula A



Fifteen different photoinitiator blends were mixed to test the five different photoinitiators (Irgacure 369, Irgacure 819, Darocure 4265, Esacure KTO46, and Esacure KIP150) at three different levels: 1%, 2.5%, and 4%. The maximum line speed supporting full cure was then determined. This line speed was recorded as shown in Figure 6 below. Figure 6 data was based on a 600 watt/inch H-bulb in a Fusion UV Processor.

Figure 6
Formula A: H-Bulb, 600 watt/inch



As Figure 6 shows, for low photoinitiator loading levels, Irgacure 819 gave the best results with a 600 watt/inch H bulb. The maximum line speed observed in this testing was 900 FPM. Darocure 4265 and Esacure KTO46 yielded moderate performance, whereas Irgacure 369 and Esacure KIP150 yielded the poorest results for a 600 watt/ inch H bulb cure.

The same fifteen blends were cured using a 600 watt/inch V-bulb in a Fusion UV Processor. These results are shown in Figure 7.

Figure 7
Formula A: V-Bulb, 600 watt/inch

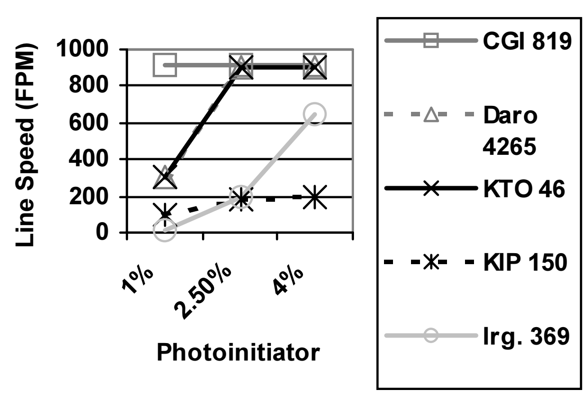
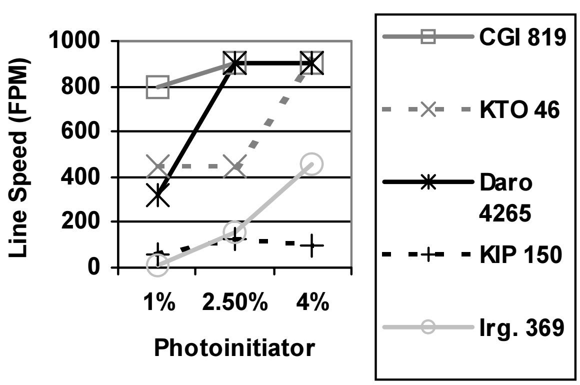


Figure 7 suggests that Irgacure 819 is designed more for the V-bulb. As little as 1% of Irgacure 819 yielded the maximum of over 900 FPM line speed with a full cure (inability to separate the films without tearing). Both Darocure 4265 and Esacure KTO46 gave failures at 300 FPM for the 1% loading of the photoinitiator, but maximized at 900 FPM for both the 2.5% and 4% loading levels. Again, Esacure KIP150 and Irgacure 369 yielded the poorest results.

Finally, the fifteen photoinitiator blends were tested using a 300 watt/inch D-Bulb on a Fusion UV Processor. The results are shown in Figure 8 below.

Figure 8
Formula A: D-Bulb, 300 watt/inch



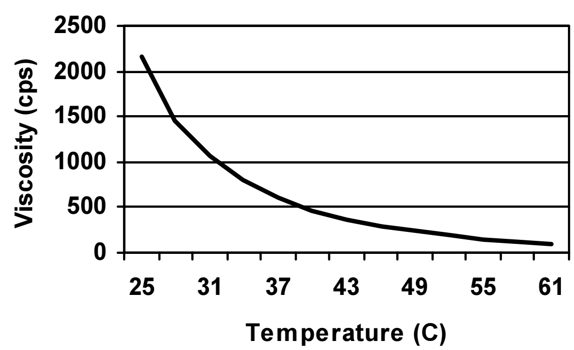
Again, the Irgacure 819 yielded the highest results with very little photoinitiator. Note that the lamp was a 300 watt/inch, not a 600 watt/inch as used for both the H-bulb and the V-bulb. Esacure KTO46 and Darocure 4265 again produced moderate results. As before, Irgacure 369 and Esacure KIP150 gave the poorest results in terms of line speed.

Under all three lamps, Irgacure 819 yielded the best results with Formula A. Both Esacure KTO46 and Darocure 4265 gave the next best results. The best overall results were based on 1% Irgacure 819 using a 600 watt/inch V-bulb.

High Temperature Heat Resistant Laminating Adhesive Formulation B

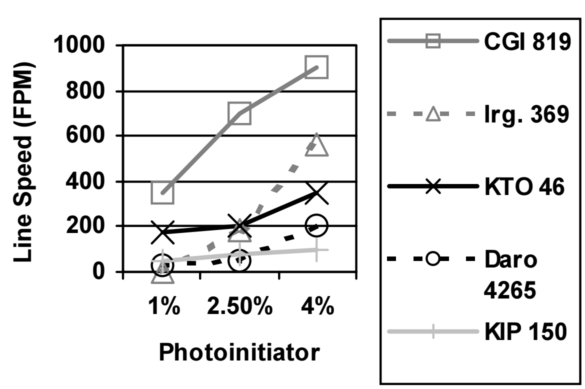
Formula B was developed as a starting point for a high temperature resistant laminating adhesive. The formulation was prepared in the same manner as Formula A. The adhesive was again cured using 5% Esacure KIP150 and a mercury vapor lamp at 25 FPM. The resultant 5 mil cured film yielded an elongation of 52%, a tensile stress of 225 psi, and a Tg via DSC of 9.1 oC. The temperature versus viscosity profile is shown in Figure 9 below. This adhesive had a higher viscosity than Formula A, but minimal heat reduced the viscosity greatly.

Figure 9
Formula B



As previous, a series of photoinitiator blends was prepared and tested under various lamps. Figure 10 shows the results for the H-Bulb.

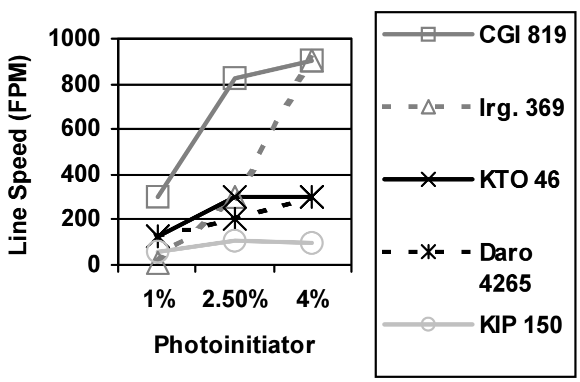
Figure 10
Formula B: H-Bulb, 600 watt/inch



As illustrated, Irgacure 819 is superior to the other four photoinitiators. The only mid-point photoinitiator that works at the one percent loading is Esacure KTO46. Irgacure 369 performs better than Esacure KTO46 at loadings above 2.5%. Both Darocure 4265 and Esacure KIP150 perform poorly in this system as well.

The same fifteen photoinitiator blends were tested using the 600 watt/inch V-Bulb. The results are shown in Figure 11.

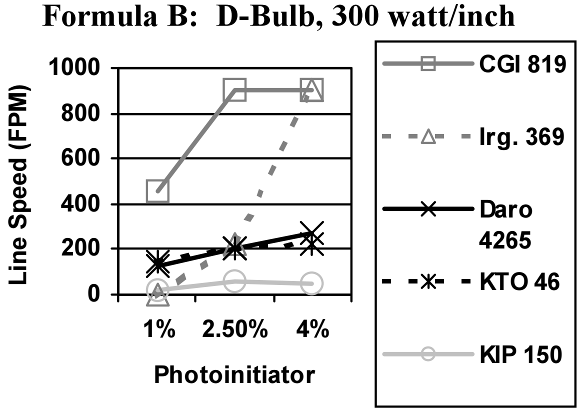
Figure 11
Formula B: V-Bulb, 600 watt/inch



Irgacure 819 again cures the best in the series, but the result for the one percent loading is slightly worse than that for the H-bulb. Irgacure 369 was interesting in that no cure was achieved at one percent in the adhesive. However, the four percent loading resulted in a line speed of 900 feet per minute. The other three photoinitiators performed poorly.

Finally, the fifteen photoinitiator blends were tested using a 300 watt per inch D-Bulb. The results are shown in Figure 12.

Figure 12
Formula B: D-Bulb, 300 watt/inch



Again Irgacure 819 yielded the best results under all lamp conditions. Four percent of Irgacure 369 produced a line speed of 900 feet per minute, whereas very little cure was observed at one percent. The other three photoinitiators performed poorly by comparison.

CONCLUSIONS

In general, we have found that CN135 and CN137 outperform most oligomers developed for laminating adhesives.

When using CN135 in a typical laminating adhesive formulation, it appears that Irgacure 819 is the best overall photoinitiator. Even at low levels - one percent photoinitiator - this photoinitiator is nearly twice as good as the other photoinitiators tested.

When using CN137 in a heat resistant laminating adhesive formulation, Irgacure 819 is again the best photoinitiator. One percent of Irgacure 819 produced nearly twice the line speed of the other four photoinitiators at equal addition levels.

REFERENCES

1. "Examination of Several UV Cure Pressure Sensitive Adhesives for Physical Properties", Project 6782, Daniel Pogany, Adhesive Consultants Inc., Akron, OH.