Characterizing the Efficiency of UV LED Curing

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Abstract

This paper examines the effects of some key operating parameters of UV sources on the curing of various UV inks, coatings and adhesives. The effects of peak UV wavelength and peak intensity are quantitatively evaluated using FT-IR. The data are helpful in guiding both formulators, raw material suppliers and end users to better understand the impact of the UV sources, especially UV LED sources, on the speed and degree of cure for various systems.

Introduction

The objective of the testing for this paper was to gain a better understanding of the effect of delivery of dose (peak intensity) and of wavelength on cure rates especially as these relate to UV LED sources. The data reported suggests that for the range of UV materials selected that the cure rate reaction dynamics are similar whether curing with a traditional mercury vapor lamp with a broad UV spectral emission or a narrow spectral emission UV source with UV emissions at 365nm (345 to 385nm) or 395nm (380 to 420nm).

Equipment and Materials

For this paper, materials that were formulated or were known to work in the UV-A region were chosen. Practically all of these materials cure with UV-A wavelength UV LED sources but will also typically cure well with a traditional mercury vapor lamp system with sufficient UV-A energy (typically centered at 365nm).Of course, not all UV curable materials will cure in this region, but photoinitators have a broad absorption range and even if a material is marketed as being optimized for a specific lower wavelength peak, it doesn't mean the material won't cure with other wavelengths.

The UV curable materials selected were:

Inks:	Piezo inkjet ink from vendor 1 formulated for UV-A
	Piezo inkjet ink from vendor 2 formulated for UV-A
Adhesives:	General purpose adhesive typically used in medical applications
	Custom formulated adhesive for speaker application
Coating:	Custom formulated top coat for wood panels

UV Sources Used

For this paper, cure rates were evaluated using three different light sources two of which were UV LED sources and one traditional mercury vapor lamp. The table below showing the relative intensity measurements for the UV sources which were measured in the same way at the output of the UV emitting window using an EIT Power Puck radiometer.

Table 1: Measured Intensity – UV Light sources

UV Source	Measured Intensity* (W/cm^2)	Manufacturer/Model
100W Lamp with 6mm Light Guide	UV-B 0.898 UV-A 4.268 UV-V 1.805	Lesco Super Spot MKII
395nm LED (380-420nm)	2	Phoseon Technology RX FireFlex
	4	
	8	
365nm LED (345-385nm)	2	Phoseon Technology StarFire MAX

*Intensity measurements were made using the EIT Power Puck.

Due to the constraints of the FTIR experimental setup (see Figure 1), the light source had to be positioned at an angle above the IR beam and was positioned approximately 50mm from the material to be cured. Therefore, the peak intensity for the UV LED sources at this distance was reduced by approximately 85% from the peaks measured at the glass output since UV LED sources are by their nature are divergent and tend to “spread” quickly. In a typical UV curing application, the light source would be 2 to 3mm from the material and the peak intensity would be much higher. The 100W mercury vapor lamp source was configured with a light guide that could be positioned much closer, approximately 10mm from the material to be cured.

FTIR System

The FTIR data was gathered at the University of Akron in Akron Ohio. The FTIR system used was a Nexus 870 manufactured by Nicolet.

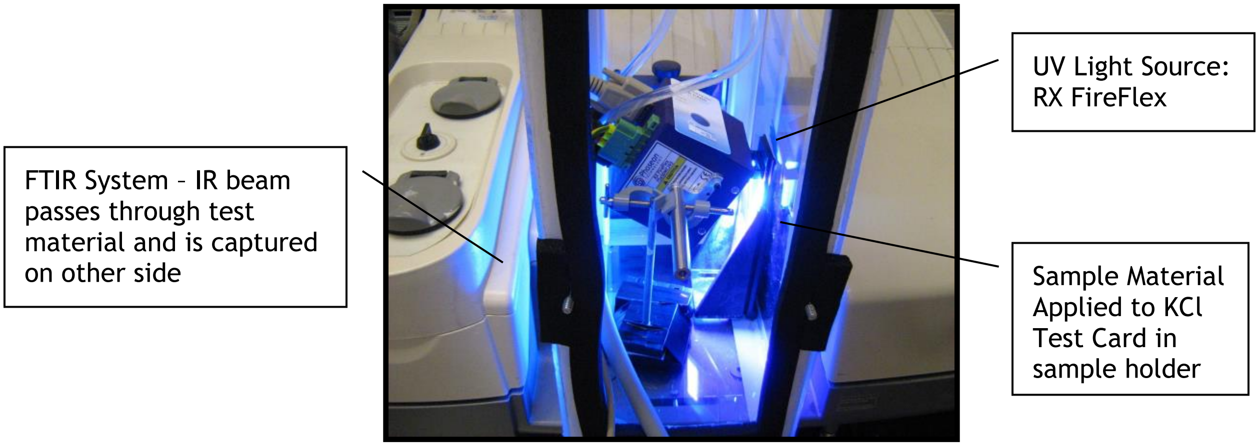

Figure 1: FTIR System Setup

Data Analysis:

FTIR – Real Time Fourier Transform IR is based on how infrared radiation is absorbed by chemical bonds. Each bond type has a distinctive response at a given wavenumber (1/wavelength) where the peak represents the number of bonds. The peak will decrease and finally disappear over time as the polymer chain is formed. This allows the measurement of percent conversion as a function of time by measuring the change in area under the curve at different time intervals.[3]

$$\text{Conversion (\%)} = \frac{\text{[Initial Area]} - \text{[Area at Time t]}}{\text{[Initial Area]}} \times 100\%$$

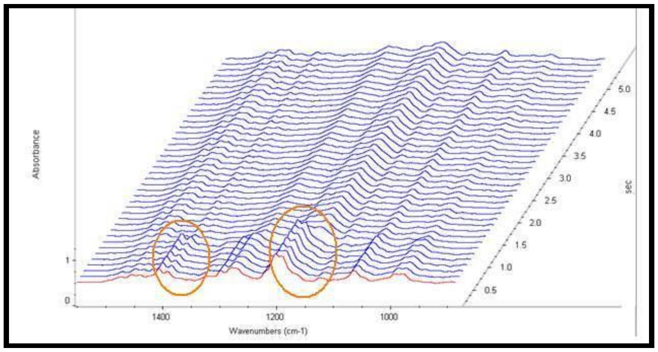

Figure 2: Absorbance versus Wavenumber FTIR Plot for Ink Sample

Typical UV Curing Bond formation:

The inks tested in this paper show diminishing peaks around ~1200, 1400, and 1650 cm^{-1} these correspond to the C=C bond in an alkyl group

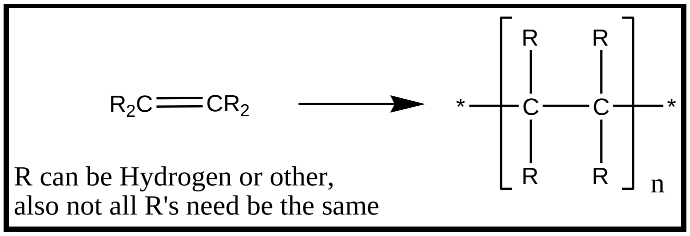

Figure 3: Depiction of C=C bond reaction in alkyl group

The adhesives and coating tested in this paper both show at least one diminishing peak ~800-815 cm^{-1}. This corresponds to the C=C bond in an acrylate group being consumed.

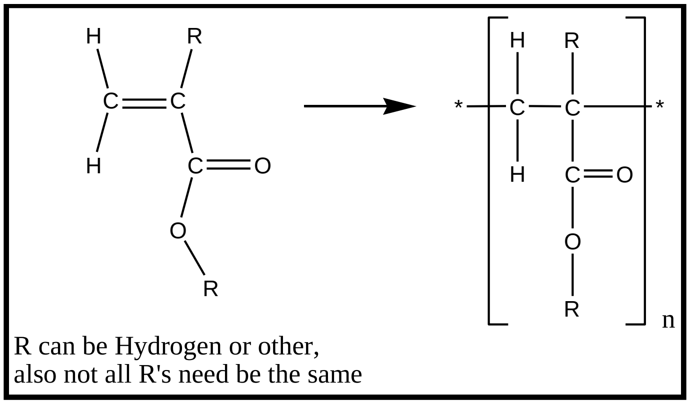

Figure 4: Depiction of C=C bond reaction in acrylate group

As the reaction takes place, the double bond is converted into a single bond as the polymer grows, so at 100% conversion no absorption for the double bond will take place.

Inks:

Two inks that were formulated to cure in the UV-A region were tested with 3 different light sources and at different peak intensities. The results shown in Figure 5 show the effect of different peak intensities of the UV LED 395nm UV light source on curing a black piezo inkjet ink. As noted, this ink was formulated to cure with UV-A, and as the conversion rate curves show increasing the intensity does increase the cure rate. However, the relationship between the cure rate and intensity is not linear (see Figure 6).

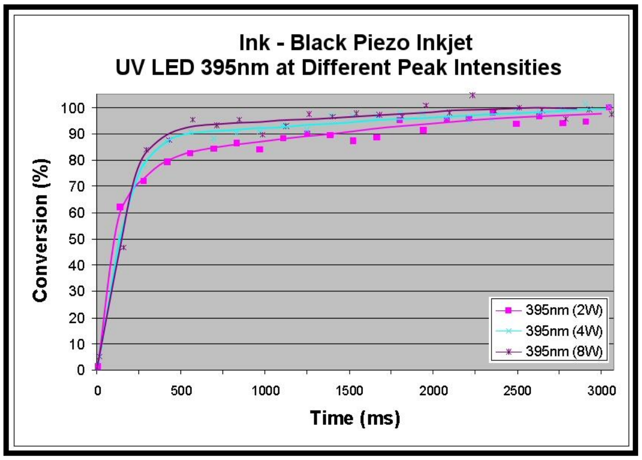

Figure 5: UV LED 395nm UV Source at Different Peak Intensities curing black ink

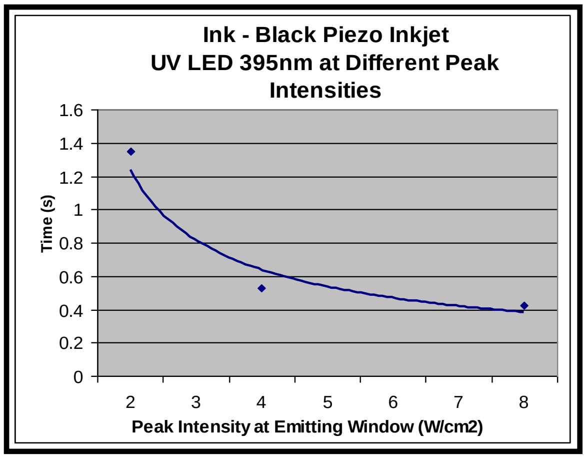

Figure 6: Non-linear Relationship Peak Intensity vs. Cure Rate

Note that the FTIR cure rate analysis does not show other cure properties such as gloss or hardness, nor does it give any indication of surface cure. In addition, there are physical properties of jetted ink that can be improved with increased intensity; e.g. dot gain.

Next, the cure rates of different UV light sources were tested using the same black piezo inkjet ink. As shown in Figure 7, the conversion rate when comparing the 395nm LED UV light source, a mercury vapor lamp and a 365nm LED UV source shows that the fastest cure rate occurs with the highest intensity UV light source that is well matched to the material formulation. Although the mercury vapor lamp and 365nm UV LED UV sources both cure the material, the cure rate is not as fast. When the peak intensity of the UV LED sources is set to the same output, the cure rate for either the 395nm or 365nm UV LED source shows the same cure rate.

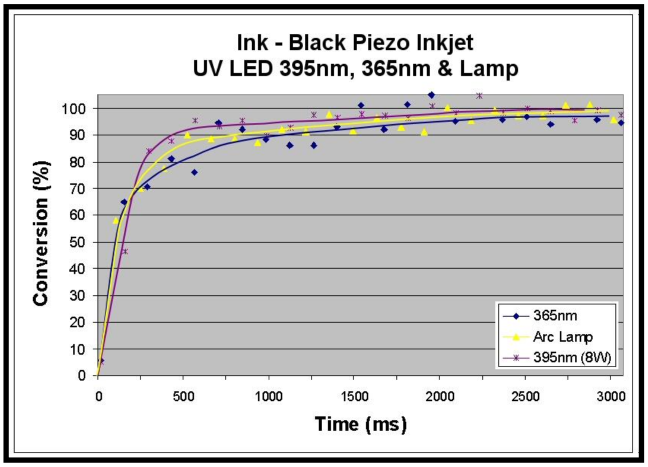

Figure 7: Conversion Rate for UV LED 395nm, 365nm and Mercury Vapor Lamp

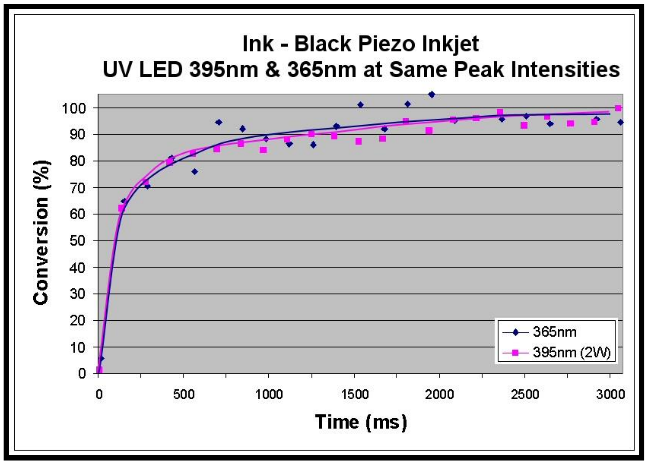

Figure 8: Conversion Rate for UV LED Sources 395nm and 365nm at Same Intensity

Conversion rates for ink from another vendor but also formulated for UV-A show similar results. Different color inks were also tested and the results show that the addition of the pigment does affect cure rates which has been verified in actual printing application testing.

Adhesives:

Two different adhesives were tested, one custom formulated to cure with UV-A LED UV source and one off-the-shelf adhesive commonly used in medical device applications for which the data sheet shows cure preference for a 365nm UV source.

The adhesive that was not formulated to cure exclusively with a UV LED light sources was tested with 3 different light sources, LED sources with peak wavelength emission centered at 395nm and 365nm and a mercury vapor lamp. As Figure 9 illustrates, even when a material is formulated for 365nm, it not only cures at different wavelengths but can cure at a faster cure rate overcoming the non-optimal formulation with the higher peak intensity 395nm UV LED light source. Since this formulation was optimized for 365nm, the cure rate for the 365nm UV LED source does cure at a noticeably higher rate for the adhesive when compared to the ink which was not formulated for this wavelength. The data also suggests that higher peak intensities for the 395nm UV LED source cure at a faster rate and there is a linear relationship between increased intensity and cure rate for this formulation.

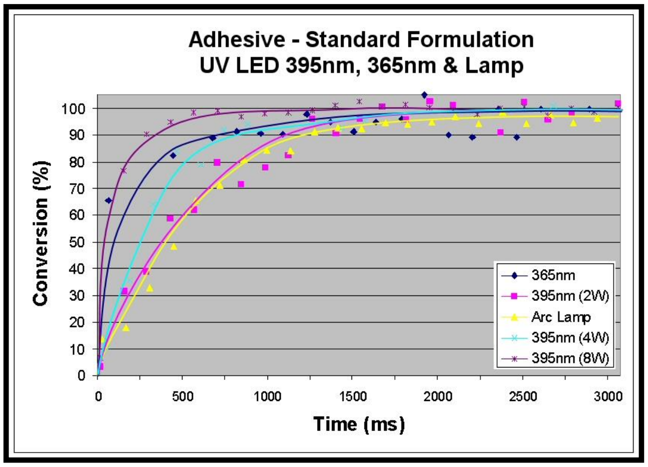

Figure 9: Conversion Rate for UV LED395nm, 365nm and Mercury vapor Lamp

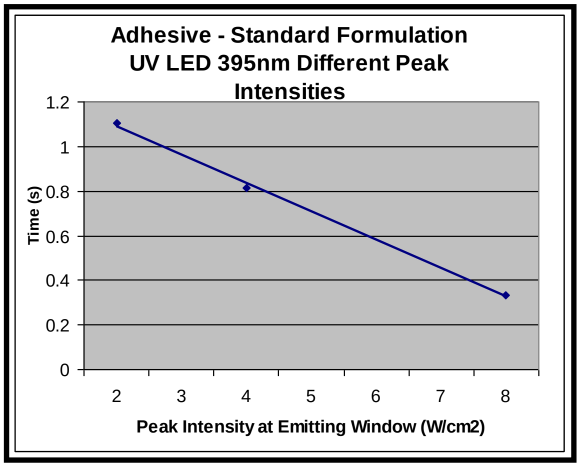

Figure 10: Linear relationship between Peak Intensity and Cure Rate

A custom formulated adhesive that was developed to cure in the UV-A region was tested at different peak intensities of the UV LED source with an output centered at 395nm to show the effect of peak intensity on cure rate. As the peak intensity is increased, the cure rate increases, but as Figure 11 shows, there is a diminishing return when higher peak intensities are used. While the 2W/cm2 light source cures noticeably slower than the others, there is not a significant benefit to increasing the intensity.

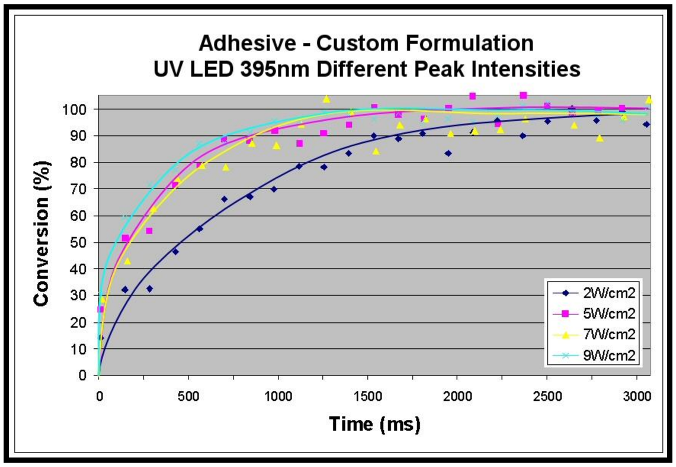

Figure 11: Conversion Rate for UV LED395nm, 365nm and Mercury Vapor Lamp

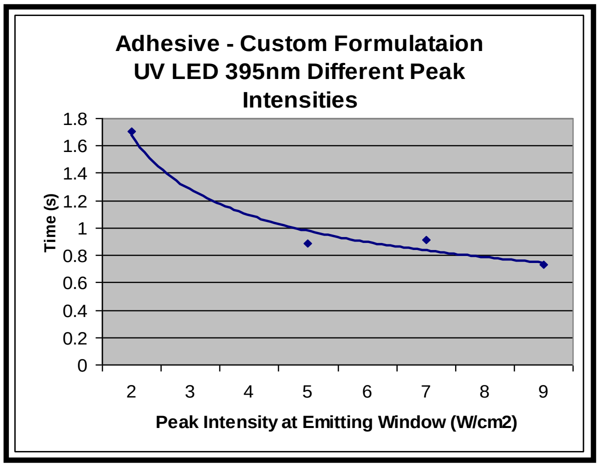

Figure 12: Non-linear relationship between Peak Intensity and Cure Rate

Coating:

A single coating that was formulated to cure with a UV LED light sources was tested with 3 different light sources and at different intensities of the UV LED source with an output centered at 395nm. Even though this material was formulated for the UV-A region, it shows that a mercury vapor lamp not only cures the material, but can cure the material at the same rate as the UV LED source with its peak centered at 395nm. The 365nm UV LED light source is not well matched to this material and does not cure as effectively with the light source. As shown in Figure 13, as the peak intensity is increased, the cure rate increases linearly. This underscores the importance of knowing how a given material formulation will interact with a specific wavelength.

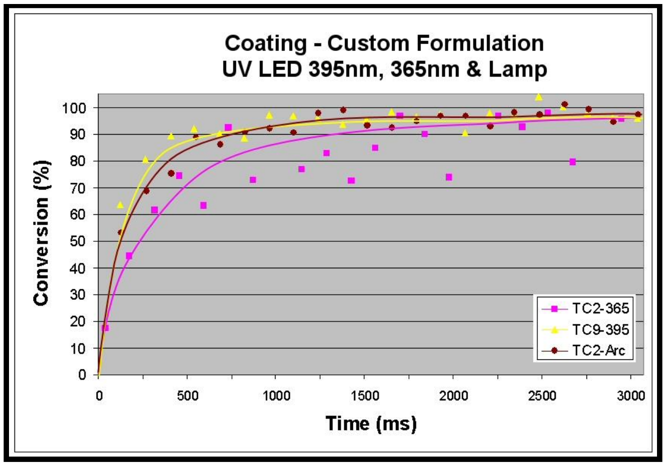

Figure 13: Conversion Rate for UV LED 395nm, 365nm and Mercury vapor Lamp

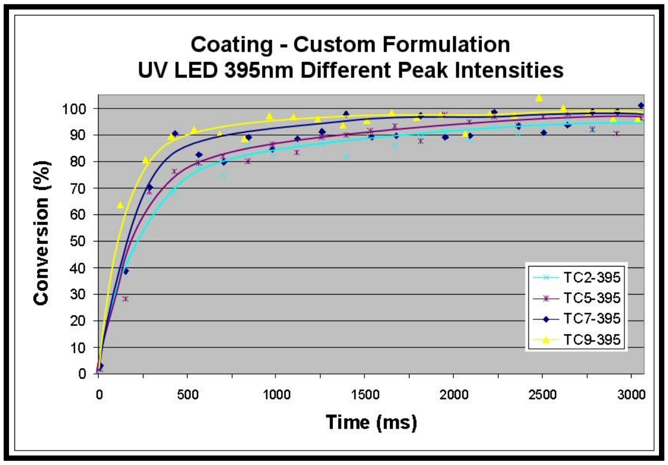

Figure 14: Conversion Rate for UV LED and Mercury Vapor Lamp

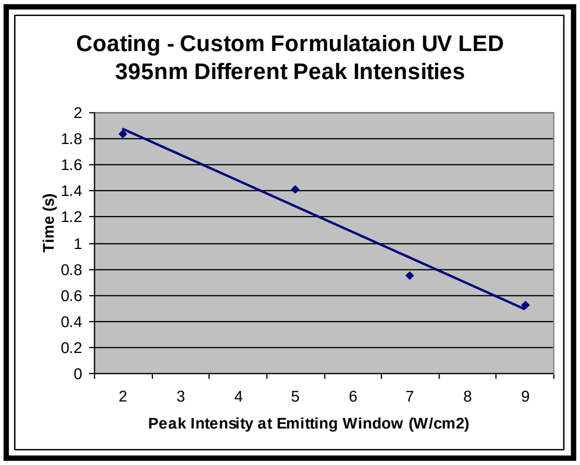

Figure 15: Linear relationship 395nm, 365nm Peak Intensity vs. Cure Rate

Conclusion

The data taken is at least suggestive of some general conclusions - many of which have been validated by our own experiences with testing many different UV materials over several years (both UV LED optimized and "non-optimized"). However we should acknowledge that the data gathered is limited in scope in terms of UV sources used and UV curable materials chosen. Future test should assist with increased confidence in these conclusions.

UV LED Sources are more than capable in most UV curing applications when the material is formulated to accept the energy provided. While there are still limitations in the range of base UV materials available to formulators there is no doubt that practical formulation is possible – for almost any UV application.

The peak intensity and total energy of a UV LED source in the UV-A region is relatively more important for cure performance than the specific peak wavelength of the UV LED source in the UV-A region (365nm vs. 395nm). In the end Energy trumps wavelength in terms of the reaction – at least when the wavelength ranges are relatively close together in the spectrum.

Increasing the speed of cure is important in many practical UV applications but can be limited by the formulation of the material regardless of any practical increase in peak intensity or energy input. As a practical matter, know that not all UV materials show better material performance with faster cure rates.

While not specifically part of the experimental setup and data collected, it should be noted, of course, that UV cure rate as measured using FTIR is only one of several decision factors in determining suitability of UV sources for curing. The physical, mechanical, chemical and process properties of the cured material are in often cases even more critical.

But there is no doubt that the availability of UV LED optimized materials makes UV LED sources, with their inherent advantages, a very attractive option for many applications. But UV LED sources do have technical limitations in terms of their application to a wide range of existing UV materials where they are often not suited and where existing Mercury Vapor lamp technology still remains the best option.

However the prospect of ever increasing availability of suitable base materials to allow for optimized UV LED formulations - as well as the fast increasing capability and cost effectiveness of commercially available UV LED systems – is likely to accelerate the use of UV-LED systems in many applications.

Acknowledgments: We would like to acknowledge Paul Mills of UV Robotics for help with conducting testing and Dr. Jun Hu, PhD and Dr. Mark Soucek, PhD. for resources that aided this research.

Endnotes

[1] I.T. Strategies, 2009 Wide Format Inkjet Graphics Forecast, Hardware Install Base by Technology Units

[2] Lesco a Division of American Ultraviolet, 2002 Datasheet, “Super Spot MK III”

[3] Chen, J.; Soucek, M. D. Photoinitiated Cationic Polymerization of Cycloaliphatic

Epoxide with Siloxane or Alkoxysilane Functionalized Polyol Coatings, *European Polymer Journal* 2003, *39*, pp 505-520.