

## **Characterization & Optimization of UV/EB Curable PSAs by their Dynamic Mechanical Properties**

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### **Abstract:**

Design experiments were conducted to demonstrate the design latitude possible with UV/EB curable pressure sensitive adhesives (PSA). Besides the regular physical testing (tack, peel adhesion, and shear properties), dynamic mechanical analysis were used to characterize the viscoelastic properties of these UV/EB curable PSAs and compare with those of traditional waterborne and hot melt PSAs. Basic chemistry design differences among hot melt, waterborne, and UV/EB systems will be discussed. Effects of adhesive thickness and curing dosage, which are unique characteristics of UV curable PSA systems, will also be discussed to help converters better master the uses of UV/EB curable PSAs.

### **Introduction**

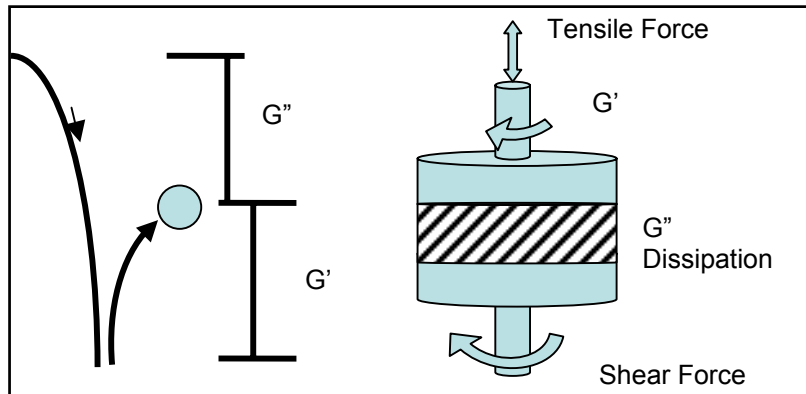
Pressure sensitive adhesives (PSAs) are found in many everyday applications including labels, masking tapes, packaging tapes, note pads and many other different types of applications. Conventional PSAs come in three different physical forms: solvent borne (solution), water borne (emulsion), and hot melts. Due to environmental regulations and valuable floor space in press rooms, some of the applications have been challenged by UV and EB curable PSAs which offer zero VOC, lower energy processing costs, short run flexibility and process enabling capability. UV/EB PSAs also come in several forms including room temperature (RT) liquid, warm melt, and hot melt and, more often than not, higher viscosity systems can satisfy higher demand requirements than do lower viscosity RT liquid systems.

PSAs have been studied extensively in the field of viscoelasticity using dynamic mechanical analysis (DMA) pioneered by Dr. Dahlquist.<sup>1</sup> Many adhesive properties and performance characteristics have been correlated and predicted very well by characterizing their viscoelastic behaviors using DMA.<sup>1-7</sup> While showing the performance attributes of UV curable PSAs with our design of experiment, we have also used DMA to characterize these UV adhesives and compared the results with those from hot melt and water borne PSAs. The formulation windows of UV/EB curable PSAs will be demonstrated with the exhibits and the effects of process parameters (such as thickness, cure dosage, and line speed) on the properties of the adhesives will be illustrated.

### **Correlation of Viscoelastic Properties and Adhesive Performance**

The overall performance of a pressure sensitive adhesive depends not only on its bulk properties (viscoelastic behavior) but also on the surface chemistry, chemical affinity, the fracture mechanism, and the structural design of the tape. For any given PSA application, once the backing material and adherent are specified and the interfacial criteria are met, the performance of the tape is then

dominated by the viscoelastic properties of the adhesive itself. A simplistic graphical representation of the “**elastic**” and “**viscous**” responses and testing of a typical polymeric material such as a PSA is shown in Fig.1.



**Fig.1 Simplified schematics of the viscoelastic behavior and testing of a polymeric material.  $G'$ : elastic modulus (elastic response, stored energy);  $G''$ : loss modulus (viscous response, dissipated energy).  $\tan \delta = G''/G'$ : dissipating factor.**

Performance of the PSA is evaluated in both the bonding and de-bonding stages. The response of the adhesive in these two stages is through the participation of two primary mechanisms of deformation of the polymer i.e., viscous flow and elastic deformation. The viscous flow proceeds through biased diffusion and requires appreciable time whereas the elastic deformation stores energy and dominates at higher speed. The participation of these two processes varies with temperature and time (rate, frequency) as illustrated in Fig. 2 (typical temperature scan) & Fig. 3 (frequency scans at various temperatures, i.e., master curve). It is this time and temperature dependency that sometimes makes it difficult to correlate the performance of the adhesive with results from standard testing methods.

There are two papers that suggested the so-called “tape performance windows” constructed from DMA data which allow us to describe, predict and categorize adhesive tapes. Dr. Lin<sup>2</sup> constructed the **performance windows** with the master curve that described and predicted the performance of adhesives for almost every condition of use. This method is thorough but is too time consuming for screening purposes in a design of experiment. Carper<sup>8</sup> suggested the **tape category windows** which are based on end use categories of the adhesive. We decided to use this tape category window for demonstration purposes. The basic diagram is shown in Fig.4.

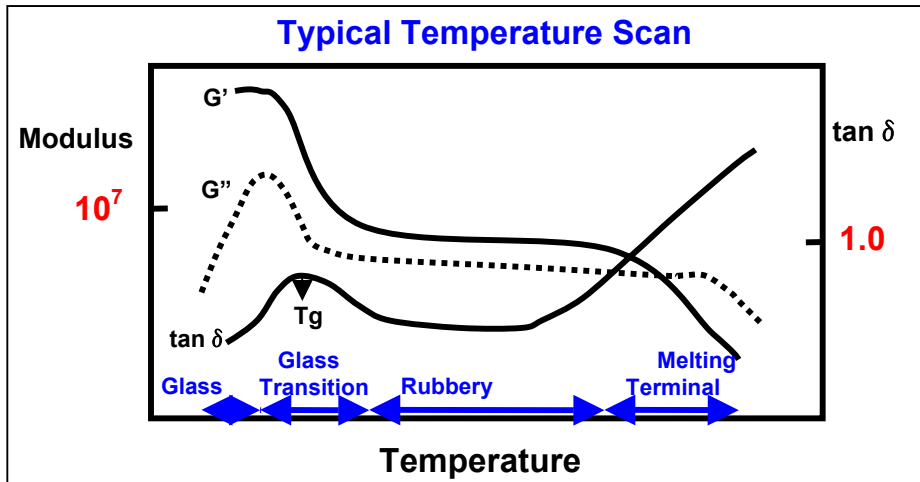


Fig. 2 DMA Scan of temperature sweep of a typical pressure sensitive adhesive

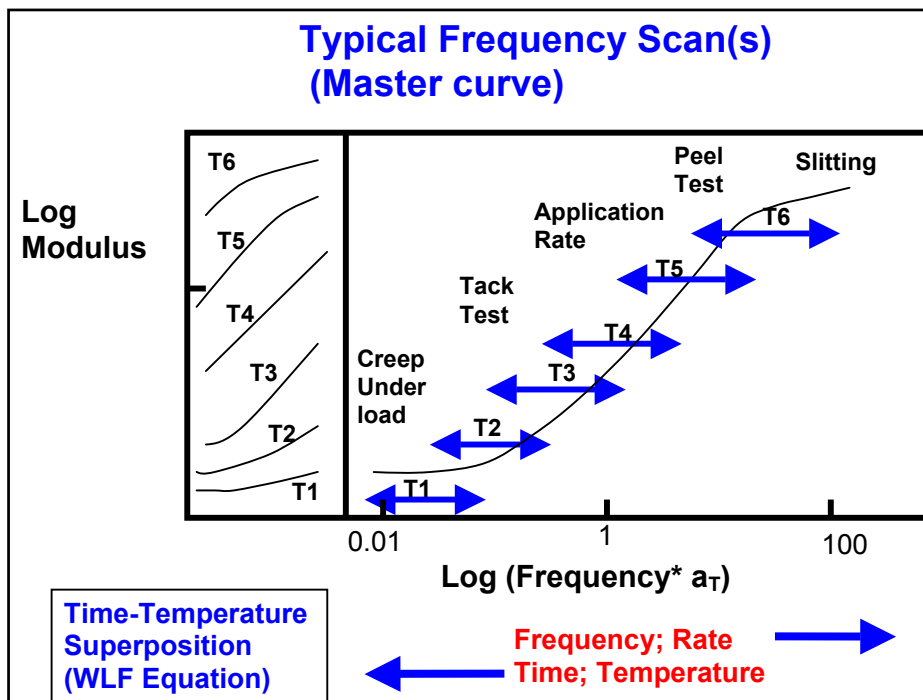


Fig. 3 DMA scan of frequency sweep and construction of master curve of a typical pressure sensitive adhesive.

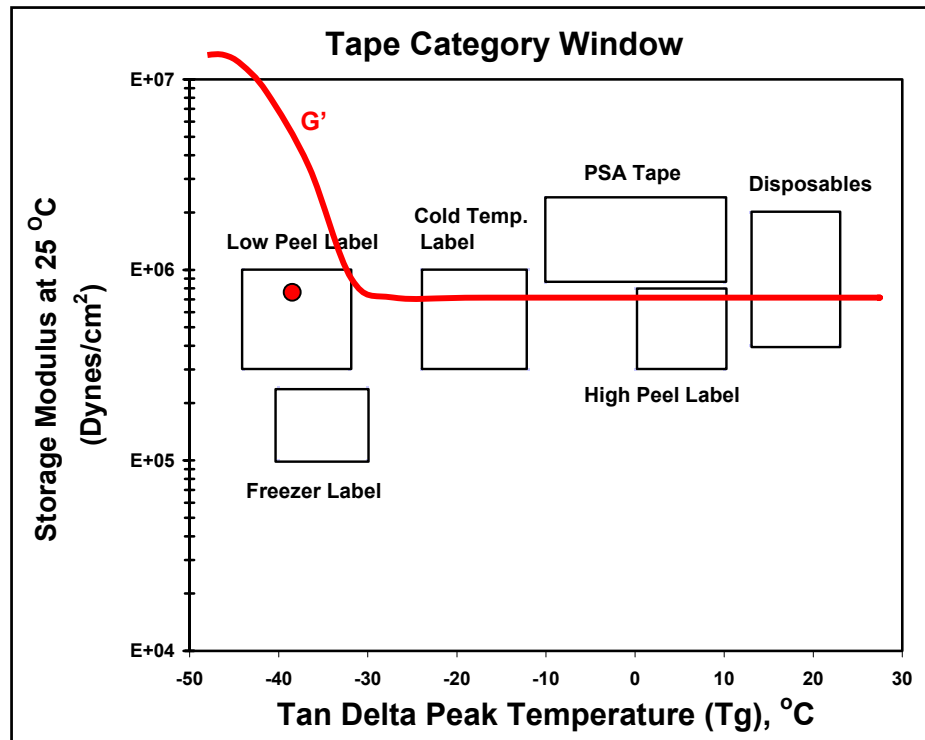


Fig. 4 Basic Tape category windows by end-uses constructed from DMA data.

**Experimental:**

All adhesives were coated on 2 mil corona-treated Mylar and dried or cured to a dry adhesive thickness of 1.0-1.1 mils. UV curing was accomplished with a UV Processor from Aetek International equipped with one 300 Watt/inch medium pressure mercury arc lamp (H-bulb) at an appropriate dosage as specified in the results. The dosage was measured with EIT's Uvicure Plus radiometer.

180 degree peel testing was performed per PSTC-101 and loop tack testing per PSTC-16. DMA runs were conducted with an ARES model rheometer from Rheometrics.

**Results and Discussion:**

For comparison purposes, three classes of commercial hot melt PSAs and five classes of commercial water borne acrylic PSAs were evaluated. Their tack and peel values along with their class description are listed in Table 1 and Table 2 respectively. Their DMA data were analyzed and placed into the tape category windows as shown in Fig. 5 & 6. The data are combined in Fig. 7 for trend analysis.

Even though the end-use categories of these selected adhesives understandably do not necessarily match those listed by the original authors, we can still see the general trend as shown in Fig. 7. Higher peel, more permanent PSAs are located toward the right upper corner of the graph as circled by the larger oval whereas, the lower peel, more removable PSAs are located toward the lower left corner.

Table 1. Peel and Tack Values of 3 Commercial Hot Melt PSA

| <b>Hot Melt PSA</b> |                             |                          |                         |                          |
|---------------------|-----------------------------|--------------------------|-------------------------|--------------------------|
|                     | <b>Class</b>                | <b>Immed. Peel (gli)</b> | <b>24 Hr Peel (gli)</b> | <b>Loop Tack (lb/in)</b> |
| <b>HM-1</b>         | Permanent<br>Low Viscosity  | 4100                     | 3990                    | 4.20                     |
| <b>HM-2</b>         | Permanent<br>High Viscosity | 2300                     | 3430                    | 6.86                     |
| <b>HM-3</b>         | Removable                   | 335                      | 625                     | 1.61                     |

Table 2. Peel and Tack Values of 3 Commercial Hot Melt PSA

| <b>Water Borne PSA</b> |                            |                          |                         |                          |
|------------------------|----------------------------|--------------------------|-------------------------|--------------------------|
|                        | <b>Class</b>               | <b>Immed. Peel (gli)</b> | <b>24 Hr Peel (gli)</b> | <b>Loop Tack (lb/in)</b> |
| <b>WB-1</b>            | Permanent<br>Industrial    | 1460                     | 1810                    | 4.12                     |
| <b>WB-2</b>            | Permanent                  | 1280                     | 3025                    | 2.70                     |
| <b>WB-3</b>            | Permanent<br>Paper/GP      | 350                      | 690                     | 2.70                     |
| <b>WB-4</b>            | Removable                  | 460                      | 910                     | 1.27                     |
| <b>WB-5</b>            | Removable<br>(Microsphere) | 540                      | 870                     | 1.38                     |

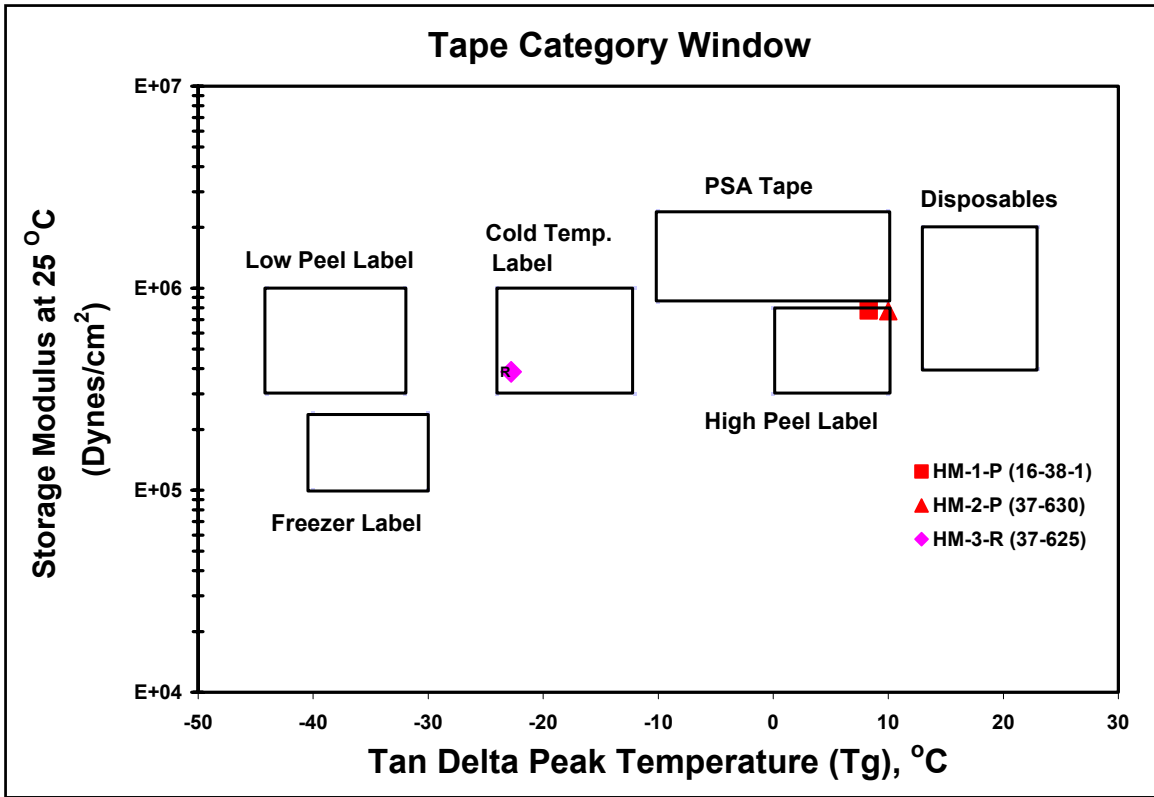


Fig. 5 Three classes of Hot melt PSA in end-use Tape Category Windows based on DMA data.

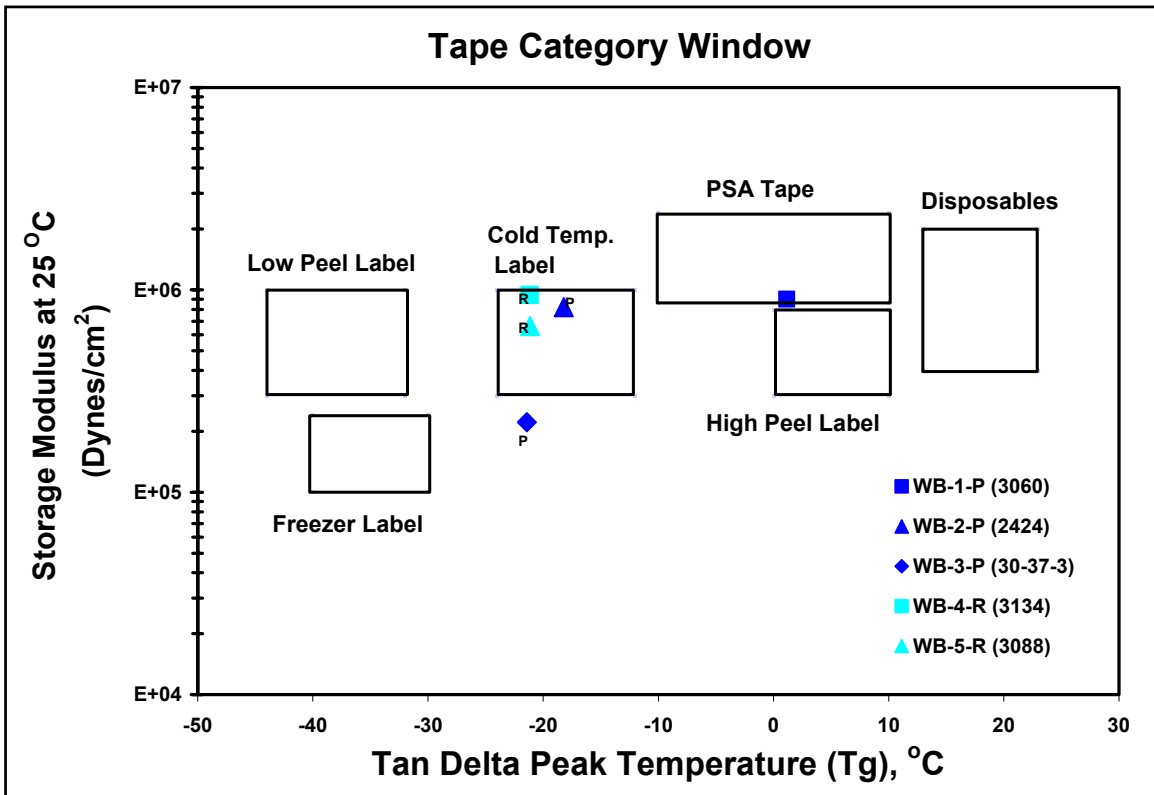


Fig. 6 Five classes of WB PSA in end-use Tape Category Windows based on DMA data

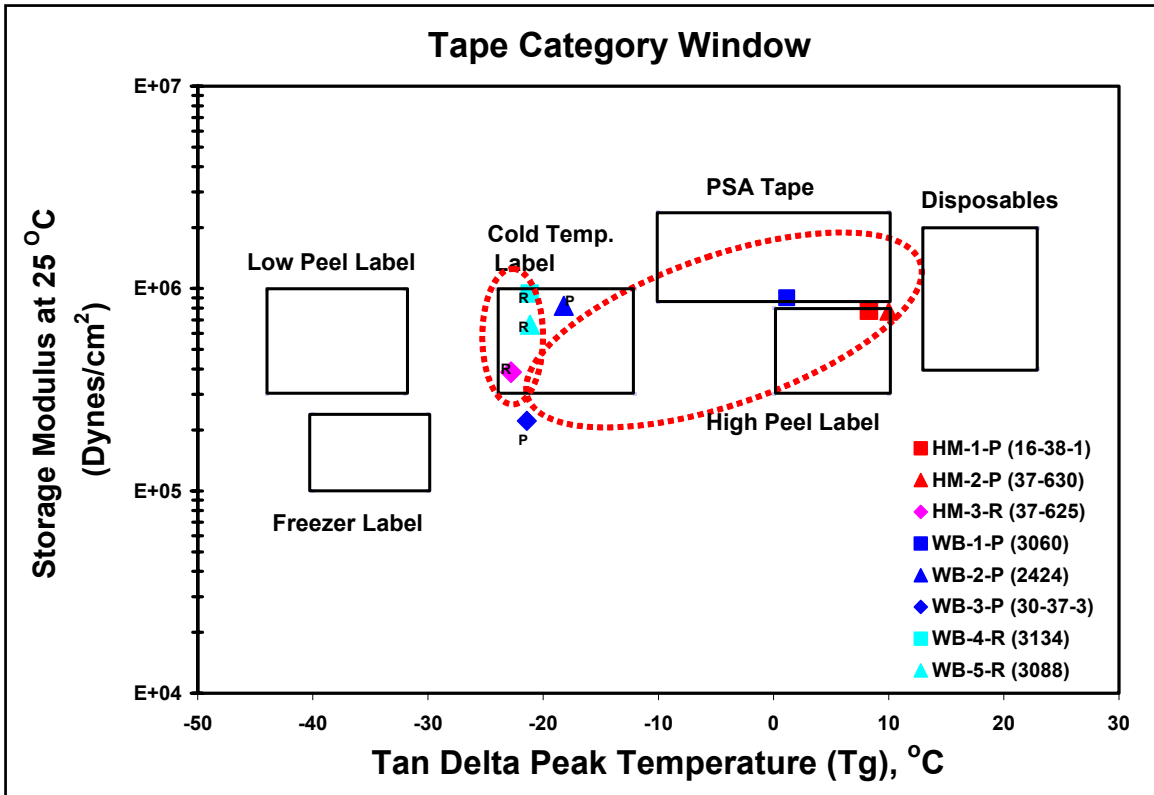


Fig. 7 Summarized plot of 3 HM PSAs and 5 WB PSAs in end-use Tape Category Windows based on their DMA data.

UV PSAs generally consist of blends of acrylated oligomers, monomers, non-reactive tackifiers, and proper additives for processing and performance purposes. The experimental design for our demonstration is listed in Table 3. As the viscosity data suggest the adhesives are all RT liquid systems except UV-A, which should be a warm melt system that will need mild heating for application purposes.

Table 3. UV PSA Formulation for Design Experiments

| <b>UV PSA Design of Experiment</b> |                    |                         |                   |                        |
|------------------------------------|--------------------|-------------------------|-------------------|------------------------|
|                                    | <b>Oligomer, %</b> | <b>Monomer Blend, %</b> | <b>Tackier, %</b> | <b>(cps) Viscosity</b> |
| <b>UV-A</b>                        | 20                 | 50                      | 30                | 7700                   |
| <b>UV-B</b>                        | 5                  | 65                      | 30                | 945                    |
| <b>UV-C</b>                        | 20                 | 70                      | 10                | 495                    |
| <b>UV-D</b>                        | 13.5               | 79                      | 16                | 690                    |
| <b>UV-E</b>                        | 5                  | 79                      | 10                | 150                    |
| <b>UV-F</b>                        | 11                 | 57.5                    | 30                | 175                    |
| <b>UV-G</b>                        | 12.5               | 61.67                   | 21.67             | 2795                   |
| <b>UV-H</b>                        | 16.67              | 25.51                   | 61.74             | 1280                   |

The corresponding tack and peel values are listed in Table 4. From the designed experiment, several optimized formulas were developed based on specific applications. Their tack and peel data are listed in Table 5. The tack & peel range for HM, WB and UV PSAs are summarized in Table 6.

**Table 4. Tack & Peel Values of UV PSA Formulations**

| <b>Results of UV PSA Design of Experiment</b> |                          |                         |                          |                        |
|---|--------------------------|-------------------------|--------------------------|------------------------|
|   | <b>Immed. Peel (gli)</b> | <b>24 Hr Peel (gli)</b> | <b>Loop Tack (lb/in)</b> | <b>(cps) Viscosity</b> |
| <b>UV-A</b>                                   | 1320                     | 1600                    | 0.33                     | 7700                   |
| <b>UV-B</b>                                   | 1440                     | 1585                    | 0.56                     | 945                    |
| <b>UV-C</b>                                   | 160                      | 185                     | 1.46                     | 495                    |
| <b>UV-D</b>                                   | 735                      | 820                     | 0.86                     | 690                    |
| <b>UV-E</b>                                   | 235                      | 365                     | 1.68                     | 150                    |
| <b>UV-F</b>                                   | 100                      | 120                     | 1.38                     | 175                    |
| <b>UV-G</b>                                   | 1640                     | 1640                    | 0.52                     | 2795                   |
| <b>UV-H</b>                                   | 835                      | 1025                    | 0.78                     | 1280                   |

**Table 5. Tack & Peel Values for Optimized UV PSAs for Special Applications**

| <b>Other UV PSAs</b> |                         |                          |                         |                          |
|----------------------|-------------------------|--------------------------|-------------------------|--------------------------|
|                      | <b>Class</b>            | <b>Immed. Peel (gli)</b> | <b>24 Hr Peel (gli)</b> | <b>Loop Tack (lb/in)</b> |
| <b>UVPS-38</b>       | Permanent (Plastics)    | 1530                     | 1480                    | 1.80                     |
| <b>UVPS-76</b>       | Permanent (Paper)       | 795                      | 770                     | 4.83                     |
| <b>UVHM-1</b>        | Permanent (Film, Paper) | 895                      | 1425                    | 2.73                     |
| <b>UVHM-2</b>        | Permanent (Paper-GP)    | 1230                     | 1560                    | 2.58                     |

**Table 6. Comparison of Tack & Peel by Technology**

| <b>Comparison by Technology</b> |                      |                          |                         |                          |                     |
|---------------------------------|----------------------|--------------------------|-------------------------|--------------------------|---------------------|
|                                 | <b>Class</b>         | <b>Immed. Peel (gli)</b> | <b>24 Hr Peel (gli)</b> | <b>Loop Tack (lb/in)</b> | <b>Heat Resist.</b> |
| <b>Hot Melt</b>                 | Permanent -Removable | 335-4100                 | 625-3990                | 1.6-6.86                 | Poor                |
| <b>Water Borne</b>              | Permanent -Removable | 350-1460                 | 690-3025                | 1.27-4.12                | Good                |
| <b>UV</b>                       | Permanent -Removable | 100-1640<br>(100-1670)   | 120-1640<br>(120-1990)  | 0.33-4.83<br>(0.33-5.32) | Good                |



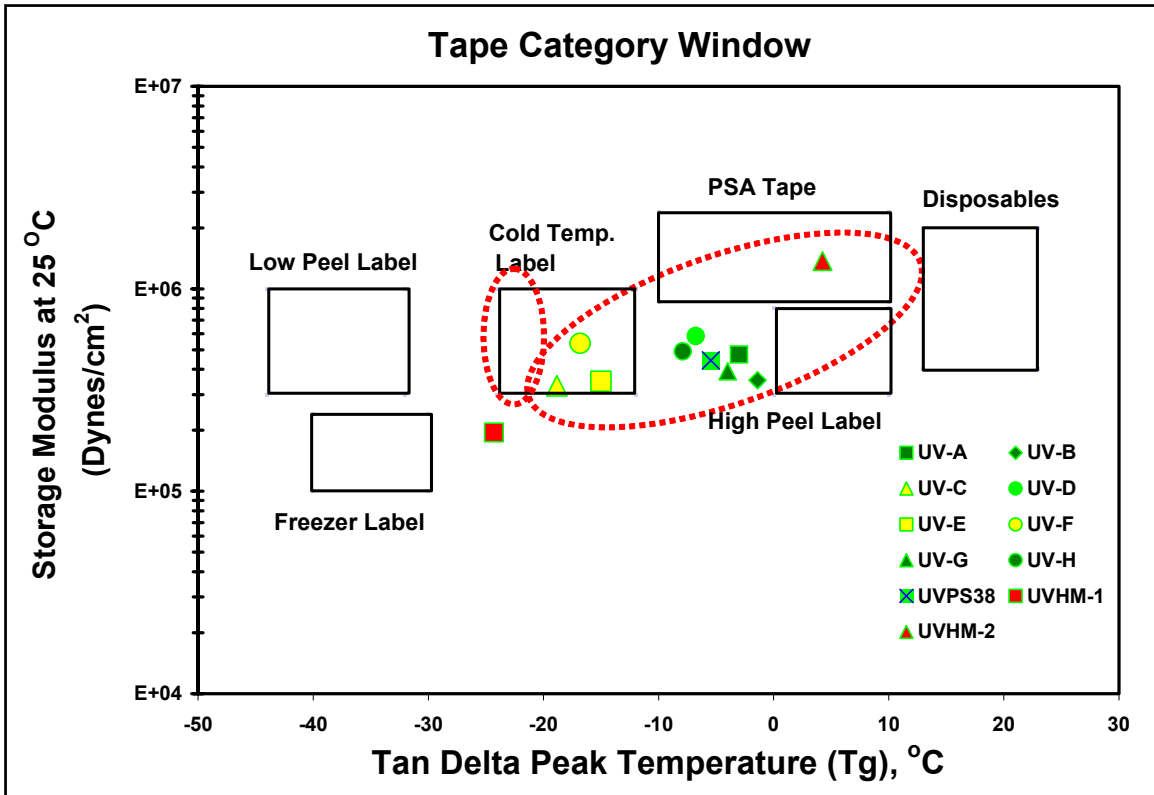


Fig. 8 UV PSA in Tape Category Windows Based on their DMA Data.

As one can see from Table 6 and Fig. 8 (UV PSAs in category windows), the formulation latitude with UV PSAs covers a good portion of the performance space defined by HM and WB PSAs. One thing the Tape Category Window cannot reveal is information related to heat resistance, even though the data to predict it are available from the DMA temperature sweep. The relative ranking of heat resistance data of the adhesives in the study is listed in Table 6.

**Effect of Adhesive Thickness**

UVPS-76 is designed to be a slower curing system with optimum performance at a thickness around 1 mil. Fig. 9 shows decreasing peel and tack performance with increasing adhesive thickness mainly due to a lower degree of reaction and poor through-cure with higher thickness. The thickness effect for UV curable PSAs, in general, is more pronounced than with HM or WB PSAs. Care must be taken to ensure consistent coating weight to ensure consistent adhesive performance.

**Effect of UV Curing Dosage/Line Speed**

Proper cure conditions (UV curing dosage, line speed, and lamp wattage) must be predetermined and optimized according to processing requirements since they will greatly affect adhesive performance. On the other hand, curing dosage can also be used to tailor performance with the same formulation. Fig.

10 shows the optimal cure dosage for the above mentioned UVPS-76 at 225 mJ/cm<sup>2</sup>, above which the adhesive develops better cohesive strength with lower tack as we would expect from traditional PSAs with some crosslinking taking place.

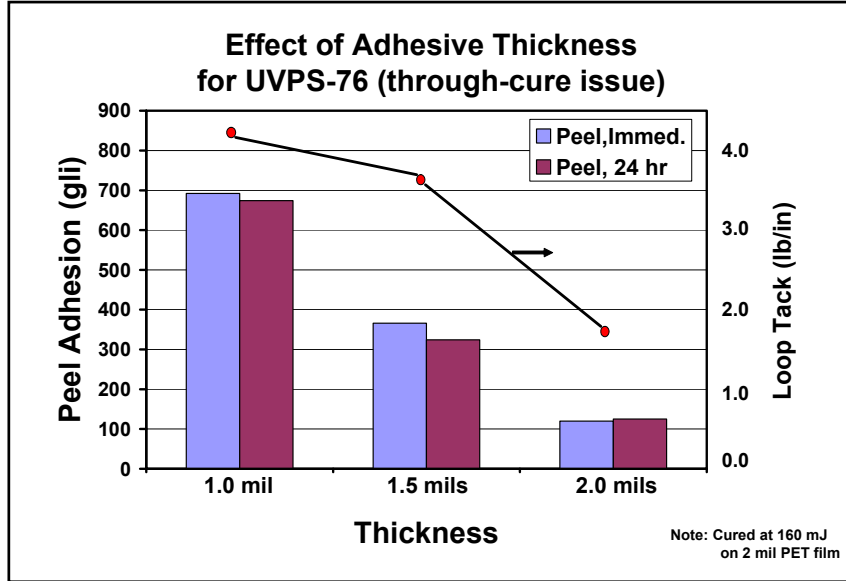


Fig. 9 Peel Performance of UVPS-76 with Thickness Variation

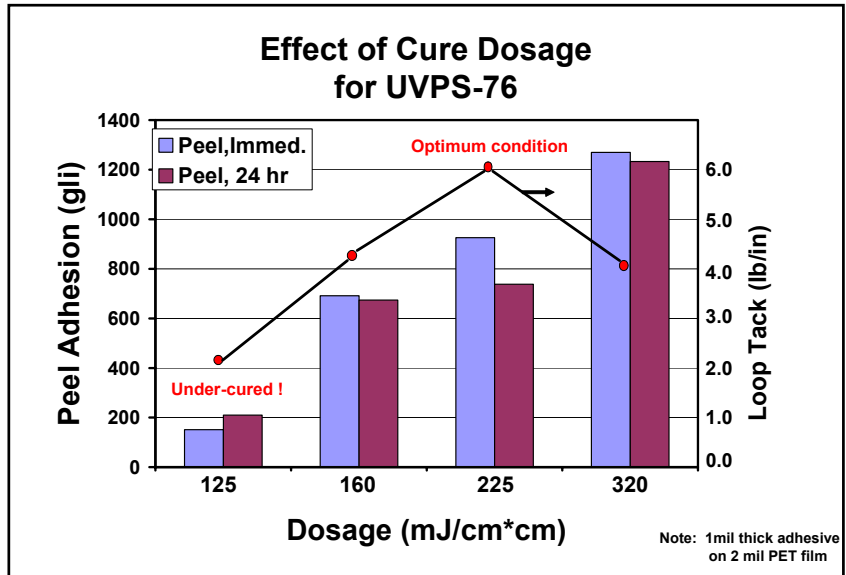


Fig. 10 Peel Performance of UVPS-76 with Dosage Variation

UVPS-38, on the other hand, is designed to be a faster curing, better through-cure, and higher cohesive strength system than UVPS-76. The curing dosage has a significant effect on the tackiness of the adhesive to the point where it

becomes a post-it type removable adhesive (Fig. 11). Its tack and peel responses with thickness variation are more like traditional systems as we know them (Fig. 12). This is due to a different reason, i.e., slight under-cure at the bottom layer.

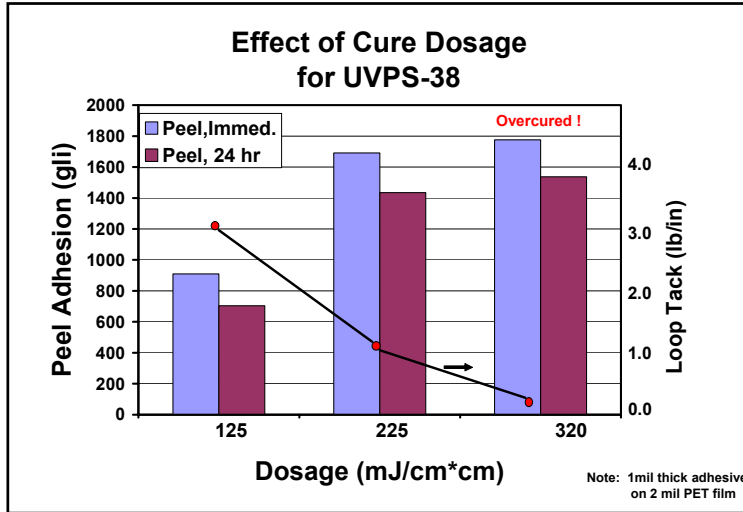


Fig. 11 Peel Performance of UVPS-38 with Dosage Variation

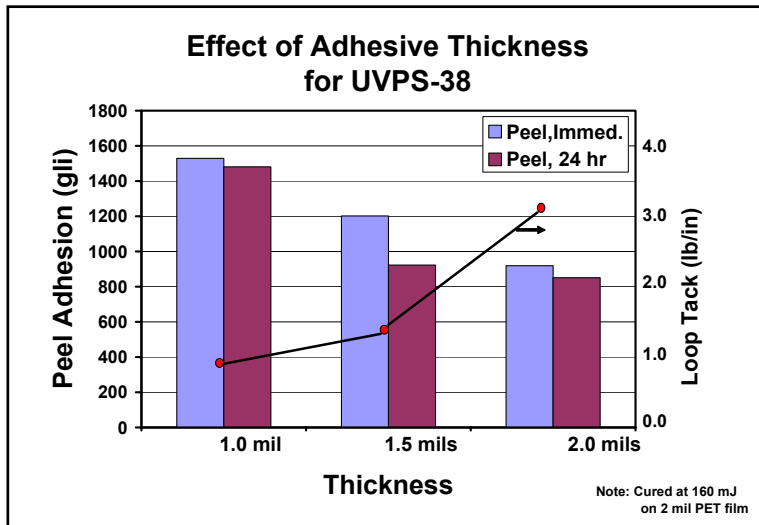


Fig. 12 Peel Performance of UVPS-38 with Thickness Variation

The curing dosage effect with UV curable PSAs is unlike what converters have grown used to with the traditional chemistries (hot melt, water borne, and solvent borne PSAs). The performance trend with thickness variation may also be different from our traditional experience. As we mentioned earlier, these can become design features providing flexibility or causing QC nightmares depending on ones' understanding of causes and effects with UV chemistry.

## **Conclusions**

We have demonstrated that the formulation latitude with UV curable PSAs can be very broad, based on the standard physical PSA testing methods and on viscoelastic properties measured from dynamic mechanical analysis (DMA). The "Tape Category Windows" approach from DMA data can be a good screening tool to understand performance trends and predict end-use categories to a limited degree. Prediction of the shear, temperature-resistance, and long term properties, however, still requires a full DMA curve or "Master Curve characterization".

From a market share perspective, UV/EB curable PSAs still represent less than 2% of the non-captive uses and are generally used in specialized situations where spot application and space limitation are of utmost concern. There are still hurdles to be overcome such as much higher raw material cost, process-sensitive performance, higher than normal residual monomers (compared to UV/EB coatings), and higher heat- & shear-resistance especially with room temperature liquid and warm melt systems. Hopefully, with more development from raw material suppliers & formulators, and education of the converters, we can turn the corner for UV/EB curable pressure sensitive adhesives.

## **Special Thanks**

The authors would like to thank Kathy Meyers, chemist, for performing all the DMA testing.

## **References:**

1. "C.A. Dahlquist "Adhesion: Fundamentals and Practice", Proc. Nottingham Conference on Adhesives; MacLaren & Sons, Ltd., London (1966).
2. A. Lin, "Prediction of Adhesive Performances by their Dynamic Mechanical Behavior", p505, TAPPI Polymers, Laminations & Coating Conference Proceeding (1991).
3. J.D. Ferry, "Viscoelastic Properties of Polymers", 2<sup>nd</sup> ed., Wiley, NY (1970)
4. S.G. Chu, Handbook of Pressure Sensitive Adhesives Technology, D. Satas editor, Chapter 8, Van Nostrand Reinhold Co., NY (1989).
5. D.W. Aubrey, M. Sherriff, J. Polym. Sci., Polym. Chem. Ed., 16, p2632 (1978).
6. M. Sheriff, R.W. Knibbs, P.G. Langley, J. Appl. Polym. Sci., 17, 3423 (1973).
7. D. Satas, Adhesives Age, 15 (10), 19 (1972).
8. J.D. Carper, Adhesives Age, Aug. p35 (1989).