# Robust PSA's Using High-Diblock Radial Styrene-Isoprene-Styrene Rubber

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Being able to produce a general purpose PSA that is robust to variations in resin and polymer addition is an advantage to the PSA market. It allows for the use of simple unsophisticated feeding systems to the adhesive mixer. It also improves quality because lot-to-lot variation in PSA performance will be reduced. This paper will show by mixture DOE that PSA's with limited variation in 180 Degree Peel, Loop Tack, and SAFT can be produced in spite of variations in the concentrations of tackifying resin and polymer.

| Table I. Adhesive Raw Materials | |
|---|---|
| Material | Description |
| Radial polymer | 18% styrene SIS, an MFR of 23dg/min, & 73% diblock |
| Resin 1 | Aromatic modified C5 with a S.P. of ~95 °C |
| Resin 2 | Hydrogenated rosin ester with a S.P. of ~100 °C |
| Oil | Paraffin oil |
| Antioxidants | Phenolic/phosphite package |

Table I lists the raw materials used in this study. Table II lists the adhesive formulae used in this study and identifies the design points. A simple simplex center-point mixture design was used. In this design, the concentrations of polymer and two tackifying resins were varied. The concentrations of oil and anti-oxidant were kept constant. The adhesives were mixed at 50% solids in toluene. A portion of the adhesives were cast on to Mylar film at 1.5 mils dry, followed by PSA testing on stainless steel surfaces. The remaining adhesive mixtures were then vacuum devolatilized and the $T_g$ and elastic modulus of each adhesive was measured by DMA.[1]

| Table II. Adhesive Formulae, % Wt./Wt. - Simplex Mixture DOE | | | | | | | |
|---|---|---|---|---|---|---|---|
| Run No. | Point Type | Polymer | Resin 1 | Resin 2 | Oil | A.O. Package | Total, % |
| 1 | Center Edge | 29.8 | 34.0 | 21.3 | 14.0 | 1 | 100.0 |
| 2 | Center | 31.2 | 31.2 | 22.7 | 14.0 | 1 | 100.0 |
| 3 | Vertex | 25.5 | 34.0 | 25.5 | 14.0 | 1 | 100.0 |
| 4 | Center Edge | 34.0 | 29.8 | 21.3 | 14.0 | 1 | 100.0 |
| 5 | Vertex | 34.0 | 25.5 | 25.5 | 14.0 | 1 | 100.0 |
| 6 | Vertex | 34.0 | 34.0 | 17.0 | 14.0 | 1 | 100.0 |
| 7 | Center Edge | 29.8 | 29.8 | 25.5 | 14.0 | 1 | 100.0 |
| The simplex Design Points were Polymer 30-40%; Resin 1 was 30-40%; and Resin 2 was 20-30%. Oil and A.O. were held constant. Thus the given formulae are calculated by Design Point*85%. | | | | | | | |

The 180 Degree Peel, Loop Tack and SAFT values are listed in Table III. The data indicates fairly limited swing in values despite the changes in polymer and tackifying resin concentration. For example,

the 180 Degree Peel values range from 5.7 to 6.2 pli; this is a fairly narrow range. Similarly, Loop Tack ranges from 7 to 12 psi, with most of the runs being 8 to 9 psi. The PSA values are maintained despite the variation of polymer concentration from 30 to 40%, C5/C9 resin from 30 to 40%, and hydrogenated rosin ester from 20 to 30%.

DMA results explain the robust response to variation in raw material concentration. Figure 1 is a contour graph of the G' at 23 °C. The red points on the graph are the design points listed in Table II. All the design points and the entire contour space meet the Dahlquist criteria.[2] Also the contour is relatively flat, with each contour spacing being fairly broad indicating a fairly shallow slope; going from a high of 50,000 Pa to a low of 40,000 Pa. The low slope contour explains the limited response in PSA properties as raw material concentration is varied. The likely cause for this response is the high amount of diblock in the polymer. Diblock acts as a viscous component, thus there is a limited amount of elastic component to be modified by the resin. A way to confirm this would be to repeat the design using a low-diblock radial polymer and compare the resulting contour graphs.

Table III. Responses – PSA Results

| Run No. | Point Type | 180 Peel, pli | Loop Tack, psi | SAFT, °C |
|---|---|---|---|---|
| 1 | Center Edge | 6.1 | 9 | 79.2 |
| 2 | Center | 6.2 | 9 | 79.1 |
| 3 | Vertex | 6.3 | 12 | 72.1 |
| 4 | Center Edge | 5.7 | 8 | 79.4 |
| 5 | Vertex | 5.7 | 7 | 79.7 |
| 6 | Vertex | 5.9 | 8 | 81.7 |
| 7 | Center Edge | 5.8 | 9 | 78.4 |

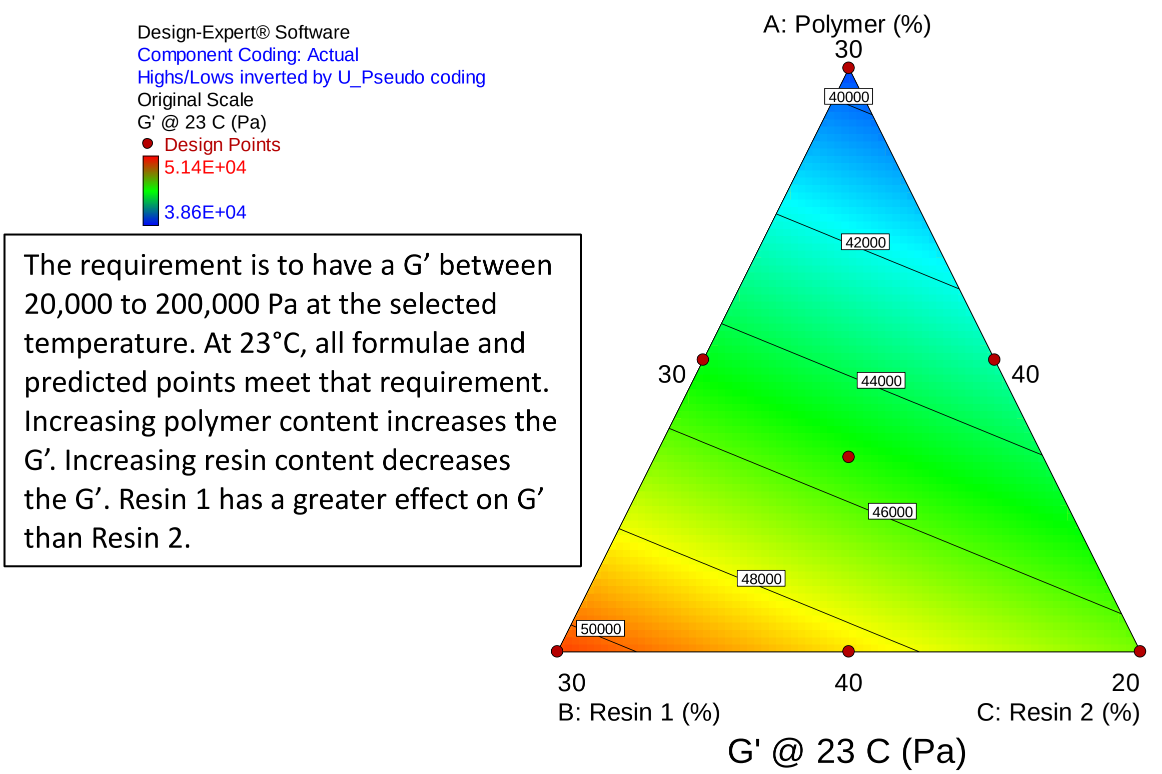
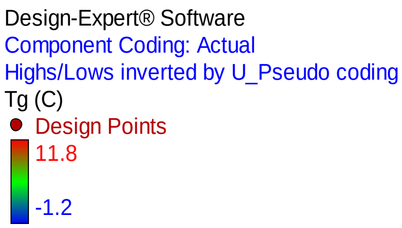



Figure 1. Contour Graph of G' at 23°C, Pa

Glass transition temperature, however, does vary with resin/polymer concentration. Figure 2 is a contour graph of the $T_g$ of the design points. The results indicated are expected. As resin concentration increases, $T_g$ increases. Conversely, as polymer concentration increases, $T_g$ decreases. Adding resin in an SIS system increases the Tg of the isoprene midblock. Thus as the resin to polymer ratio increases, the $T_g$ of the adhesives increases; and conversely if the resin to polymer ratio decreases, the $T_g$ of the adhesive decreases. Nonetheless, the $T_g$ of the PSA's fall within the recommended range of -10 to 15 C for a general purpose PSA.




As one of the main effects of adding resin is to increase the $T_g$ of the isoprene mid-block, adding resin increases the $T_g$ of the adhesive. The range of $T_g$'s, however, meets the requirement of -10 to 15°C for a general purpose PSA.

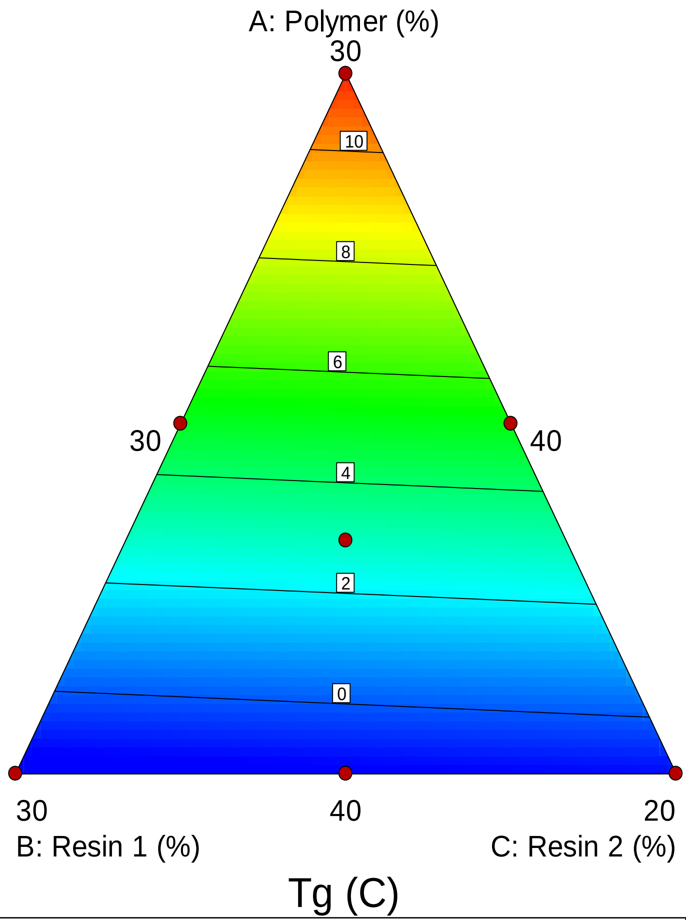



Figure 2. Contour Graph of Adhesive $T_g$, °C

On the other hand, SAFT ranges more broadly; varying from 72.4 to 81.0 °C. Figure 3, shows that there is a drop off of SAFT at 35% polymer.  Below 35% polymer, there is a steep decline in SAFT. At or above 35% polymer, however, is a shallow saddle with a narrow range of SAFT values of 79.1 to 81.7 °C. In any case, a minimum SAFT of 72°C is above typical shipping temperature of 60°C. Thus, the entire range should be sufficient for a general purpose PSA.

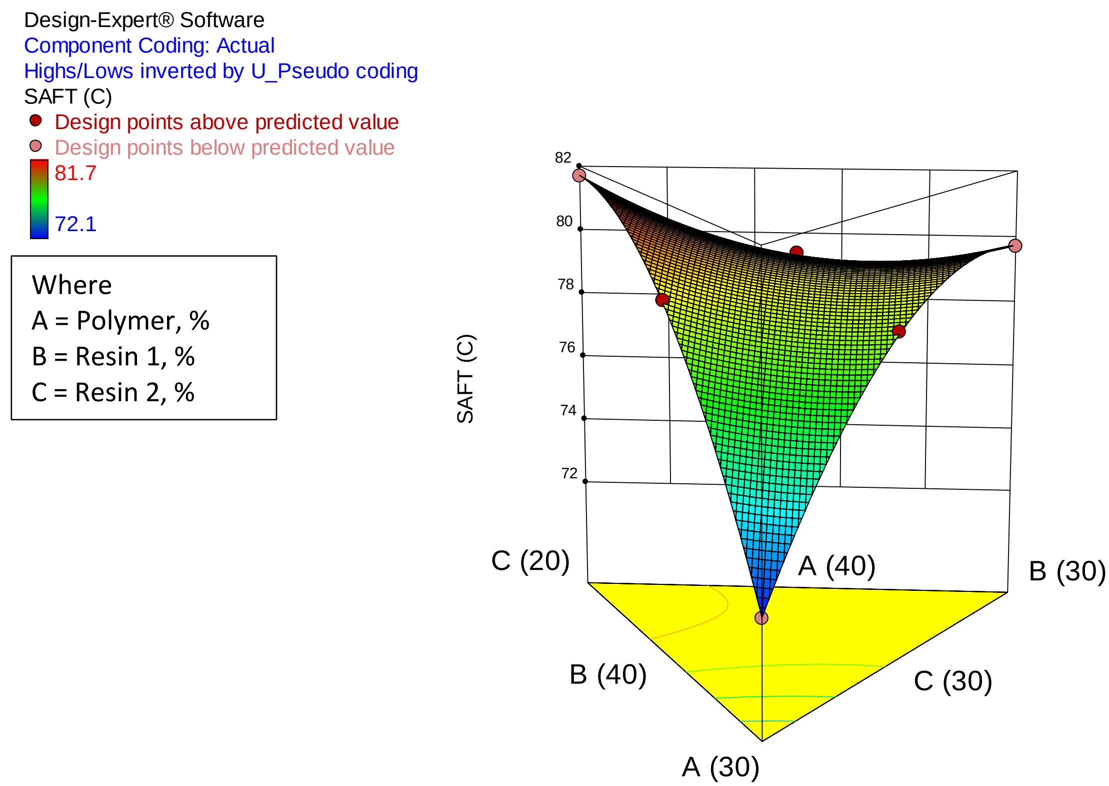



Figure 3. 3D Response Surface of SAFT, °C

Thus, the results indicate that it is possible to produce a general purpose PSA that is robust to variations in concentration of resin and polymer. Despite significant swings in concentrations of resins and polymer, there was limited variation in 180 Degree Peel, Loop Tack, and SAFT values. This robust resistance to variations in key raw material concentrations provides an advantage by simplifying the manufacturing process and reducing lot-to-lot variation.

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[1] Temperature sweep using 8mm parallel plates at a heating rate of 6° C per minute and a frequency of 10 rads/sec.
[2] In this case, an elastic modulus (G') of 20,000 to 200,000 Pa is desired.