WATER WHITENING RESISTANT PRESSURE SENSITIVE ADHESIVE FOR CLEAR LABEL APPLICATIONS

Donovan Lujan, Application Scientist, Arkema Coating Resins, Cary, NC

Clear Filmic Labels

Permanent filmic labels offer improved performance over paper labels such as superior durability (tear resistance, thermal stability and chemical resistance), flexibility over irregular shaped containers, and resistance to environmental conditions. Filmic labels can be segmented into a premium grade market where a clear or “no label look” is required. Clear filmic labels allow brands to differentiate their advertising strategies from the competition by highlighting the product rather than the packaging. Areas of application include personal care products such as shampoo/conditioner, beer/wine/spirits, and food packaging. Solvent-borne pressure sensitive adhesives have traditionally filled this role; however, the market has shifted to using waterborne adhesives due their improved environmental characteristics.

Clear Filmic Label PSA Performance Requirements

Pressure sensitive adhesives developed for clear filmic labels must meet four criteria of performance. First and most importantly the adhesive must be optically clear and be resistant to discoloration from ultraviolet radiation. Practically, the adhesive must be capable of being applied to face stock on high speed coaters. Additionally, adhesive must have a balanced adhesion profile (peel, tack and shear) to afford adhesion to a variety of substrates and resist delamination. Lastly, adhesives with end use applications where the label is exposed to aqueous environments must be resistant to water whitening, also known as “blushing”.

Water Whitening Mechanism

Conventional all acrylic water-borne pressure sensitive adhesives become opaque when exposed to aqueous environments. The proposed mechanism of water whitening is initiated when water penetrates into the adhesive coating via pores or defects present at the adhesive air interface. Water molecules then cluster into domains that grow large enough to facilitate a mismatch in refractive index between the water and the polymer film. This mismatch results in an increase in opacity within the adhesive film [1-3].

Variables known to impact water whitening include latex particle size, surfactant loading levels, surfactant package composition, and the addition of coating package additives (rheology modifiers and wetting agents) [4]. Larger latex particle sizes lead to less efficient film formation, which results in defects at the coating surface and allows water to penetrate the adhesive matrix easier. Surfactants are hypothesized to partition and concentrate during film formation, thus acting as pools for water once diffused into the adhesive. The surfactant composition (anionic vs. nonionic, extender length, and hydrophobe structure) and the incorporation of polymerizable moieties have shown to augment the compatibility and mobility of surfactants within adhesive films and impact the water whitening resistance performance. Lastly, coater packages have the ability to render adhesives with blush resistance more sensitive to aqueous environments due to the hydrophilic nature of the rheology modifiers and wetting agents.

Water Whitening Resistant PSA Development

The project for developing a water whitening resistant adhesive was initiated with developing a method to test adhesive blushing. The method employed throughout this study consisted of coating adhesive onto a 2 mil polyester film targeting a coat weight of 1.5 ± 0.1 g/100 in^2. Films were then placed in a 60°C oven for five minutes. The coated films were then left to rest at ambient temperature for 24 hours. A Lishing LS162 visible light transmission meter was then used to measure the visible light transmission of the coated film. The films were then cut into strips measuring one inch by five inches and submerged into ambient temperature water for 24 hours. After soaking the strips were shaken to remove excess water droplets and hung vertically. The visible light transmission was measured again and the percent change in visible light transmission calculated. Due to the fact that some films started to turn transparent quickly upon removal of water and potentially resulting in erroneous transmission readings, photos were taken after soaking to obtain qualitative visual observations.

An internal benchmarking study was conducted to determine the best innate water whitening resistant adhesive to be used as a starting template and control for development. Figure 1 shows that the starting prototype affords approximately an 88% improvement in visible light transmission post soaking versus a general purpose filmic (GPF) adhesive. Table 1 shows the latex properties of the starting prototype.

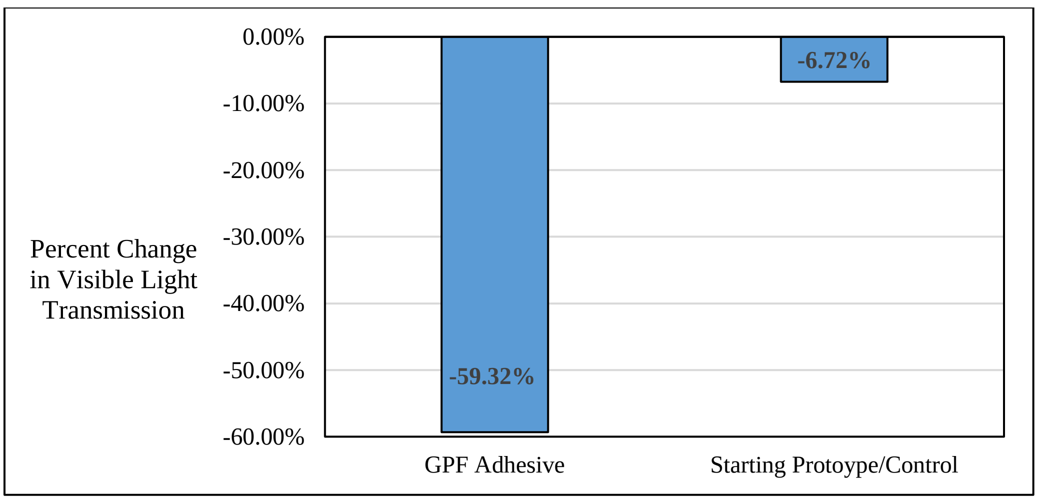

Figure 1. Water Whitening Resistance Testing Benchmark Results

Table 1. Starting Prototype/Control Latex Properties

Solids Content %	Viscosity (cps)	pH	Tg (°C)	Surface Tension (dynes/cm)	Particle Size (nm)
< 50%	<700	6.0	-40	31	<250

To further improve the water whitening resistance of the starting prototype, a synthetic strategy was employed to optimize film formation and minimize the formation and size of voids/ channels within the adhesive matrix. A design of experiment was carried to investigate two predictor variables that were hypothesized to impact water whitening resistance via this route. The first predictor variable was the incorporation of a functional monomer over three loading levels (low, medium and high). The second predictor variable investigated three augmentations to the emulsion polymerization process (modified process 1, modified process 2, and modified process 3).

Results

Figure 2 shows the qualitative results of incorporating functional monomer in a standard emulsion polymerization process at both low and high loading levels versus the control adhesive. Incorporation of the functional monomer, regardless of loading level, with the standard emulsion polymerization process resulted in no significant improvement in water whitening resistance compared to the control.

Figure 2. Pictures of the Results of Functional Monomer Incorporation with a Standard Emulsion Polymerization Process

Figure 3 shows the results of incorporating the aforementioned functional monomer over three loading levels and investigating three different emulsion polymerization processes. Figure 3 shows that when modified process 1 is implemented, all results exhibit decreased water whitening resistance regardless of functional monomer level. However, when modified process two and three are utilized with the high level of functional monomer the adhesive films appear transparent with no haze detected. Figure 4 shows that the two prototypes developed exhibit a greater than seventy five percent improvement in visible light transmission post soak compared to the control adhesive.

Figure 5 shows the results for the stepwise multiple linear regression model for the design of experiment. The model resulted in a large value for the coefficient of determination ($R^2 = 0.94$) and all predictor variables were determined to be statistically significant at a p-value of 0.05. Since the model suggests that a large amount of the variance observed within the data is predicted, the prediction expression was used to identify the optimal levels by setting the response variable (percent change in visible light transmission) to 0 %. The optimal predictor variable levels were determined to be modified process three and a functional monomer level that was increased by 40 weight percent above the high loading level.

Figure 6 shows the qualitative results of the predicted model prototype versus the prototype developed from the design of experiment. The predictive model prototype is completely opaque post soaking. The results indicate that there are extraneous variables not accounted for by the predictive model.

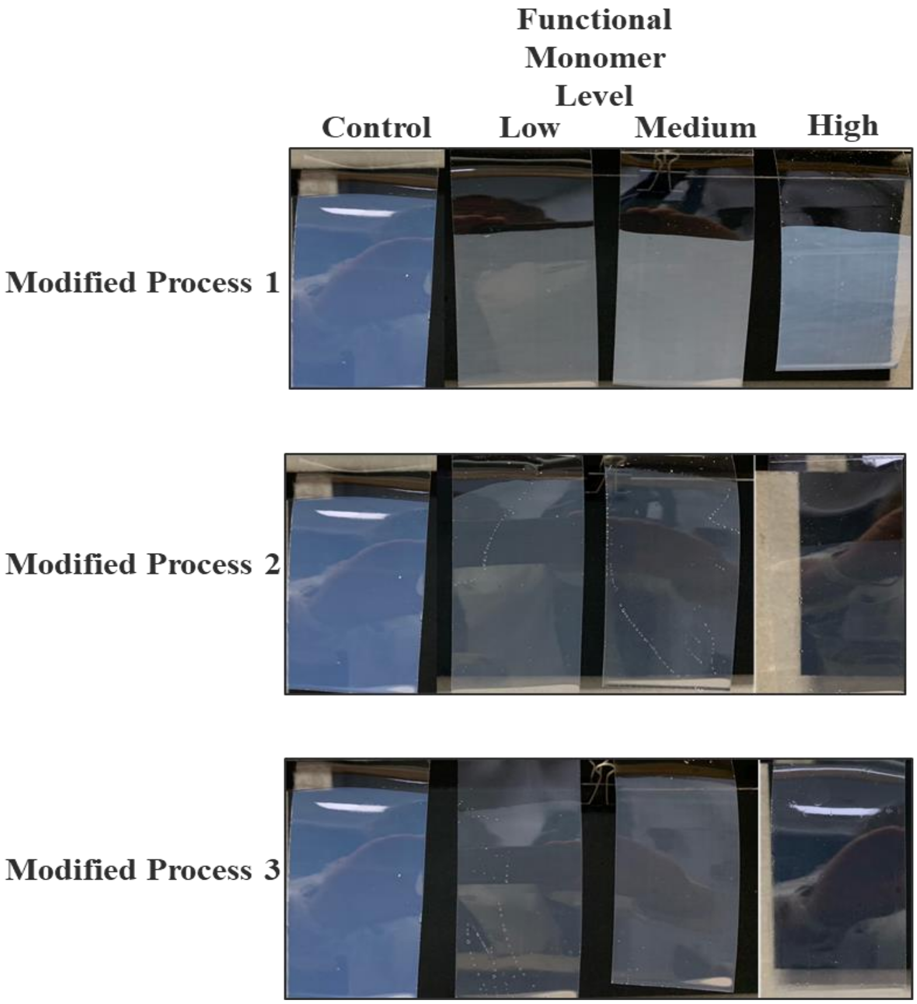

Figure 3. Pictures of Water Whitening Testing Results of Functional Monomer Incorporation with Modified Emulsion Polymerization Processes

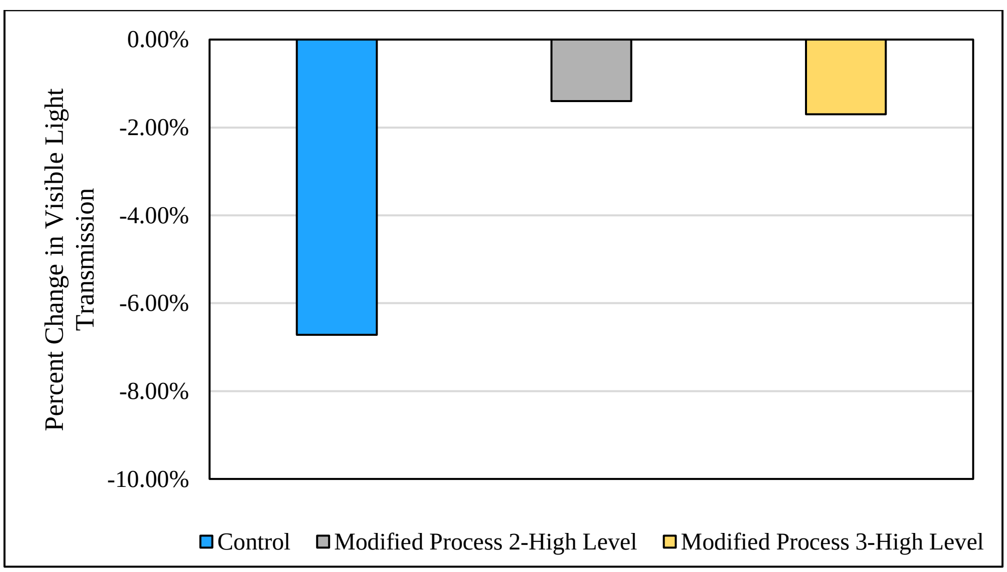

Figure 4. Water Whitening Resistance Testing of Modified Emulsion Polymerization Process and High Functional Monomer Loading Results

Summary of Fit

RSquare	0.940046
RSquare Adj	0.904073
Root Mean Square Error	1.838719
Mean of Response	-8.97778
Observations (or Sum Wgts)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	265.05111	88.3504	26.1323
Error	5	16.90444	3.3809	Prob > F
C. Total	8	281.95556		0.0018*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-8.7	0.618355	-14.07	<.0001*
Monomer Loading Level	2.5	0.737128	3.39	0.0194*
Modified Process 2	-7.088889	0.866781	-8.18	0.0004*
Modified Process 3	3.4777778	0.866781	4.01	0.0102*

Figure 5. Multiple Linear Regression Model Results from Design of Experiment Investigating the Use of Functional Monomer and Modified Emulsion Polymerization Processes

Modified Process 3 & High Monomer Level | **Predictive Model Prototype**

Figure 6. Pictures of Water Whitening Testing Results and Comparison of Predictive Model Prototypes

Figures 7 through 9 compare the 30 minute dwell 180° peel adhesion, loop tack and shear values over stainless steel (SS), glass, high density polyethylene (HDPE) and low density polyethylene (LDPE) for the developed prototype compared to the control adhesive. The peel adhesion and loop tack values were poorer with the augmentations to the prototype, while the shear values increased almost four-fold as compared to the control adhesive. The process changes to the prototype also resulted in an increase in surface tension of 9 dynes per centimeter (Table 2). This resulted in defects to occur, such as dewets, when attempting to coat the prototype adhesive on silicone release liner. In an attempt to reduce the surface tension, one weight percent of a common industrial wetting agent was added to the prototype adhesive.

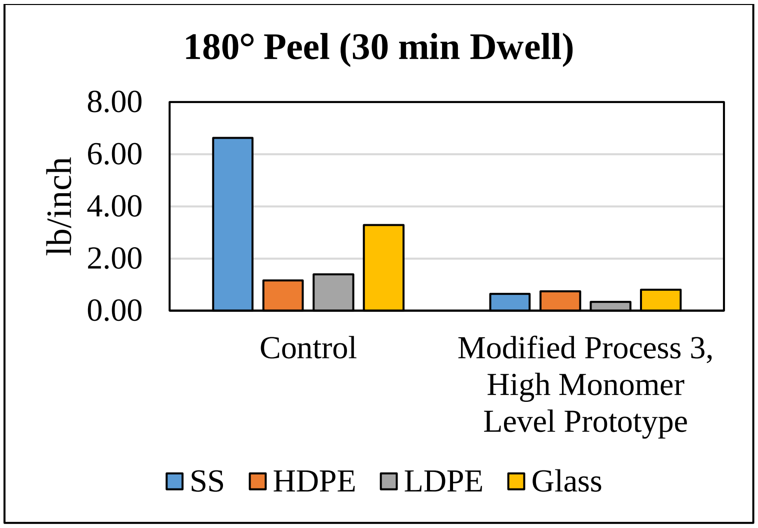

Figure 7. 180° Peel Values Comparison of Prototype and Control Adhesives

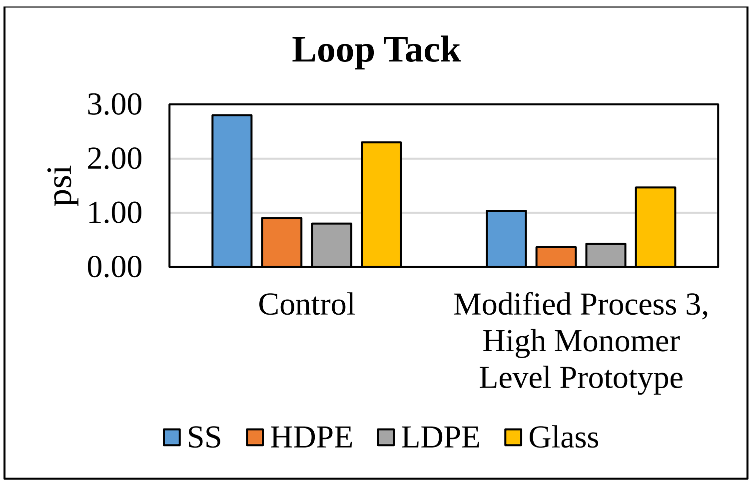

Figure 8. Loop Tack Values Comparison of Prototype and Control Adhesives

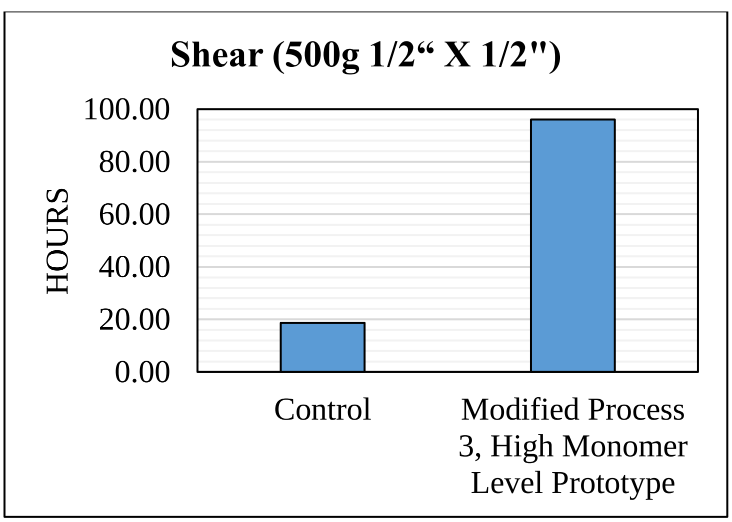

Figure 9. Shear Values Comparison of Prototype and Control Adhesives

Table 2. Surface Tension Comparison of Prototype, Formulated Prototype and control Adhesives

Sample	Surface Tension (dynes/cm)
Control	31
Prototype	40
Formulated Prototype	32

Figures 10 and 11 show the water whitening testing results of the formulated prototype and control adhesives. The addition of the wetting agent negatively impacted the water whitening resistance of both the control and prototype adhesives; however, the performance of the formulated prototype is still superior to the neat control adhesive.

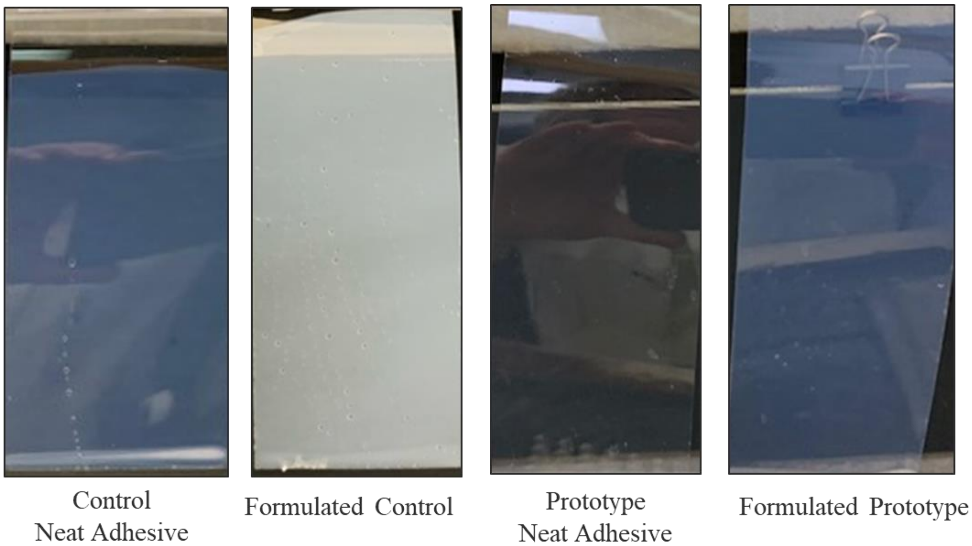

Figure 10. Pictures of Water Whitening Testing Results and Comparison of Neat and Formulated Adhesive Prototype

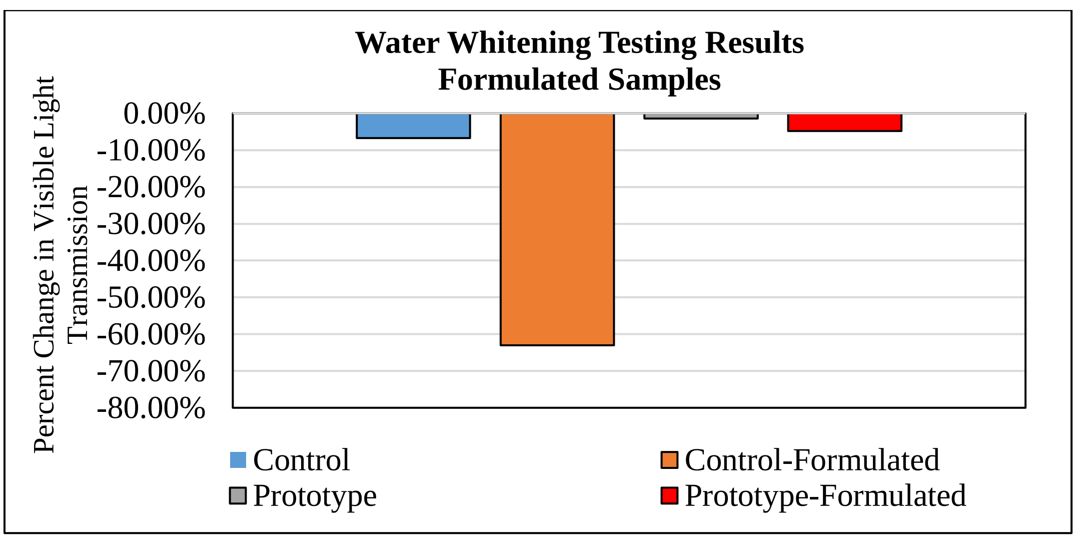

Figure 11. Water Whitening Testing Results and Comparison of Neat and Formulated Adhesive Prototype

The test method employed for water whitening testing in this study is strenuous, as the entire adhesive film is exposed to water; however, in real world applications the adhesive is sandwiched between a substrate and the filmic face stock. To test performance of the developed prototype in a more applicable condition, a bottle soak test method was developed. A 2 mil polyester film was coated out with adhesive, targeting a coat weight of 1.5 ± 0.1 g/100 in^2. Coated films were placed in a 60°C oven for five minutes. The coated films were then left to rest at ambient temperature for one hour. Adhesive films were then rolled onto glass beverage bottles and left for one hour at ambient temperature. The glass bottles were then filled with water and submerged in an ice water bath in a cooler and left for six hours.

Figure 12 shows the glass bottle ice water soak results of both the formulated control and prototype adhesives. The formulated adhesive shows blushing occurring along the edges of the label, along with significant blushing where the label appears to have partially delaminated from the glass substrate. The formulated prototype adhesive shows no indications of blushing or delamination from the glass bottle.

Figure 12. Glass Bottle Ice Water Bath Results

Conclusions

The results of the design of experiment resulted in an adhesive that exhibits water whitening resistance by incorporating the use of a functional monomer and modifying the emulsion polymerization process. The amount of functional monomer and the specific augmentation to the emulsion polymerization process were found to be critical to the prototype's water whitening resistance performance. Additionally, the prototype exhibits superior water whitening resistance when formulated with wetting agent compared to the control adhesive. The peel adhesion and loop tack were decreased and the shear strength increased with the implementation of the design factors; however, the adhesive properties of the prototype still allows for suitable adhesion over a range of industrial relevant substrates.

Appendix

All samples were prepared by direct-coating the emulsion onto 2-mil polyester, followed by covering the sample with a release liner. Unless otherwise specified, the dried sample had a coat weight of 1.5 ± 0.1 g/100 in^2. All data reported are the average of three individual tests.

Peel tests were performed following PSTC-101 Test Method A in which a strip of tape is applied to a standard test panel with controlled pressure. The tape is peeled from the panel at 180˚ angle at a specified rate, during which time the force required to effect peel is measured. A 30 minute dwell time was employed on stainless steel, glass, LDPE and HDPE substrates.

Loop tack was measured using the PSTC-16 Test Method B which involves allowing a loop of pressure sensitive adhesive with its backing to be brought into controlled contact with a 24 mm x 24 mm (one square inch) surface of stainless steel, with the only force applied being the weight of the pressure sensitive article itself. The pressure sensitive article is then removed from the substrate, with the force to remove the pressure sensitive article from the adhered measured by a recording instrument.

Shear adhesion test were conducted following PSTC-107 in which a strip of tape is applied to a standard steel panel under controlled roll down. The panel is mounted vertically, a standard mass is attached to the free end of the tape and the time to failure is determined. Instead of the standard 23 ^oC and 50% R.H.

References

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Acknowledgements

The author would like to recognize the contributions of Sonia Chalmers, Keith Andes and Patrick Shulack.