Technical Research

Comparison of Rheological Methods to Predict Gel Coat Sag Resistance

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INTRODUCTION

Gel coats are specialty coatings for fiber reinforced plastics (FRP) that are typically spray-applied to a wet coating thickness of 20-25 mils. They must be sag-resistant when sprayed on vertical or inclined surfaces. A property known as the "thix (thixotropic) index" is commonly used as an estimate of the sag resistance of a resin or gel coat. The thix index is the ratio of viscosities measured using a standard rotational viscometer at two different speeds, typically differing by a factor of ten, e.g., viscosity at 6 rpm/viscosity at 60 rpm. Thixotropy refers to a time-dependent decrease in viscosity with a constant shear rate. Viscosity measurements of thixotropic fluids will not be reproducible unless the shear history and shear times are duplicated exactly. Most gel coat systems are pseudoplastic, not thixotropic or shear thinning, meaning they decrease in viscosity with increasing shear rate in a controlled and reversible manner. Therefore the viscosity ratio measured should be called a shear thinning index.

The thix index, albeit a misnomer, has been a useful guide for decades, but may not be the best quantifier for flow properties of gel coats. A more sophisticated rheological characterization may provide a better measure. D.H. Durham presented a study on rheological properties of filled DCPD laminating resin systems. In it, typical thix index measurements were determined using standard Brookfield dial reading viscometers fitted with disc spindles. The data was compared with drainage/sag properties. While there was a general correlation between the standard thix index and sag resistance, the individual effectiveness of various rheology modifiers could be missed unless they were screened with more sophisticated methods such as using a controlled stress rheometer using cone and plate geometry.

This study seeks to extend the work of Durham to gel coat systems. A Brookfield R/S rheometer was used to obtain a viscosity profile of specifically formulated gel coat systems as a function of shear rate. One was a clear/unfilled system and the other a white/highly filled system. Various levels of rheology modifiers were added to each system to provide a range of viscosities to study.
The resultant rheological curves were characterized by fitting the curve to a power law, $y = kx^n$. This equation is comparable to the standard power law describing most rheological behavior, $\tau = kD^n$, where τ is the shear stress, k is the consistency index, D is the shear rate, and n is the flow index. Previous studies have related the flow index to high shear flow properties and the consistency index to low shear flow properties.

The gel coat systems were evaluated for sag resistance using a Leneta drawdown bar with varying gradations from 5 - 50 mils. Values obtained from the viscosity profile using the rheometer and viscometer were correlated to the sag resistance determinations. Our intent was to determine whether standard thix index, flow index, or consistency index values provide a better assessment of sag resistance for common gel coats. We also wanted to determine whether the rheometer or viscometer provided a more accurate measure of sag resistance.

EXPERIMENTAL

The rheology of an Interplastic Corporation CoREZYN® clear and a white gel coat were adjusted with different levels of fumed silica. The rheological characterization was conducted using a standard Brookfield LVF Dial Reading Viscometer with a #4 spindle and a Brookfield R/S rheometer using cone and plate geometry. The cone type was #645.

Viscosity curves were constructed by plotting viscosity versus revolutions per minute (rpm). The rpm correlates directly with shear rate, so the values for the flow index (n) and consistency index (k) will be different from the rigorously derived n and k from the standard power law expression for viscosity. They will, however, track the same way so direct correlations with sag resistance values can still be analyzed.

Gel coat formulas were prepared and allowed to equilibriate at ambient conditions for at least three days before conducting measurements.

To take the sag measurements, a film of gel coat was drawn down using a Leneta Anti-Sag Meter. The meter has various slots with identical widths but gradated heights in mils, resulting in beads of material at various heights on a panel. When placed in an upright position, the material will tend to sag, especially the thicker beads. The mil value of the first bead that does not sag and flow into a lower bead provides the anti-sag index or sag value.

RESULTS

Low Filler Content Gel Coats

Resin rich or low filler content gel coats are also called clear gel coats because they are used as clear coatings in the marine, transportation and cast marble industries. Their viscosity is adjusted by the amount of rheological additive, such as hydrophyllic fumed silica, incorporated into their formulation. A CoREZYN clear gel coat was adjusted to various viscosities, and the resultant rheological profiles determined using the Brookfield R/S Rheometer and a Brookfield LVF Dial Reading Viscometer. The viscosity profiles of the various formulations are depicted in Figures 1 and 2.The standard exponential-like decay exhibited in Figures 1 and 2 allows curves to be fitted to a power law equation. All curves exhibited an R*square value of 0.98 or better when fitted to a simple power law equation, indicating a very good correlation. Summaries of the rheological values for flow index (exponent) and consistency index (constant) derived from the power law equation for the generated curves are listed in Table 1.

The flow index (exponent) parameter decreases as the viscosity increases. The consistency index (constant) parameter, as well as the sag value increases as the viscosity increases. A practical correlation can be made between these rheological values and a measurement of sagging.

Sagging can be quantified using a Leneta Anti-Sag Bar. More viscous materials tend to give higher anti-sag indices or sag values. A plot of exponent versus sag value is given in Figure 3. It shows that even though different exponential values are obtained depending on whether a viscometer or rheometer were used, the trend can be predicted with the same amount of accuracy based on the R-squared value (linear correlation coefficient) for the line through the points.

A similar correlation can be made between Leneta Sag Values and the consistency index or the constant, k. The k values differ greatly between the data from the dial reading viscometer and the R/S rheometer, so their correlations are plotted separately in Figures 4 and 5, respectively.

This can be compared with a plot of the sag value versus thix index shown in Figure 6, which also provides a reasonably good correlation.

Overall, the correlation between sag value and various rheological parameters is comparable for these clear gel coat systems. The correlation between the sag value and the power law constant, or consistency factor, give consistently higher values, especially for the consistency factor derived from the dial reading LVF viscometer.

High Filler Content Gel Coats

High filler content gel coats can be used in a variety of applications such as tub and shower, architectural and transportation. They typically are used for white or pastel colors and consist of talc and titanium dioxide fillers that can comprise up to 30% of the formulation. The high filler load, especially the high density titanium dioxide pigment, makes these gel coats much more prone to sagging than their clear counterparts.
As with the clear gel coats, their viscosity is typically adjusted by the amount of hydrophyllic fumed silica incorporated into the formulation. Additionally, a second type of silica, namely precipitated silica, is used in filled systems. The precipitated silica boosts viscosity while allowing less use of the expensive fumed silica. A standard white CoREZYN gel coat was adjusted to various viscosities and their resultant rheological profiles determined using a R/S Brookfield Rheometer and a Brookfield LV Dial Reading Viscometer. Their viscosity profiles are illustrated in Figures 7 and 8.

The standard exponential-like decay exhibited in Figures 7 and 8 allows curves to again be fitted to a power law equation. All curves exhibited an R*square value of 0.98 or better when fitted to a simple power law equation, indicating this model is a very good fit. Summaries of the rheological values for flow index (exponent) and consistency index (constant) derived from the power law equation for the generated curves are compared with the sag values in Table 2.

The exponent and consistency index values vary in the same way as the clear gel coats, whether the rheometric curve is generated from a dial reading viscometer or an R/S rheometer. Exponents become more negative with increasing viscosity, while the constants become more positive. Sag studies were conducted as described previously with the clear gel coat formulations. Lower anti-sag index values were attained due to the higher density of the filled formulations compared with the data from the clear formulations. The correlation of the anti-sag index with the conventional thix index (6/60 rpm) is graphed in Figure 9. The correlation of the anti-sag index with exponents is shown in Figure 10. The correlation of sag value with the power law constants for the LVF viscometer and the R/S rheometer are shown in Figures 11 and 12, respectively.

The "thix index" actually gave a slightly better correlation with sag resistance than curves generated from fitting the exponents of the power law for the rheological curves. The values for the constants of the power law for the rheological curves, however, gave a much better correlation, especially the constant derived from the standard Brookfield LVF viscometer.

CONCLUSIONS

All correlations were quite good giving correlation coefficient values around 0.9. Linear correlations were conducted for ease of description and summarized in Table 3. Surprisingly, the correlation between the standard thix index and sag value was as good as between power law exponent and sag value. This was true regardless of whether the exponent was derived from the simple curve constructed from three measurements with the dial reading viscometer or from the more extensive data obtained with the R/S rheometer using cone and plate geometry. This exponent is related to the flow index which describes high shear flow properties. Thus, this exponent value or the rigorously derived flow index (plot of viscosity versus shear rate) can be used as an indicator of sag resistance with the same level of predictability as the standard thix index.

An especially high correlation was found between the power law constant and the sag value. The highest correlation at 0.98 linear correlation coefficient resulted from data obtained with the standard Brookfield LVF dial-reading viscometer. The constant, or consistency index, relates to low shear flow properties such as slow material movement or sagging. This indicates it is best to determine a viscosity curve (viscosity vs. viscometer rpm) with at least three points so a power law curve can be derived. The resultant power law constant, or consistency index, will provide the most predictable indicator of sag resistance; much better than the current two-point thix index method.

REFERENCE

This work is based on the original technical paper entitled "Assessment of Thix Index to Predict Sag Resistance in Gel Coats - Is There a Better Rheological Method?" by Peter Gottschalk, Ph.D., CCT, Technical Director, Gel Coat and Colorant Group, Interplastic Corporation. It is available through the American Composites Manufacturing Association (ACMA).

Figure 1: Viscosity vs. RPM for a Clear Gel Coat Series
Using Brookfield LV Dial Reading Viscometer - Spindle #4

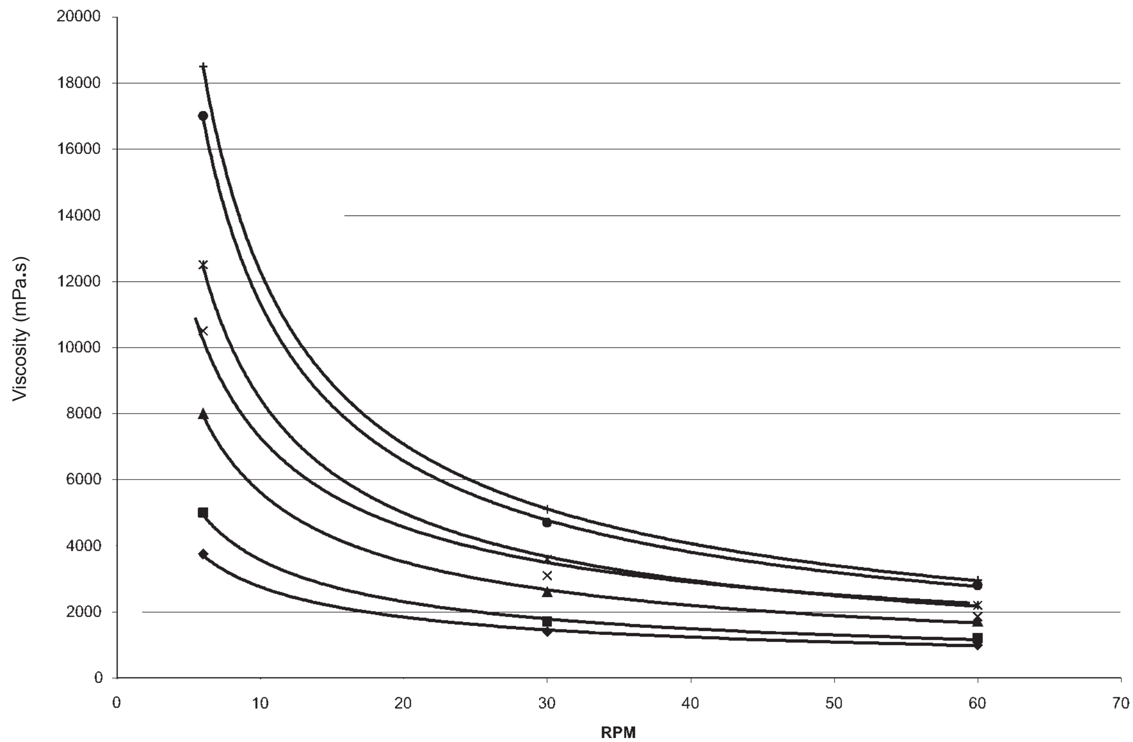

Figure 2: Rheological Profiles of Clear Gel Coat Series
Using the Brookfield R/S Rheometer

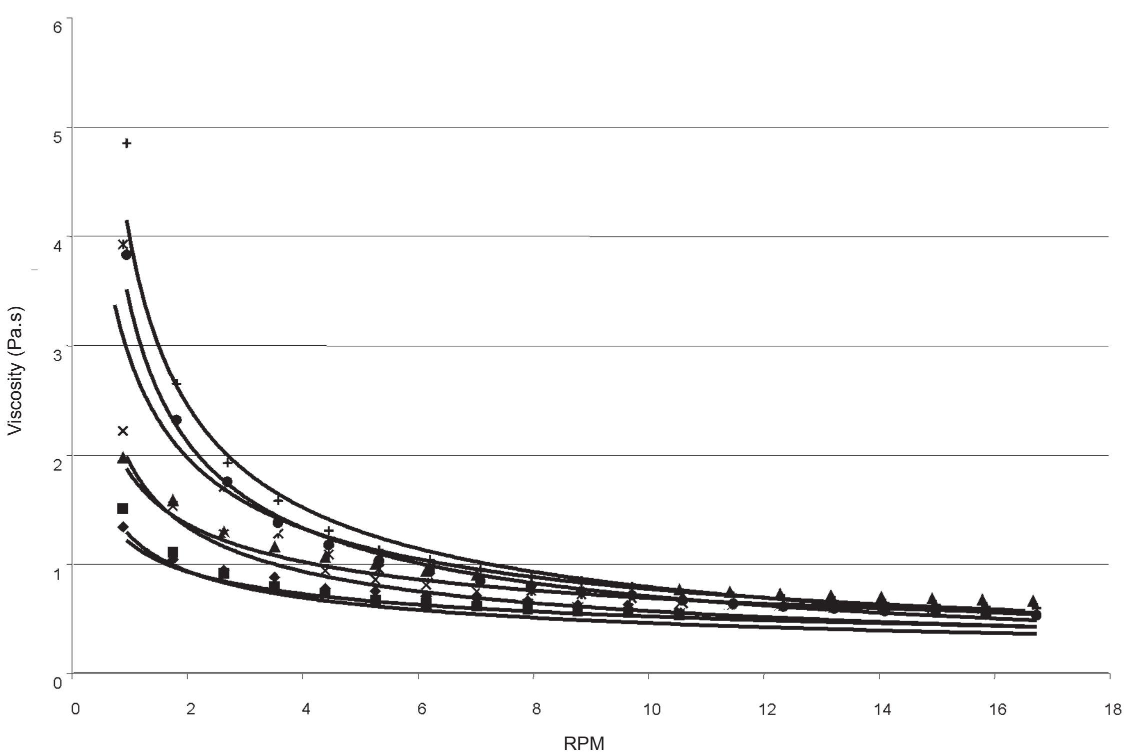

Table 1: Exponents From Power Law Equations for Viscosity Curves

Sag Value	14	25	30	35	40	50	60
Exponent (LVF)	-0.581	-0.628	-0.677	-0.755	-0.758	-0.786	-0.798
Exponent (R/S)	-0.31	-0.375	-0.377	-0.468	-0.601	-0.617	-0.643
Constant (LVF) x 1000	10.492	15.186	26.706	40.546	48.316	69.187	77.205
Constant (R/S)	1.266	1.327	1.881	1.962	3.124	3.338	3.911

Figure 3: Exponent vs. Leneta Sag Value

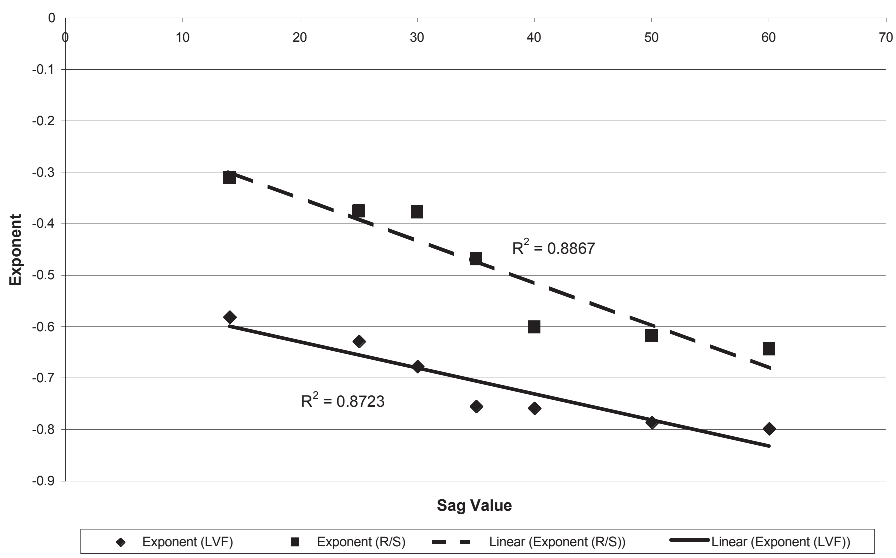

Figure 4: Power Law Constant From Dial Reading LVF Viscometer vs. Leneta Sag Value

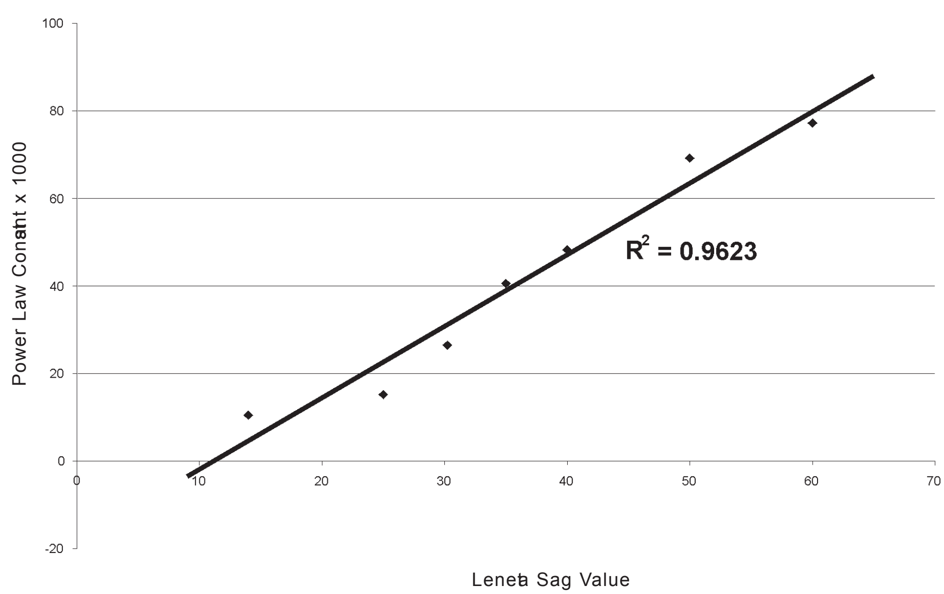

Figure 5: Power Law Constant From R/S rheometer vs. Leneta Sag Value

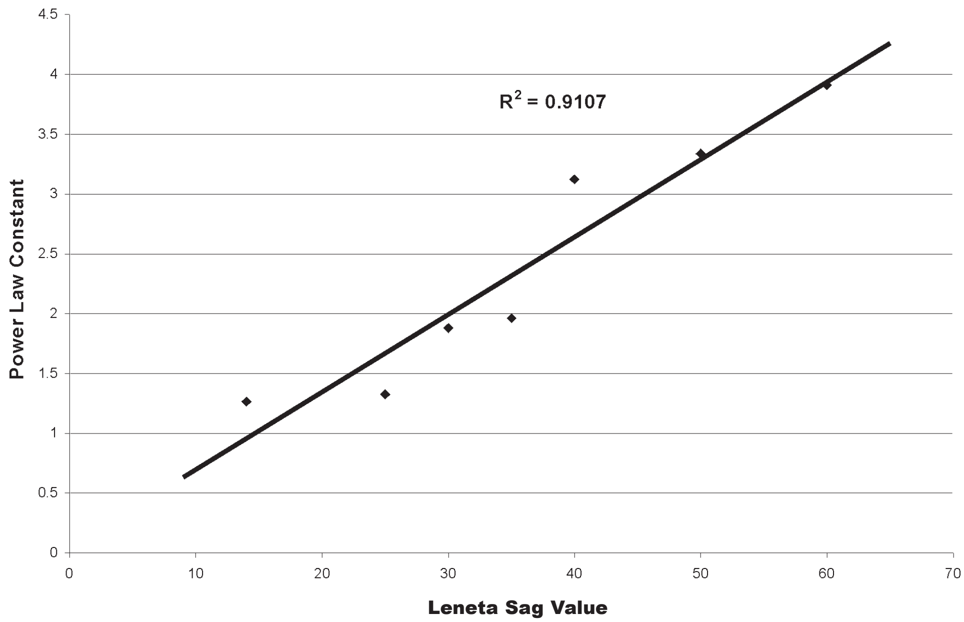

Figure 6: Thix Index from Dial Reading LVF Viscometer vs. Leneta Sag Value

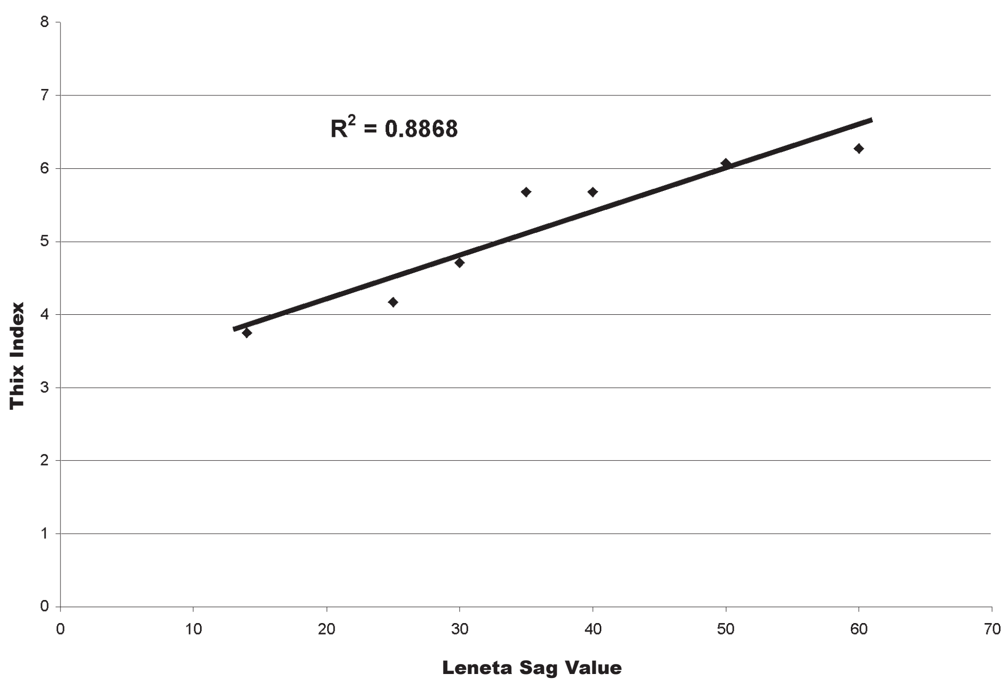

Figure 7: Viscosity vs. RPM for White Gel Coat Series
Using Brookfield LV Dial Reading Viscometer - Spindle #4

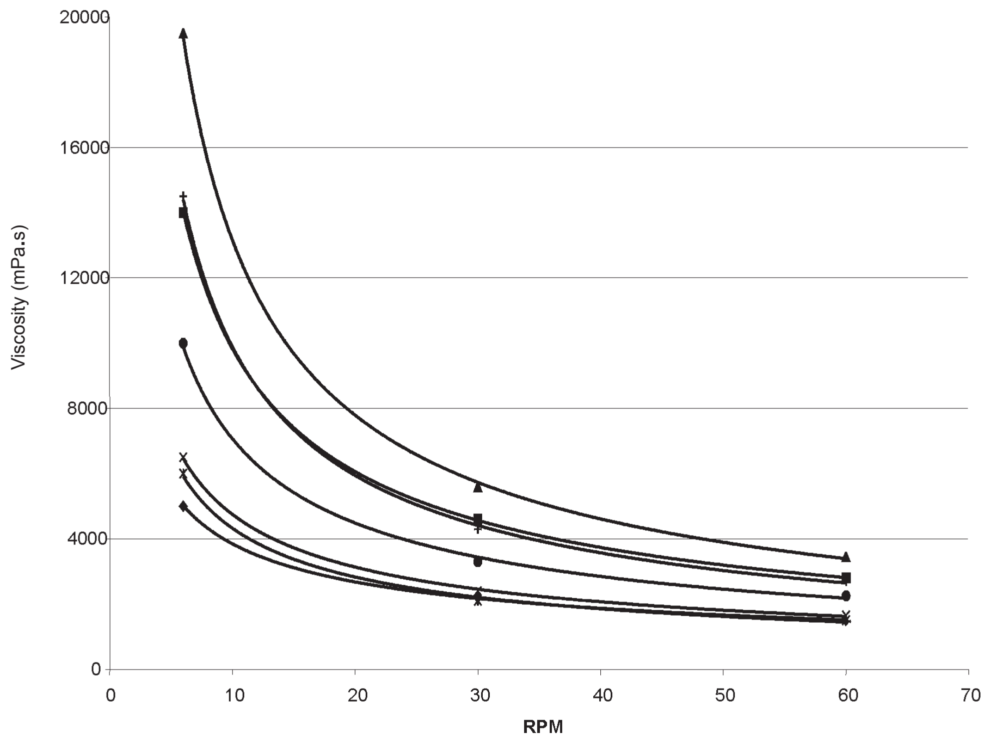

Figure 8: Rheological Profiles of White Gel Coat Series Using Brookfield R/S Rheometer

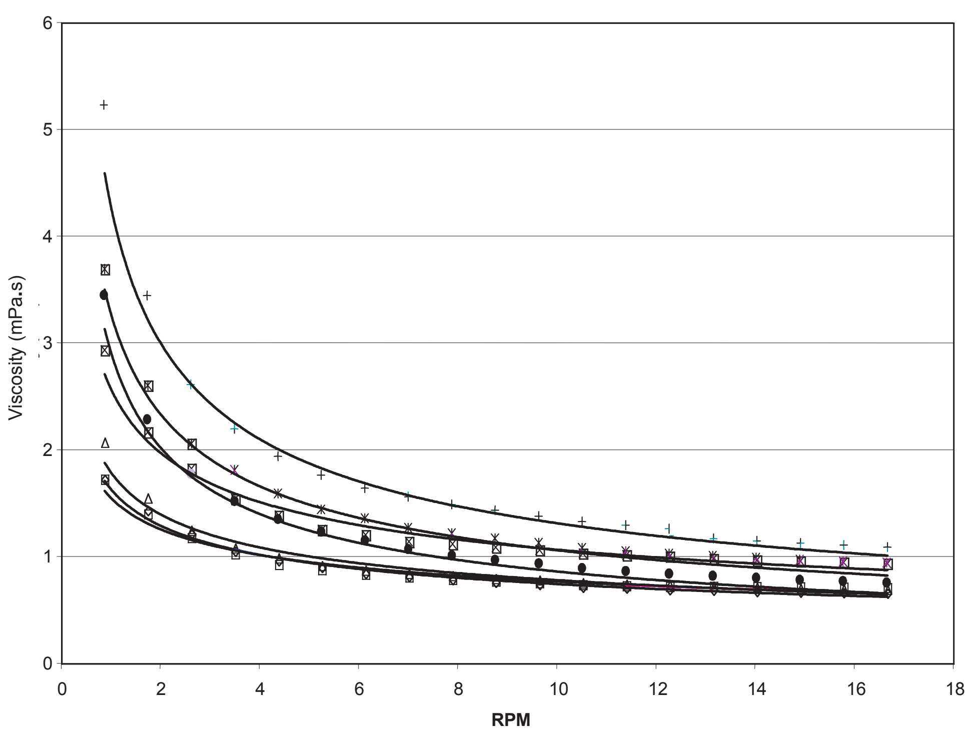

Table 2: Exponents from Power Law Equations for Viscosity Curves from White Gel Coat Series

Sag Value	14	14	16	20	25	30	35
Exponent (LVF)	-0.76	-0.61	-0.52	-0.66	-0.70	-0.73	-0.60
Exponent (R/S)	-0.34	-0.36	-0.31	-0.38	-0.49	-0.53	-0.52
Constant (LVF)	12.76	17.65	18.90	31.93	48.98	53.64	75.07
Constant (R/S)	1.64	1.79	1.55	2.58	3.29	2.92	4.30

Figure 9: Conventional Thix Index Correlated with Leneta Anti-Sag Index for White Gel Coat Series

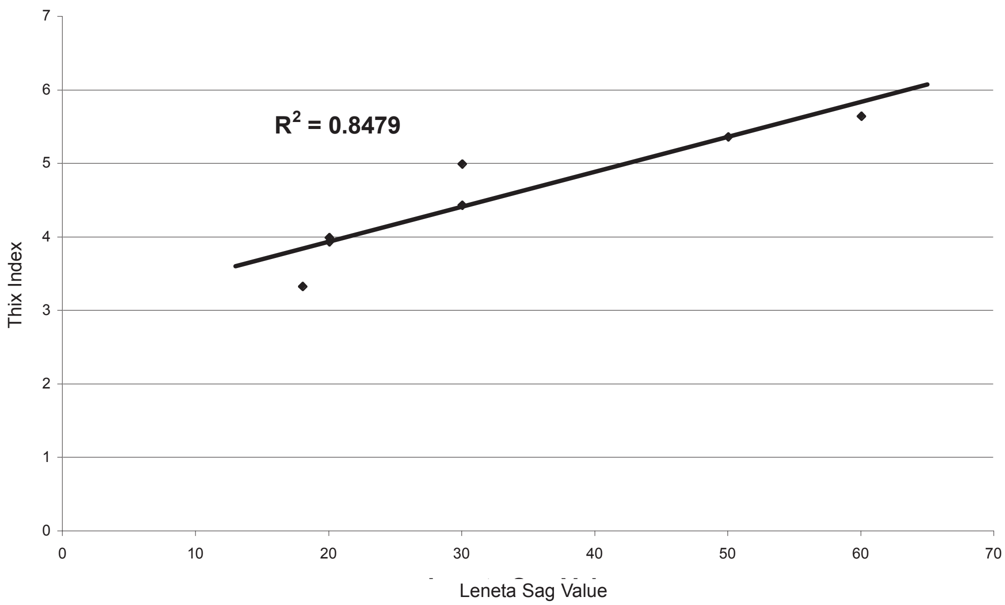

Figure 10: Exponent Values of Viscosity Curves Correlated with Leneta Sag Values

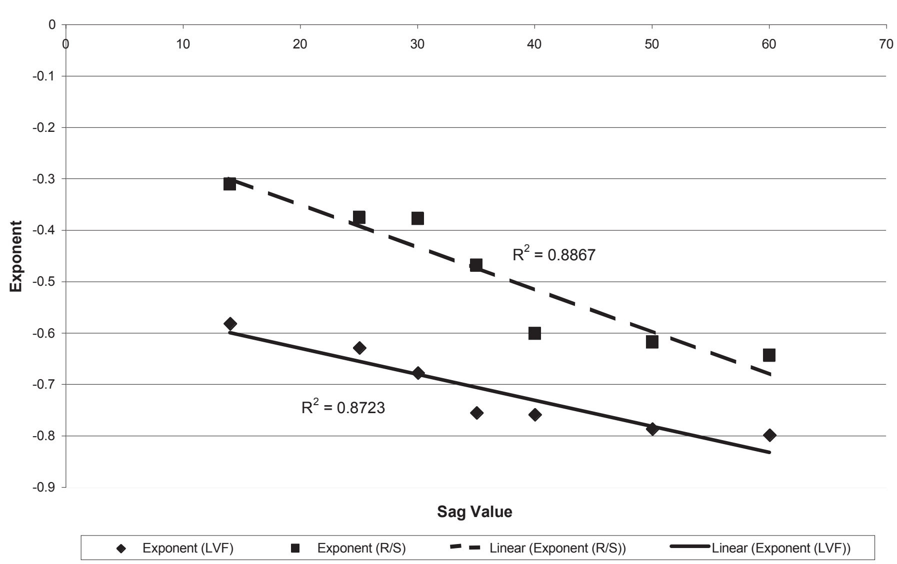

Figure 11: Consistency Index Constant from the LVF Viscometer Correlated with Leneta Sag Values

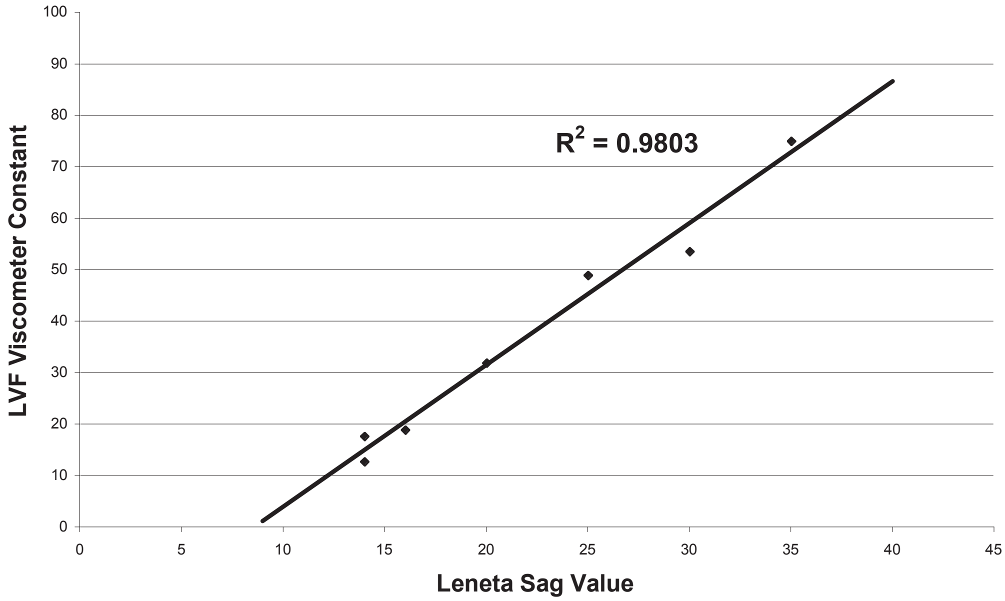

Figure 12: Consistency Index Constant from the R/S Rheometer Correlated with Leneta Sag Values

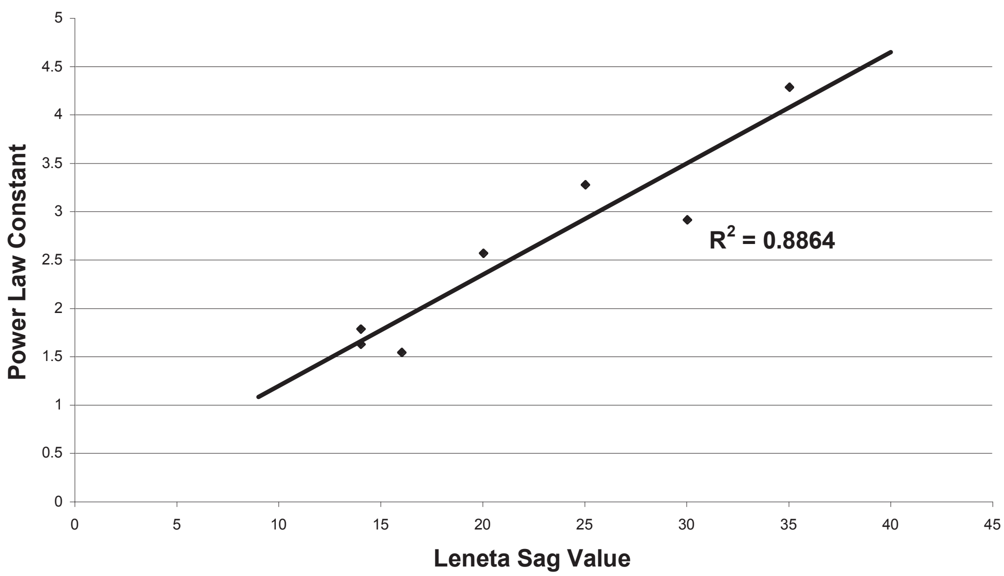

Table 3: Summary of Linear Correlation Coefficients for Leneta Anti-Sag Index Values

Leneta Anti-Sag Index Correlated to:	Linear Correlation Coefficient (R^2) for Clear Gel Coats	Linear Correlation Coefficient (R^2) for White Gel Coats
Exponent from Brookfield LVF Viscometer	0.87	0.93
Exponent from Brookfield R/S Rheometer	0.89	0.87
Constant from Brookfield LVF Viscometer	0.96	0.98
Constant from Brookfield R/S Rheometer	0.91	0.89
Thix Index (Brookfield LVF @ 6/60 rpm)	0.89	0.85

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