SEAL-THROUGH-CONTAMINATION AND "CAULKABILITY"
AN EVALUATION OF SEALANTS' ABILITY TO ENCAPSULATE CONTAMINANTS IN THE SEAL AREA

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ABSTRACT

Channel leaks are a recurring problem in flexible packaging that lead to product waste and premature spoilage. A common cause of channel leaks is product contamination in the seals. If the sealant fails to flow around or fully encapsulate a solid contaminant in the seal, an air channel may form through the seal resulting in package integrity loss. A perceived strategy for minimizing contaminant-channel leakers is to choose sealants with better "caulkability" or seal-through-contamination ability. While these concepts seem intuitive, there is no consensus on what fundamental properties makes a sealant "caulkable" and no standard test for quantifying this attribute.

The purpose of this study was to evaluate the ability of various sealants to encapsulate contaminants and minimize the channel or void area around a contaminant. A practical test was developed to measure a sealant's ability to encapsulate a simulated contaminate in multilayer, heat-sealed films. The results suggest that sealants with the optimal combination of zero shear viscosity (ZSV), hot tack strength window (HTS) and other sealant properties are required to fully encapsulate seal area contaminants. The optimal combination depends on the contaminate size, converting conditions and multilayer film structure.

INTRODUCTION

When designing multilayer films for flexible packaging applications, the sealant resin choice is generally considered the most critical material decision. The sealant must be strong enough to maintain package integrity throughout the converting process and distribution chain yet be soft and pliable enough to weld at low temperatures. A common seal problem in flexible packaging is channel leakers caused by product contamination in the seal area. Properly designed seal jaws can push low-viscosity liquids out of the seal area insuring good sealant to sealant contact. Solid or particulate contaminants however are less mobile and may stay trapped within the seal area creating an incomplete seal around the particle. If the incomplete sealed area extends beyond the length of the seal, a channel leak may result causing barrier and/or package integrity loss. Small leaks may not be caught at the packaging line and only become evident later in the distribution cycle.

To minimize contaminant-channel leaks, resin suppliers recommend using sealants which offer better "caulkability". This term suggests that certain sealants will flow around and fully encapsulate contaminants more effectively to minimize voids in the seal area. However, it is unclear what specific sealant properties will provide caulkability. Also, there is no standard test method for measuring caulkability.

A reliable test method that compares how effectively different sealants will encapsulate contaminants in the seal would help package designers identify the best sealant for mitigating channel leakers. A quantitative test method would also enable resin suppliers to identify polymer properties that improve a sealant's ability to effectively encapsulate and thus design sealants which are less susceptible to channel leaks.

The objectives of these studies were to:

- Develop a reliable test method for quantifying a sealant ability to effectively encapsulate a contaminant in the seal.
- Compare and evaluate the ability of different sealants to encapsulate contaminants and flow into void areas.
- Identify fundamental sealants properties that help maintain package integrity when contaminants or voids are present in the seal area

VIRTUAL CHANNEL DIAMETER (Hole Size) TEST

A new quantitative test method was developed for estimating the virtual channel diameter or void area around a simulated contaminant in a flexible package seal. The new method incorporated procedures outlined in two existing tests: ASTM F2095: Standard Test Methods for Pressure Decay Leak test for Flexible Packages, and ASTM F1929: Test Method for Detecting Leaks in Nonporous Medical Packaging by Dye Penetration.

ASTM F2095 is a quantitative test that involves inflating a converted package to a fixed pressure and measuring the rate of pressure loss. The package leak rate is determined by equation 1.

$$Q = (\Delta P * V) / \Delta t \tag{1}$$

Where Q is the package leak rate, ΔP is the loss of interior package pressure, V is the initial volume of the package and Δt is the time elapsed during test pressure readings.

Assuming all leakage occurs through a single channel leak, the diameter of this virtual channel can be calculated using equation 2 based on Hagen-Poiseullie's Law

$$d = 2 * [Q * 8 * N * l / \Pi * (P - \Delta P / 2)]^{1/4}$$

Where P is the final interior package pressure, ΔP is the initial differential pressure between package interior and atmosphere, N is the Dynamic Air Viscosity, and l is the channel length.

ASTM F1929 is a non-quantitative test method for detecting the presence or absence of a channel leak in sealed packages by injecting a liquid dye into a sealed pouch. The precision of this test method was determined in an inter-laboratory study which involved creating 50 μm channel in the seal using Tungsten wire.

In this study, we combined the approach of sealing through a wire introduced in ASTM F1929 and using pressure decay to estimate the void or channel area around the wire. By sealing through a thin gauge copper wire that extends from the outside to the inside of a flexible pouch and measuring pressure decay, we were able to estimate the void or channel area around the wire. Sealants which had low pressure decay had narrower channel leaks and would be expected to encapsulate contaminants in the seal area more effectively.

TEST FILMS AND MATERIALS

A standard 89 μm gauge, 9-layer film with the below structure was used for all tests:

Table 1: COEX Film structure

Layer	A	B	C	D	E	F	G	H	I
Material	Various	sLL	sLL	sLL+ 20% tie concentrate	Nylon	sLL+20% tie	sLL	sLL+20% tie	Nylon
Ratio (%)	11.2%	11.1	11.1	11.1	11.1	11.1	11.1	11.1	11.1
Layer Designation	Seal	-	sLL	Tie Layer	Core	Tie	sLL	Tie Layer	Skin

The films were produced on a 9-Layer blown film line produced by Brampton Engineering with a 160 mm diameter die. All process and run conditions such as temperatures, blow up ratios and output rates were held constant for all the films produced. The specific materials used in layers B through I of the coex structure are summarized in Table 2.

Table 2: Materials used in coex film structure

Material Designation	Specific Grade	Supplier	Comments
Nylon	CAPRON® C40 L07	BASF	Nylon 6,6-6 copolymer
Tie	BYNEL® 41E710	Dupont	Anhydride Modified LLDPE Tie Concentrate
sLL	SURPASS® FPs016-C	NOVA Chemicals	Octene sLLDPE. Solution Reactor

Over 30 commercial or experimental sealant resins were evaluated in the standard film structure. The sealants were chosen to provide a broad range of viscosities, densities and molecular weight distributions. Table 3 summarizes some of the sealants evaluated.

The melt index, density, 1% secant modulus, Hot-tack, stress exponent and zero shear viscosity of each sealant was measured to for correlation analyses. Melt Index was measured as outlined in ASTM D 1238 at 190^{o} C and 2.16 kg load, density according to ASTM D 792, 1% secant modulus according to ASTM D992, hot tack according to ASTM F 1921 and zero shear viscosity according to ASTM D 4287 at 190^{o} C. Stress exponent is a NOVA Chemicals rheological test generated from the slope of the log shear rate versus log shear stress plot. Stress exponent generally corresponds with the polymer's molecular weight distribution and also provides an indication of a polymer's flow properties at higher shear rates.

The test films were hand-converted into pouches by folding a 46 cm by 15 cm film along the width and impulse sealing both sides of film to create a 23 cm long by 15 cm wide pouch. Pouches were filled with 5 foamed packing peanuts to simulate product. Cylindrical copper wire; 254 μm and 356 μm gauge and 2.5 cm long, was placed in the top opening and the pouches were then sealed using a 5 mm width heat sealer. The virtual channel diameter or hole-size was then determined using the procedure described in the previous section. A control pouch without a wire contaminant in the seal was also produced for each film structure to determine burst strength and residual leakage levels.

Table 3: Selected Sealant Evaluated in 9-Layer Films

Sealant	Code	Melt Index	Density	Zero Shear Viscosity 190C (MPa)
SURPASS® FPs117-C	sLL-1.0-917	1	0.917	8908
SURPASS® FPs016-C	sLL-0.6-916	0.65	0.917	13830
SURPASS® EX-FPs116-CX01	sLL-0.8-917	0.82	0.917	12500
SCLAIR® FP112-A	zLL-1.0-912	0.9	0.912	12210
SCLAIR® FP120-C	zLL-1.0-920	1	0.92	10410
NOVAPOL® PF-0118-F	Butene - LL	1	0.918	9679
NOVA Experimental 1	Experimental 1	1.58	0.916	5235
NOVA Experimental 2	Experimental 2	1.06	0.917	8400
NOVA Experimental 3	Experimental 3	1	0.924	8130
SURLYN® 1601-2	Ionomer	1.75	0.94	3300
ELVAX® 3135X	EVA	0.35	0.93	17021

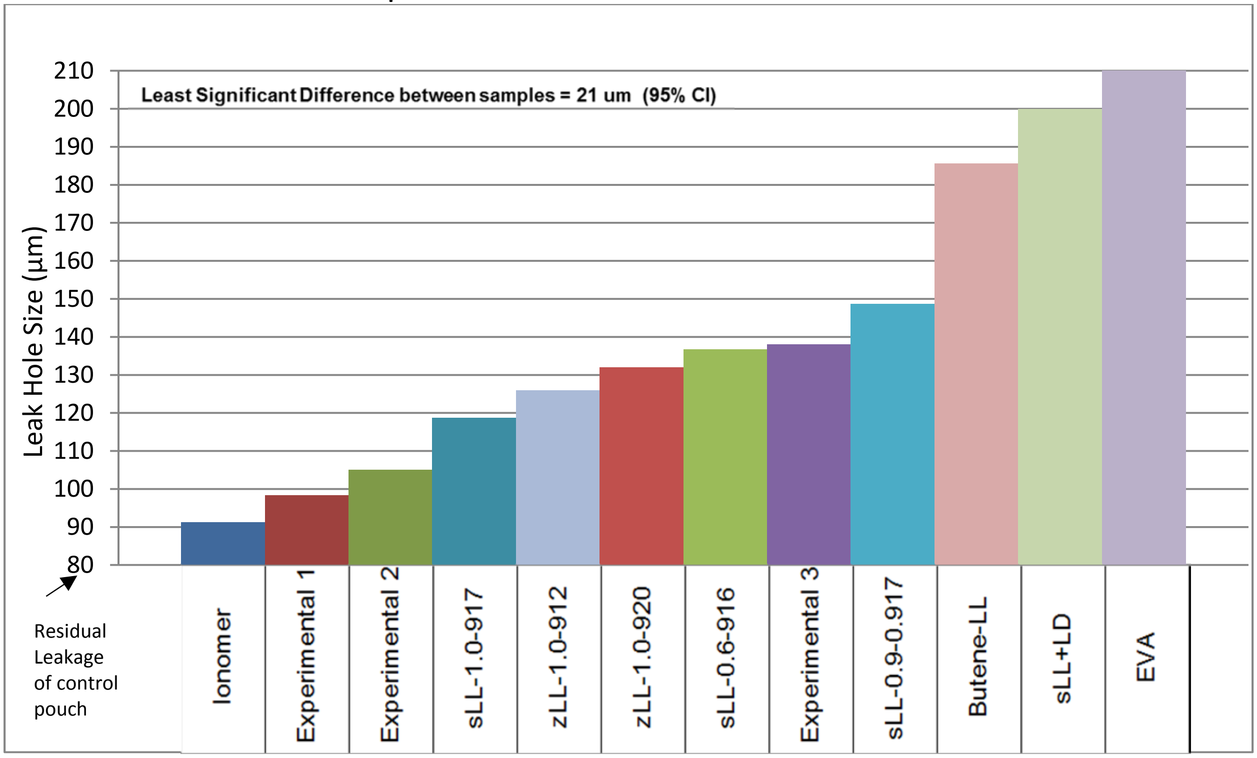
RESULTS AND DISCUSSION

Selected results and correlations from the study are presented in charts 14 through 18 of the presentation. All pouches containing LLDPE sealants had burst strengths between 62 and 76 kilopascals (kPa) and thus the (50%) pressure used for the pressure decay test was between 31 and 38 kPa. Films containing softer sealants such as ionomer and EVA had lower burst strengths so these results may not be directly comparable to the LLDPE films. All control pouches without wire contaminant in the top seal had similar residual leakage which corresponded to a virtual channel diameter or hole size of approximately 80 μm. Most of the leakage occurred at the bottom end-folds at the junction where the impulse seal met the fold line. At the other limit, hole sizes greater than 400 μm correlated with total pressure loss inside the package during the test.

Hole Size Results for Different Sealants and Test Conditions

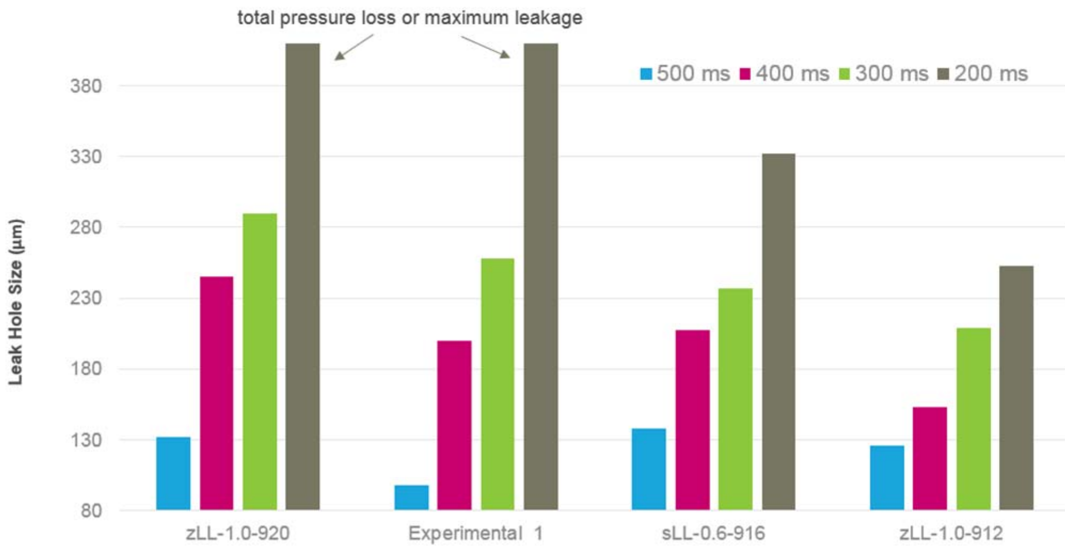
The mean hole size of pouches containing 15 different sealants sealed with a 254 μm wire at 130^{o} C for 500 milliseconds is presented in Figure 1. Fisher's Least Significant Difference between mean sample hole sizes is 21 μm, i.e. sample hole sizes which differ by more than 21 μm are considered to be significantly different from each other at the 95% confidence interval.

Figure 1: Hole Size for Various Sealants in 9-Layer Coex Film 254 micron gauge wire contaminant. Test Conditions: 500 millisecond dwell time 130^{o} C seal temperature



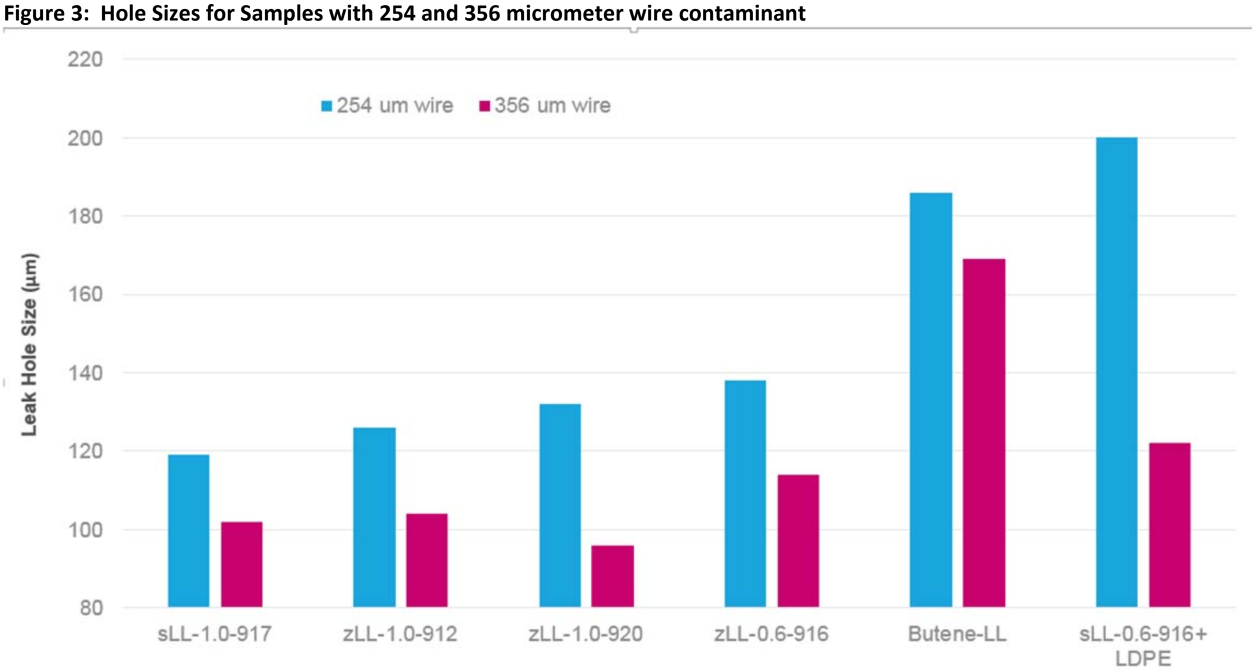
The effect of variable dwell time for selected sealants sealed with a 254 μm wire at 130^{o} C is shown in Figure 2. As expected, the hole size increased with decreasing dwell times for all sealant types. The effect was more pronounced however with higher density sealants which have higher seal initiation temperatures. At these shorter dwell times, there may be insufficient heat at the seal interface to melt or soften the higher density sealants. If the sealant does not reach softening temperatures it will retain high viscosity and not flow around or encapsulate the wire.

Figure 2: Hole Size vs. Seal Dwell time for 254 micron wire contaminant. Seal Conditions: 130° C seal temperature with variable dwell times



A comparison between identical samples sealed through the thinner 254 μm and thicker 356 μm gauge wire is presented in Figure 3. Hole sizes were consistently smaller in sealed pouches containing the thicker wire contaminate. This unexpected decrease might be explained by the higher pressures at the apexes of the thicker wire. The concentrated pressures at these top and bottom points could force the sealant to flow laterally from the top around the wire thus encapsulating it more effectively with less void area.

Figure 3: Hole Sizes for Samples with 254 and 356 micrometer wire contaminant



Overall, there was less differentiation between samples containing 356 μm gauge wire and thus fewer samples showed significant statistical differences using the t-test. All analyses correlating hole size with basic sealant properties were completed using data from the 254 μm gauge wire samples.

Effect of Sealant Polymer Properties on Hole Size

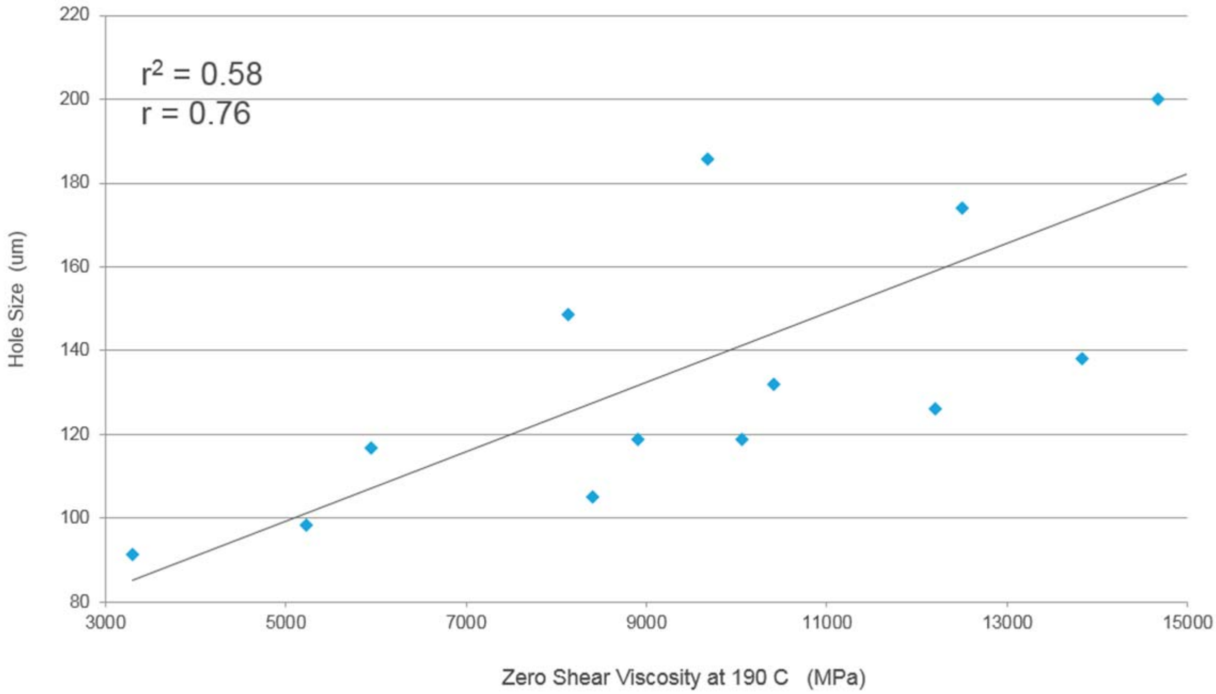
Pearson correlation coefficients between hole sizes and different sealant parameters are presented in Figure 4. The sealant's Zero Shear Viscosity (ZSV) at 190° C had the strongest overall correlation with hole size. The positive correlation is not surprising; sealants with lower ZSV or higher melt indices would be expected to flow around and fully encapsulate a contaminant better than more viscous sealants. It should be noted that the range of sealant melt indices used for this study was relatively narrow and typical for blown film coextrusions: 0.35 to 2.20 g/10 minutes. These results should not be extrapolated to lower viscosity sealants. In other studies, low viscosity sealants or sealants with rapid viscous temperature decay were more prone to excessive flow or "squeeze-out" which led to weak seal strength under pressure [2,4,].

Table 4: Pearson Correlation Coefficients between Sealant Parameters and Hole Size

Sealant Parameter	Test Method	Pearson Correlation between factor and hole size 254 μm wire, 500 ms dwell, 130 C
Zero Shear Viscosity	ASTM D 4287 (190° C)	0.76
Hot Tack Strength Window*	ASTM F1921	- 0.58
Density	ASTM D 792	0.52
Stress Exponent**	NOVA Chemicals Method	0.38
1% Secant Modulus (MD)	ASTM D 882	- 0.36

Figure 4 shows hole size versus ZSV for selected sealants. In general, sealants with a greater hot tack strength window fell below the linear regression line or had smaller hole size than would be predicted by ZSV alone and vice-versa. The hot-tack strength window is an estimate of the area under the hot tack curve for the coex films from hot-tack initiation to the seal-bar temperature of 130° C. This window provided better correlations with hole size which suggests it is a better predictor of encapsulation performance than the hot tack initiation temperature or hot tack strength at 130° C. Coextruded films or laminates are susceptible to weak seals around a contaminant or fold since the stiffer backing layers will release stored energy and spring-back away from the fold immediately after seal bar pressure is released. A sealant's hot tack strength will resist this spring-back force effectively keeping the hole size smaller.

Figure 4: Hole Size Versus Sealant Zero Shear Viscosity for Octene LL Sealants with 0.65 to 2.0 MI



Other factors which correlated with hole size were the sealant's density and stress exponent, and MD secant modulus. Stress Exponent is a NOVA Chemicals rheological test that correlates with a polymer's molecular weight distribution. Sealants with smaller stress exponents would be expected to flow around and encapsulate a contaminant better since they transition from a high-viscosity semi-solid to a low viscosity liquid quickly and over a narrow temperature range. The negative correlation of hole size with sealant secant modulus was unexpected but may relate to the sealant's ability to resist elongation after the seal is formed. The high pressures inside the formed pouch could lead to sealant stretching which effectively increases the hole diameter.

Proposed Mechanism for Effective Sealant Encapsulation

The above findings suggest that effective encapsulation of contaminants in the seal area is influenced by the sealant's rheological, hot-tack and solid state physical properties. Rheological properties are important while the sealant is in the melt phase, hot tack properties are important while the sealant is transitioning from the melt to solid phase and physical properties are important after the sealant has cooled to the solid state. This suggests effective encapsulation of contaminants occurs in three distinct phases. Stage 1 occurs during the sealing process and requires the sealant to effectively melt and flow around the contaminant. Stage 2 occurs between the time the seal jaws are released and the polymer solidifies and requires a sealant with sufficient hot-tack strength to resist spring back forces of stiffer adjacent layers. Stage 3 occurs after the seal is cooled and requires a sealant that maintains physical integrity as pressures or forces are exerted on the hole.

CONCLUSIONS

A sealant's ability to completely encapsulate a contaminant without resulting in a channel leak through the seal is influenced by multiple properties of the sealant and structure. The results of this study suggests that three important sealant properties are zero shear viscosity, the breadth of the hot tack strength window and the secant modulus or ability to resist elongation under stress. To eliminate channel leaks caused by contaminants in the seal area, it is important that all three of these properties are met simultaneously or sequentially.

The new method for measuring the virtual channel diameter around a sealed wire or filament is a simple and effective test that can be implemented in many laboratories or production areas. This new test can be used to compare sealants with respect to their ability to encapsulate contaminants and should help film manufacturers choose sealants and design coex films that provide better package integrity.

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TRADEMARKS