PHYSICAL PROPERTY MODELING OF MULTILAYER HEAVY DUTY SACK FILMS

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ABSTRACT

The heavy duty sack (HDS) market covers a broad range of applications, each with unique attributes required of the film. Multilayer film extrusion is used to achieve the demanding balance of properties required. To develop resins for this market it is necessary to understand how resins perform in complex structures. A design-of-experiment (DOE) approach was adopted to study the effects of film structure and processing conditions on finished properties of HDS films. Models developed based on the experimental program were used to predict properties for a range of end use applications. The models show how resin selection and processing conditions can be used to optimize properties in three layer films.

INTRODUCTION

To develop new resins for the flexible packaging market, it is standard practice to compare products based on data generated from a monolayer film. With today's trend to multilayer film extrusion for performance films there is a need to go beyond monolayer film properties and understand how a resin behaves in actual, coextruded structures. When NOVA Chemicals developed a new high performance single-site grade for the heavy duty sack market, the product evaluation was designed to look beyond standard monolayer data sheet properties to the use of the material in real world coex structures.

The heavy duty sack market covers a broad range of packaged products, each requiring unique attributes of the film. For highly compressed products such as insulation bags, stiffness may be valued over some toughness properties. For materials packaged in high dust environments, seal through contamination or hot tack properties may be critical. There is also an ongoing challenge to do more with less. Film producers are being asked to make stiffer, thinner films while maintaining or even improving toughness properties. To this end there has been a trend in the HDS market to move from monolayer films to three layers, with five on the horizon.

For multilayer film extrusion many HDS film producers will work with a handful of stock resins to produce a wide range of finished films. NOVA Chemicals has a suite of resin offerings dedicated to the production of heavy duty sacks and has worked to develop a high-performance grade specifically designed for the toughness demands of this market. The purpose of this study is to look at this enhanced product offering in film structures covering a range of process conditions and film designs for the heavy duty sack market. A project to look at all possible resin combinations in coextruded films would be overwhelming in terms of extruder and lab testing time. For this reason a design of experiment (DOE) approach was employed. Predictive models were then developed to examine the effect of selected parameters on finished properties. These models were used to predict film properties as well as to explore new ways of expanding the typical toughness stiffness balance.

BACKGROUND

The study was designed to look at the effects of film composition as well as processing conditions on finished properties. Previous studies have shown that single site octene resins have unique properties and when placed in the skins with butene or hexene resins in the core there can be a synergistic effect on properties[1]. Placement of LDPE has also been shown to have a significant impact on finished properties. Two main objectives of this work were to identify how 3-layer film properties rely on the choice of LLDPE resin in the core and to explore if and how the presence of LDPE in the skin layer contributes to the overall performance of the multi-layer film. The current study looks at three layer coextrusion with the intent to expand to five layer structures in the future.

Process conditions were studied by looking at different blow up ratios and film thicknesses. To study the effect of orientation on film properties, parameters such as process time and draw down ratio were calculated and included in the modeling.

NOVA has developed a new high performance grade for HDS structures with exceptional toughness and seal properties. The experimental design was set up with this product as the main component for all films. The core layer was used to look at alternate LLDPE choices to determine the effect on final film properties. The core resins were ranked and evaluated based on their inherent dart impact strength. The dart impact strength was chosen because it is one of the key requirements of the film. It reflects both the resin architecture [2] and solid morphology as a function of the film blowing process [3]. For example, the dart impact strength can be modeled as function of resin specifications (density, MI and stress exponent, TREF weight content of 25 - 60°C fractions, and a corrected Deborah number) and a process parameter (machine-direction strain rate)[4]. The MD tear of some solution octene LLDPE films resins can be modeled in a similar way[5]. Moreover, a change in dart with architecture and process frequently coincides with tear and optical properties.

The use of LDPE in film extrusion to improve bubble stability is commonplace but is known to have a negative effect on properties. While LDPE use has been reduced, it is still an ingredient in most formulations. This study looked at the effect of placement of a low level of fractional melt index high performance LDPE on final properties.

Stiffness, or modulus, is a key property for heavy duty sack films. The stiffness properties are typically modified through use of a higher density blend resin. This resin is normally placed in the core so as to maintain seal properties in the skins. A HDPE product with good toughness properties was selected for this study and used to adjust the density in the finished films. Films were formulated to a target density with the level of HDPE adjusted to reach the target. The final film density range for this study was selected based on typical end use modulus requirements.

EXPERIMENTS AND ANALYSES

The resins explored as the major component in the core are listed in Table 1 along with their typical monolayer film properties. The single site octene resin was used in the skins for all films. The HDPE and LDPE blend resin properties are also provided in Table 1.

Table 1. Resins Used in the Study and Some Basic Properties

Resin Type	Resin Code	Melt Index g/10min	Density g/cm^3	Dart g/mil	MD Tear g/mil	1% Sec. MD MPa
Single Site Octene LLDPE	sLLDPE	0.85	0.921	475	275	165
Z-N octene LLDPE	o-LLD	0.80	0.926	156	295	234
Z-N hexene LLDPE	h-LLD	0.80	0.926	130	250	270
Z-N butene LLDPE	b-LLD	0.80	0.921	110	100	210
HDPE	HD	0.72	0.962	34	21	1330
LDPE	LD	0.25	0.920	160	320	190

DOE DESIGN AND ANALYSES

A DOE approach was used to reduce the level of testing that would be required to study the broad range of parameters of interest.

The experimental design was modified from a 4-factor, 3-level Taguchi L18, which in principle includes one qualitative factor of 2 levels and 4 quantitative factors each with 3 levels. The absence/presence of the LDPE in the skin layer is taken as the qualitative factor. The dart ranking of the four LLDPE resins is taken as the first quantitative factor and is changed to a 4-level instead of 3 for the standard Taguchi L18 design. The dart ranking is based on the logarithm of the resin dart value, with the highest and the lowest designated as +1 and -1; the other two are ranked using the corresponding interpolated values on the logarithmic scale. The values are shown in Table 2. The other 3 quantitative factors are film thickness, blow-up-ratio (BUR), and film density. The factors and the values for each factor are:

1. 4% LDPE in all layers or 7% LDPE in the core only. (The overall amount of LDPE in all films is constant).
2. Core composition, 4-level: ranked by the dart of the LLDPE resin in the core.
3. Film thickness, 3-level: 1, 3, and 6 mil.
4. Blow-Up-Ratio (BUR), 3-level: 1.7:1, 2.0:1, and 2.5:1.
5. Film density, 3-level: 0.923, 0.926, and 0.929.

Table 2. Regularized resin properties for the DOE design

Resin	Dart
sLLDPE	1.000
o-LLD	-0.522
h-LLD	-0.772
b-LLD	-1.000

A standard Taguchi L18 design would require 18 runs with no replicates. Adding one level to the first quantitative factor requires another 9 runs for the orthogonal arrangement of the last three factors. An extra 6 runs were included in addition to validate the models. A total of 33 films were therefore produced to be tested for all physical properties.

FILM CONFIGURATION AND BLOWING

For the film extrusion, samples were run on a Brampton Engineering three layer co-extrusion line with a standard temperature profile, 4" die and 50 mil die gap. The output rate was kept constant at 100 lbs. per hour. All processing conditions were recorded including Frost line height and line speed. To control the film processing condition for desired BUR and film thickness, other parameters had to be adjusted. For this reason process time and DDR were used as additional modeling factors to look at orientation effects.

The process time is defined as[6]:

$$t_p = \frac{0.06 \times FLH}{S\left(1 - \frac{T_f \times BUR}{T_o}\right)} \times \ln\left(\frac{T_o}{T_f \times BUR}\right)$$

Where t_p = Process Time (sec)

FLH = Frost line height (mm)

S = Line speed (m/min)

BUR = Blow-up ratio

T_f = Film thickness (microns)

T_o = Die gap (microns)

The Draw down ratio (DDR) is defined as:

$$DDR = \frac{T_0}{T_f \times BUR}$$

RESULTS AND DISCUSSION

Building the Statistical Model

A total dataset of 33 runs was available to analyze as a historical design using the DOE® PRO XL2010 software. Standard film physical properties were measured on each film. Models were first developed based on a subset of 27 runs and then validated with the remaining 6 runs.

The results from the analyses were cross-checked using the coefficient of Variation and the least total standard deviation. It was found that the 27 runs allow the models to be built for each of the film properties measured with moderately good confidence. The calculated coefficient of determination (R^2) is about 0.8 or above for all parameters. The P(2 tail) (probability that the term is relevant for the property being modeled) are all close to 0, while the tolerance (probability that the predicted coefficient is as shown) is statistically acceptable as well. The general match between the predicted and measured values for the models based on the original 27 runs is good as shown in Figure 1.

In Figure 2 the additional six validation runs have been examined with the models and the predicted values remain in good agreement with the experimental measurements. The predictive models also show physical properties to be dependent on the expected inputs based on an understanding of material science. The dart was found to be a function of mainly the BUR and finished film density. The MD tear relates to density, the process time and the core resin choice. Puncture resistance correlates with the film density and DDR in addition to the film composition. Haze turned out to be a strong function of the LDPE location and film thickness in addition to the dependence on film processing parameters.

The developed models can therefore be used with some confidence to predict properties for films made within the range of parameters studied.

Figure 1. Comparison between predicted and measured values for the 27 runs to build the models

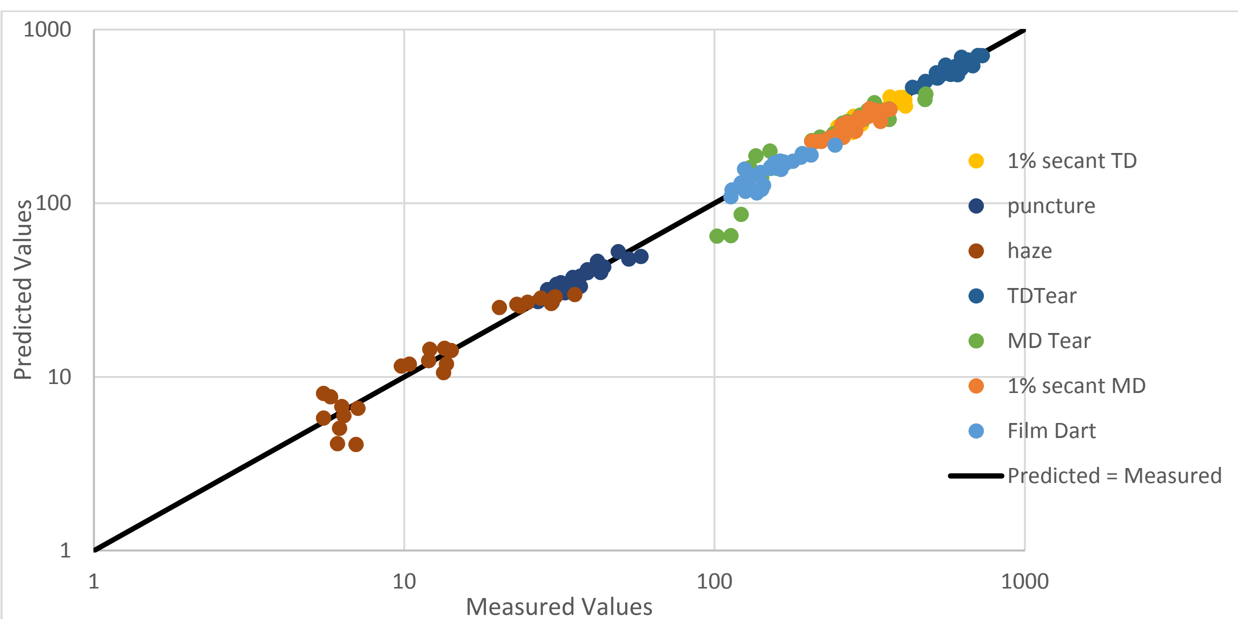
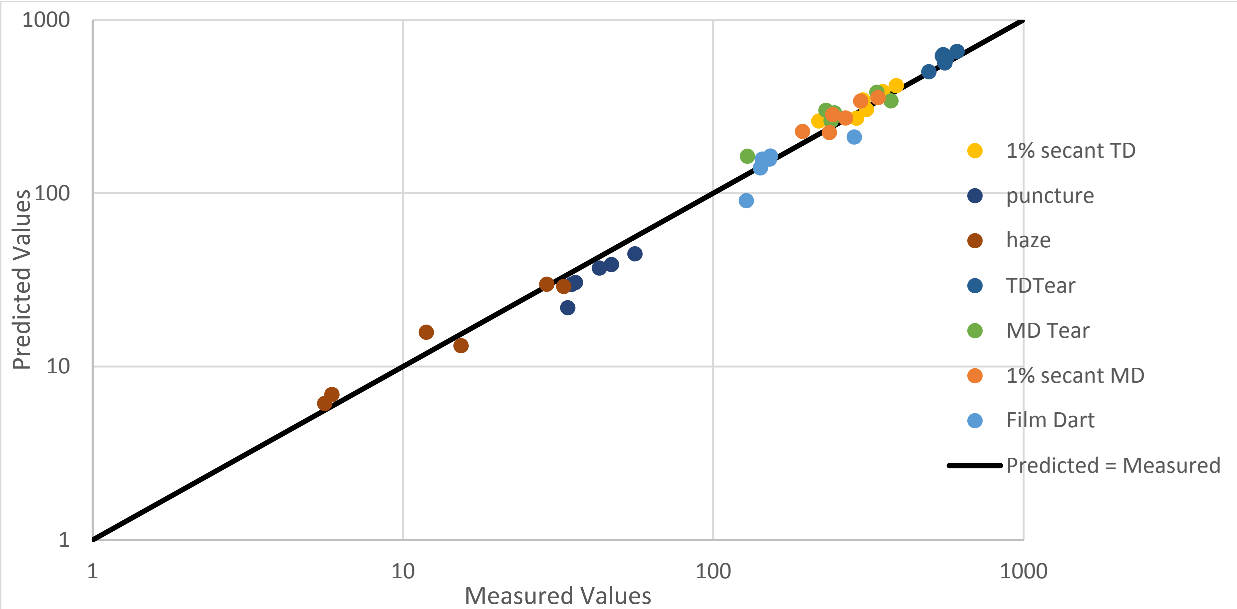


Figure 2. Predicted values as compared to the measured values for the 6 validating runs



FILM FORMULATION MODEL PREDICTIONS

With the models that have been developed it is possible to predict film properties over a range of conditions. We first selected three case studies to demonstrate the usefulness of the developed models. Table 4 shows property targets for three common applications for heavy duty sack films. In each case bags were purchased and some typical physical properties measured. It was not possible to obtain a full range of physical properties due to the limited sample size.

Table 3. Three Cases as Target for Prediction Based on the Models

Film Description	Current thickness, mil	Measured 1% MD secant, MPa	Measured MD Tear, g/mil	Minimum Dart requirement, g/mil
40 lb. salt bag	6.75	248	131	100
Loose fill insulation bag	4.5	345	112	100
Batt Insulation bag	2.5	227	140	100

Using the developed models, property predictions were developed for each film example using a range of resin choices and processing conditions. For all samples the modeling was run based on an assumed bag stiffness and thickness. The model predictions in Table 4 show that the measured MD tear targets can be reached for all resin configurations and processing conditions selected. If we assume a minimum dart requirement of 100 g/mil it is clear that for the loose fill insulation bag there are some cases where the dart value may be too low.

Table 4. Model Predictions for 3-layer films with Configuration of Table 3

LDPE In core only	Core Resin	Thickness Mil	BUR	Dart g/mil	MD tear g/mil	Puncture j/mm	1% Secant MD MPa
Salt bag Target				100	131		248
yes	s-LLDPE	6.75	1.7	178	453	41.5	251
yes	s-LLDPE	6.75	2.5	204	498	41.1	251
yes	b-LLDPE	6.75	1.7	141	363	30.9	251
yes	b-LLDPE	6.75	2.5	167	408	30.6	251
Loose fill insulation Target		4.5		100	112		
yes	s-LLDPE	4.5	1.7	133	309	33.7	345
yes	s-LLDPE	4.5	2.5	159	354	33.1	345
yes	b-LLDPE	4.5	1.7	95	220	23.1	345
yes	b-LLDPE	4.5	2.5	122	264	22.6	345
Batt insulation Target		2.5		100	140		
yes	s-LLDPE	2.5	1.7	186	395	44.9	234
yes	s-LLDPE	2.5	2.5	213	433	43.9	234
yes	b-LLDPE	2.5	1.7	149	305	34.4	234
yes	b-LLDPE	2.5	2.5	175	344	33.4	234

A second approach was to take a standard bag structure with established minimum property requirements and look at whether the models can be used to down-gauge the film thickness. The example presented is for a standard 25 kg resin bag. The current bag thickness and corresponding minimum performance properties were established.

The models were used to predict properties using a range of layer configurations and process conditions. The predicted values for all properties are well above the targets. The models were used to look at the effect of reducing the film thickness by roughly 10%. For this example the film stiffness was increased the required amount to compensate for the thinner gauge. Using these inputs, film properties were developed for different core resin and BUR selections. At the thinner gauge it is no longer possible to maintain dart and tear properties with the lower performing butene resin in the core. The puncture value is also marginal. The model was run with the option of placing LDPE in the skins and using either the single site octene or the hexene grade in the core and found to give an acceptable set of film properties.

The data in the chart below are predictive only and based on data generated from one extrusion line. It is therefore still necessary to run film trials on the commercial extrusion line to establish actual film properties but it is clear that the models can help direct the resin choices, film construction and processing conditions.

Table 5. Model Predictions for down-gaged 3-layer films with Configuration of Table 3

LDPE In core only	Core Resin	Thickness mil	BUR	Dart g/mil	MD tear g/mil	Puncture, j/mm	1% Secant MD, MPa
Target				**100**	**233**	**21**	**294**
Yes	s-LLDPE	5.6	1.7	153	409	37.1	302
Yes	s-LLDPE	5.6	2.5	180	418	36.7	302
Yes	b-LLDPE	5.6	1.7	116	319	26.6	302
Yes	b-LLDPE	5.6	2.5	142	329	26.2	302
Target				**112**	**260**	**24**	**330**
Yes	s-LLDPE	5	1.7	137	330	34.3	336
Yes	s-LLDPE	5	2.5	163	370	33.8	336
Yes	b-LLDPE	5	1.7	99	241	23.7	336
No	s-LLDPE	5	2.5	126	280	23.2	336
No	h-LLDPE	5	2.5	115	229	26.7	336

SUMMARY

The properties of three layer films as a function of the film configuration and processing conditions have been studied using a DOE approach. The film configuration variables studied were placement or absence of LDPE in the skin layer and choice of LLDPE resin in the core layer. The films were made at three different thickness and BUR levels. The total density was also controlled at three levels.

Using the DOE approach the full range of parameters could be studied with only 33 experimental runs. The measured film properties were well modeled as a function of a few key parameters. Model predictions were used to develop film formulation for a range of heavy duty sack applications.

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TRADEMARKS