four factors at two levels requires only 8 runs. This design is a resolution IV with the main factors clear of two factor interactions. The two factor interactions will be confounded in pairs but we can still determine their significance. Three center point runs are included to determine the pure error and test for curvature.

Experimental

The barefoot blown film resin was a butene 1.0 melt index LLDPE having a 0.918 g/cc density. Talc antiblocks and erucamide slip were first dispersed in the barefoot resin at 50% and 5%, respectively. These masterbatches were reduced to their final film concentrations in the barefoot resin. A Leistritz 27 mm co-rotating twin screw extruder, with side-stuffer, vacuum port and 40:1 L/D was used to compound the masterbatches and dilutions. Blown films were produced with a Killion Davis Standard single barrier screw (30:1 L/D) with a 425°F melt temperature. The spiral die had a 2.5 inch diameter die with 50 mil die gap.

The four factors levels are given for talc antiblock median particle size, antiblock loading, erucamide slip loading and film thickness (see table I). Seven film properties or responses were measured for all films including haze, clarity, gloss, blocking, reblock 60°C, static COF and kinetic COF (see table II). The half factorial was designed using a statistical software package (5, 6) (see table III).

Results

There were two objectives in this analysis. The first was to determine which variables significantly affect each film property. The second was to develop a regression model to predict film properties from variable levels not evaluated in actual film trials. The process for this is described below.

A four factor half factorial was first created in a statistical software package. The experiment had 8 runs with 3 center points for estimating error. The runs or films were made and tested. The data was then analyzed by the statistical software. The estimated effects were plotted on Pareto charts for each film property. The estimated effect indicates how much each variable and two factor interactions change from high to low level and positive or negative correlation. The p-values and R^2 (adjusted) in the ANOVA (analysis of variances) were used to decide which factors were significant. Factors, with P-values greater than 0.05, were eliminated and added to the error term. An increase in the R^2 value means the elimination of a factor or interaction has improved the correlation. Since two factor interactions were confounded, at least one of each pair had to be eliminated in order to calculate regression coefficients. Film property data can now be generated from factor levels outside of the experimental runs. Contour plots show how film properties change with two factors. Plots of observed versus predict data indicate the quality of the regression.

Discussion / Conclusions

Antiblock and erucamide loadings had the most significant affect on film properties. Antiblock loading affected all film properties, most significantly were optical and blocking/reblock properties. Erucamide loading most significantly affected COF (static and kinetic) and blocking/reblock properties. Reblock and kinetic COF were also affected by an interaction, which is possibly antiblock and slip loadings. Antiblock median particle size and film thickness had less of an effect on film properties including clarity, gloss, reblock and kinetic COF (see Table VI).

Based on this design of experiments (DOE), a model was developed that can be used to predict or determine antiblock size and antiblock / slip additive levels to achieve the desired film properties. These DOE can be very useful to minimize the number of experiments required to optimize film properties. Note all suggested conditions from the model should be validated experimentally.

References

1. Radosta, J. A., Polyolefins VII Conference Proceedings, 1991, pp 561-74.
2. Wooster, J. F., Simmons, B.E., Vol. 1, Antec 1996 Conference Proceedings, 20.
3. Catino, J. W., Deutsch, D. R., Gardiner, J. L., Tappi Place Conference 2002, pp 1322-1364.
4. Maltby, A., Marquis, R. E., J. of Plastic Films and Sheeting 14, 1998, 111.
5. Design-Expert® v.7.0.0, Stat-Ease Inc.
6. Montgomery, Douglas C., Design and Analysis of Experiments, 6th Ed., John Wiley and Sons, New York.

Table I. Factor Levels

Factor	High	Center	Low
Talc MPS, microns	4.0**	2.5	1.0
Antiblock, ppm	6000	4000	2000
Erucamide, ppm	2000	1500	1000
Thickness, mils	1.6	1.2	0.8

** Note: Experimental talc product.

Table II. Film Properties / Responses

Film Property	ASTM Method
Haze / Clarity, %	D 1003-92
45° Gloss, %	D 2457-90
Blocking, 23 °C Induced Reblock (24 hrs @ 1.0 psi) 60 °C	D 3354-89
Coefficient of Friction, COF Static and Kinetic	D 1894-93

Table III. Half Factorial – Runs and Variable Levels

Sample	Antiblock MPS microns	Antiblock ppm	Erucamide ppm	Film Thickness mils
1	**2.5**	**3500**	**1500**	**1.2**
2	1.0	6000	1000	1.6
3	4.0	1000	2000	0.8
4	4.0	6000	2000	1.6
5	4.0	6000	1000	0.8
6	1.0	6000	2000	0.8
7	1.0	1000	2000	1.6
8	4.0	1000	1000	1.6
9	**2.5**	**3500**	**1500**	**1.2**
10	**2.5**	**3500**	**1500**	**1.2**
11	1.0	1000	1000	0.8
12	Control	3500	1500	1.2

Note: Center point runs in bold.

Table VI. Film Property Results – Haze, Clarity, Gloss

Antiblock MPS microns	AB ppm	ER ppm	mils	Haze %	Clarity %	Gloss %
2.5	3500	1500	1.2	11.2	88.9	57
2.5	3500	1500	1.2	11.9	88.7	54
2.5	3500	1500	1.2	11.9	88.6	54
4.0	6000	2000	1.6	14.7	83.1	51
4.0	6000	1000	0.8	14.6	82.3	49
4.0	1000	2000	0.8	7.4	96.2	63
4.0	1000	1000	1.6	8.1	96.6	64
1.0	6000	2000	0.8	15.1	85.9	46
1.0	6000	1000	1.6	14.0	88.8	53
1.0	1000	2000	1.6	8.6	97.7	62
1.0	1000	1000	0.8	8.1	96.9	59
Control	3500	1500	1.2	9.4	94.7	60

Table V. Film Property Results – Blocking, Reblock, COF

Antiblock MPS microns	AB ppm	ER ppm	mils	Block g	Reblock 60C, g	COF Static	COF Kinetic
2.5	3500	1500	1.2	20	81	0.12	0.10
2.5	3500	1500	1.2	25	89	0.10	0.08
2.5	3500	1500	1.2	25	88	0.10	0.09
4.0	6000	2000	1.6	4	23	0.08	0.07
4.0	6000	1000	0.8	22	57	0.13	0.10
4.0	1000	2000	0.8	42	110	0.14	0.11
4.0	1000	1000	1.6	78	131	0.15	0.14
1.0	6000	2000	0.8	10	51	0.10	0.08
1.0	6000	1000	1.6	17	69	0.10	0.08
1.0	1000	2000	1.6	42	81	0.09	0.07
1.0	1000	1000	0.8	67	135	0.25	0.22
Control	3500	1500	1.2	54	91	0.11	0.09

Table VI. Significant Effects – Pareto Charts

Film Property	Significant Effect
Haze	AB loading
Clarity	AB loading, MPS
Gloss	AB loading, thickness
Blocking	AB & ER loading
Reblock 60C	AB & ER loading, AB / ER interaction, thickness
Static COF	AB & ER loading
Kinetic COF	AB & ER loading, film thickness & AB / ER interaction

AB=antiblock, ER=erucamide slip

Table VII. Statistical Variation

Center Points	Haze %	Clarity %	Gloss %	Blocking 23C	Reblock 60C	COF Static	COF Kinetic
1	11.20	88.9	56.8	20.2	82.2	0.117	0.097
9	11.90	88.7	53.6	25.0	88.5	0.103	0.084
10	11.90	88.6	54.4	25.0	87.5	0.104	0.086
1 σ (68%)	0.40	0.2	1.7	2.8	3.386	0.008	0.007
90% (1.65 σ)	0.67	0.3	2.8	4.6	5.654	0.013	0.012