Title:
The Effects of Processing Aids on the Hot-Tack Properties of LLDPE Blown Films

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Fluorine-based processing aids are an effective means for improving the surface quality of polymer extrudates, especially in LLDPE film applications. By reducing the shear induced stresses which can cause melt fracture and die-lip build-up, processing aids have also allowed many film processors to increase the output of their equipment. Although their effects on extrusion processes are generally viewed as positive, the effects of processing aids on down-stream film properties have been a historical concern. Many studies have been conducted which indicate that most commercially available processing aids have little or no effect on surface related film properties such as print adhesion and heat-seal integrity. In terms of heat-seal, these studies have focused primarily on the effects of fully quenched seals. In high-speed filling operations, such as vertical form and fill loading, the film processor must often rely on the hot-tack strength of the film to bear the weight of the load until the seal has fully quenched. In this study, three different fluorine-based processing aids were evaluated in a monolayer LLDPE film application over a range of concentrations. The hot-tack performance of these films was not adversely affected by the processing aids.

Date Published: January 1999

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The Effects of Processing Aids on the Hot-Tack Properties of LLDPE Blown Films

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ABSTRACT

Fluorine-based processing aids are an effective means for improving the surface quality of polymer extrudates, especially in LLDPE film applications. By reducing the shear induced stresses which can cause melt fracture and die-lip build-up, processing aids have also allowed many film processors to increase the output of their equipment. Although their effects on extrusion processes are generally viewed as positive, the effects of processing aids on downstream film properties have been a historical concern. Many studies have been conducted which indicate that most commercially available processing aids have little or no effect on surface related film properties such as print adhesion and heat-seal integrity. In terms of heat-seal, these studies have focused primarily on the effects of fully quenched seals. In high-speed filling operations, such as vertical form and fill loading, the film processor must often rely on the hot-tack strength of the film to bear the weight of the load until the seal has fully quenched. In this study, three different fluorine-based processing aids were evaluated in a monolayer LLDPE film application over a range of concentrations. The hot-tack performance of these films was not adversely affected by the processing aids.

INTRODUCTION

Fractional melt index LLDPE film resins are often used in high strength bag applications. Because of the relatively high melt viscosity of these resins, film producers often depend on fluorine-based processing aids to improve processability and film quality. Downstream conversion of these films can often include high speed bag-filling operations which depend on the hot-tack strength of the film to support the bags content until the heat-seals have fully quenched. It is important that fluorine-based processing aids do not adversely affect the hot-tack strength of these films.

EXPERIMENTAL

Resin & Film Sample Preparation

Three different types of fluorine-based processing aids were selected for this study. These processing aids were supplied by Dyneon LLC and are shown in Table I.

Table I. Processing Aids

Code	Commercial Name
PPA-1	Dynamar FX-9613
PPA-2	Dynamar FX-5920A
PPA-3	Dynamar FX-5911X

Each of these processing aids was compounded into a 1-hexene LLDPE film resin with a melt index of 0.50 g/10 min. and a density of 0.920 g/cc. This base resin was produced on a British Petroleum gas phase technology reactor. The samples were prepared through the neat addition of the processing aid using a 58 min twin-screw compounding extruder with an underwater die-face pelletizer. Other than a standard film grade antioxidant package, no other additives were incorporated into the resin.

For each type of processing aid studied, three concentrations were compounded into the base resin. A blank base resin sample containing no processing aid was also compounded for use as a control. The concentration of processing aid in the compounded resins was confirmed using a Parr bomb technique and ion specific electrode to measure the total fluorine content. Microscopic analysis of the compounded resins verified that the processing aids were adequately dispersed in the resin. A full description of the resins can be found in Table II.

The resins were converted into films using a 63.5 min extruder fitted with a 20.3 cm diameter blown film die with a 2.54 min die gap. To insure that each film sample in the study had similar surface qualities, the samples were extruded at a relatively low shear rate. The film fabrication output for each sample was maintained at a nominal 1.07 kg/hr/cm of die circumference and the film thickness was targeted at 50.8 µm. The films were allowed to age one week prior to testing.

Test Specimen Preparation & Experimental Design

Test specimens were prepared by cutting 25.4 min wide strips oriented in the machine direction (MD) so that the hot tack seal was perpendicular to the film orientation . The hot tack strength of duplicate specimens was tested using a J&B (formerly Top Wave) hot tack tester. The specific instrument settings for testing the specimens are listed in the Table III.

A modified 2 factor, 3 level factorial design of experiment was selected for this study. The order in which the samples were tested was randomized and blanks were analyzed at the beginning, middle, and end of the series to monitor for possible shifts in test response.

RESULTS AND DISCUSSION

The average peel strength for each processing aid level is reported in Table IV. Also included in Table IV is the pooled average peel strength for each resin across all levels of processing aid at each temperature. Plots of the hot tack peel strength curves are shown in Figures I & 2.

Model

The data was analyzed using an analysis of variance (ANOVA) allowing for eleven levels of temperature. The level of processing aid and the replicate runs were included as factors in the ANOVA.

The model is $Y_{ijk} = T_i + A_j\ TA_{ij} + R_{jk} + \varepsilon_{ijk}$

where:

Y_{ijk} is the observation.

T_i is the seal temperature, °C (75 to 125 at 5° increments). This factor is fixed.

A_j is the additive level: low, middle, high. This factor is fixed.

TA_{ij} is the temperature by additive level interaction.

R_{jk} is the replicate run within each additive level: I and 2. This factor is random.

ε_{ijk} is the random variability of an observation.

Summary and Interpretation

The data for the ANOVA can be found in Tables V & VI in the appendix. As expected, temperature had the most influence on the hot tack peel strength of the samples. Temperature accounts for 93% - 98% of the variability as measured by the sum of squares about the mean. (R-sq Temp). Although some of the other factors in the model appeared to be statistically significant, such as the temperature / processing aid level interaction, it can be argued that none of these factors had practical significance. This is further illustrated in the pooled data in Figure 3.

The hot tack average peak value for the three types of processing aids is not significantly different over the concentration range studied. The maximum peel strength is approximately 3.9 - 4.0 N for all three processing aids. Ranging from 115 - 120°C, the temperature at which the maximum peel strength is achieved is also not significantly different. These results are summarized in Table VII.

There are no clear trends associated with the type or concentration of the three processing aids studied. In other words, the general hot tack curve profile, or rate at which the maximum peel strength is achieved, is similar for the types and levels of processing aid. These results are summarized in Table VIII.

The major source of variability for hot tack was the individual analysis, which had a 95% confidence half-interval of ± 0.31 N.

CONCLUSION

For the types and concentrations of processing aid considered in this study, there appears to be no negative effect on the hot tack performance of the LLDPE films. Within the scope of this study, converting operations which depend on the hot tack performance of high strength LLDPE films should not be adversely affected by processing aids.

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1. A. Valenza and F.P. LaManita, Intern. Polymer Processing, "Extrusion Characteristics of Lubricated Linear Low Density Polyethylene", p 220 (1988).
2. A. Rudin, A. T. Worm, and J. E. Blalock, J. Plastic Film & Sheeting, "Fluorocarbon Elastomer Processing Aid in Film Extrusion of Linear Low Density Polyethylenes", p 189 (1985).
3. T. J. Blong and D. Duchesne, SPE PM& D Div. Newsletter, "Effects of Anti-Block/Processing Aid Combinations on LLDPE Blown Film Extrusion", vol. VII, No 1 (1989).

Table II. Experimental Design

Sample Code	Processing Aid	Target Concentration, ppm		Experimental Concentration, ppm
ER2343	Blank	-	0	0
ER2344	PPA-1	Low	200	182
ER2345	PPA-1	Middle	600	512
ER2346	PPA-1	High	1000	946
ER2347	PPA-2	Low	200	351
ER2348	PPA-2	Middle	600	505
ER2349	PPA-2	High	1000	876
ER2350	PPA-3	Low	200	147
ER2351	PPA-3	Middle	600	452
ER2352	PPA-3	High	1000	810

Table III. Hot-Tack Instrument Variable Settings

Variable	Setting	Description
Δ Temperature, °C	75 - 125, 5 degree increments	Seal jaw temperature setting.
Dwell, sec.	0.5	Length of time seal jaws are in contact with the film.
Pressure, kPA	206.7	Seal jaw pressure when in contact with the film.
Speed, mm/sec	200	Speed of seal separation.
Delay, sec	0.5	Time lag between when the seal is made and the seal separation begins

Table IV. Average Peel Strength by Processing Aid Level

Processing Aid	Level	N	Seal Temperature °C										
			75	80	85	90	95	100	105	110	115	120	125
Average Peel Strength, N													
PPA-1	Low	2	.87	1.13	1.48	1.95	2.04	2.93	3.47	3.66	3.91	3.82	3.87
PPA-1	Mid	2	.99	1.11	1.43	1.83	2.41	2.69	3.01	3.50	3.76	4.02	4.25
PPA-1	High	2	.79	.93	1.15	1.91	2.28	2.91	3.63	3.47	3.84	3.82	4.69
PPA-1	Average	6	.89	1.06	1.35	1.90	2.24	2.84	3.37	3.54	3.84	3.89	4.27
PPA-2	Low	2	.86	.99	1.38	1.92	2.36	3.09	3.37	3.56	3.86	3.72	5.46
PPA-2	Mid	2	1.00	1.15	1.33	1.78	2.73	3.01	3.05	3.41	3.84	3.52	3.49
PPA-2	High	2	.84	1.16	1.33	2.07	2.39	3.12	3.19	3.36	4.21	3.71	3.63
PPA-2	Average	6	.90	1.10	1.35	1.92	2.49	3.07	3.20	3.45	3.97	3.65	4.19
PPA-3	Low	2	.98	1.05	1.05	1.60	2.24	2.82	3.32	3.46	3.96	3.72	3.47
PPA-3	Mid	2	.94	1.21	1.29	1.99	2.45	3.14	3.56	3.59	3.84	3.80	3.93
PPA-3	High	2	.89	1.11	1.11	1.53	2.06	3.07	3.31	3.56	3.88	3.69	4.70
PPA-3	Average	6	.94	1.12	1.15	1.71	2.25	3.01	3.40	3.53	3.89	3.74	4.03
Blank	Low	2	.96	1.17	1.35	1.83	2.27	3.04	2.92	3.31	3.74	3.79	3.55
Blank	Mid	2	.87	1.04	1.36	1.46	2.50	2.98	3.18	3.25	3.72	3.70	3.52
Blank	Last	2	.91	1.25	1.50	1.81	2.38	3.31	3.39	3.60	4.18	3.97	3.47
Blank	Average	6	.91	1.15	1.40	1.70	2.38	3.11	3.16	3.39	3.88	3.82	3.52

Table V: Analysis of Variance

Analysis of Variance for FX-9613

Source	DF	SS	MS	F	P	SIG	VC	PCT VC
Temp	10	89.8921	8.9892	111.08	.000	***		
Add. level	2	.0179	.0090	.32	.749			
Level* Temp	20	1.6185	.0809	2.78	.006	**		
Rep w/i Level	3	.0843	.0281	.97	.423		.0000	0%
Observation	28	.8150	.0291				.0291	100%
Total	65	92.4278					.0291	100%
s(resid)=	.17			R-sq (Temp)	97.3%			

Analysis of Variance for FX-5920A

Source	DF	SS	MS	F	P	SIG	VC	PCT VC
Temp	10	83.6927	8.3693	33.21	.000	***		
Add. level	2	.4767	.2384	6.39	.083	(*)		
Level* Temp	20	5.0409	.2520	11.20	.000	***		
Rep w/i Level	3	.1119	.0373	1.66	.197		.0013	6%
Observation	30	.6750	.0225				.0225	94%
Total	65	89.9972					.0238	100%
s(resid)=	.15			R-sq (Temp)	93.0%			

Analysis of Variance for FX-5911X

Source	DF	SS	MS	F	P	SIG	VC	PCT VC
Temp	10	87.9319	8.7932	92.95	.000	***		
Add. level	2	.4016	.2008	17.02	.023	*		
Level* Temp	20	1.8920	.0946	3.49	.001	**		
Rep w/i Level	3	.0354	.0118	.44	.729		.0000	0%
Observation	30	.8127	.0271				.0271	100%
Total	65	91.0736					.0271	100%
s(resid)=	.16			R-sq (Temp)	96.6%			

Analysis of Variance for Blanks

Source	DF	SS	MS	F	P	SIG	VC	PCT VC
Temp	10	74.3030	7.4303	234.28	.000	***		
Block	2	.5000	.2500	22.12	.016	*		
Level* Temp	20	.6343	.0317	1.63	.111			
Rep w/i Block	3	.0339	.0113	.58	.633		.0000	0%
Observation	30	.5850	.0195				.0195	100%
Total	65	76.0562					.0195	100%
s(resid)=	.14			R-sq (Temp)	97.7%			

Table VI: Replicate Run Averages

Processing Aid Level	Run	N	FX-9613	FX-5920A	FX-5911X	Blank	Block
Low	1	11	2.67	2.71	2.53	2.55	First
Low	2	11	2.63	2.85	2.50	2.53	First
Low	Pool	22	2.65	2.78	2.52	2.54	First
Low	DIff		.04	-.14	.03	.01	First
Mid	1	11	2.58	2.57	2.71	2.47	Middle
Mid	2	11	2.69	2.58	2.70	2.55	Middle
Mid	Pool	22	2.64	2.57	2.71	2.51	Middle
Mid	Diff		-.11	.00	.01	-.08	Middle
High	1	11	2.69	2.66	2.59	2.71	Last
High	2	11	2.66	2.62	2.66	2.70	Last
High	Pool	22	2.68	2.64	2.63	2.71	Last
High	Diff		.03	.04	-.07	.01	Last

Table VII. Peak Peel Strength Comparisons

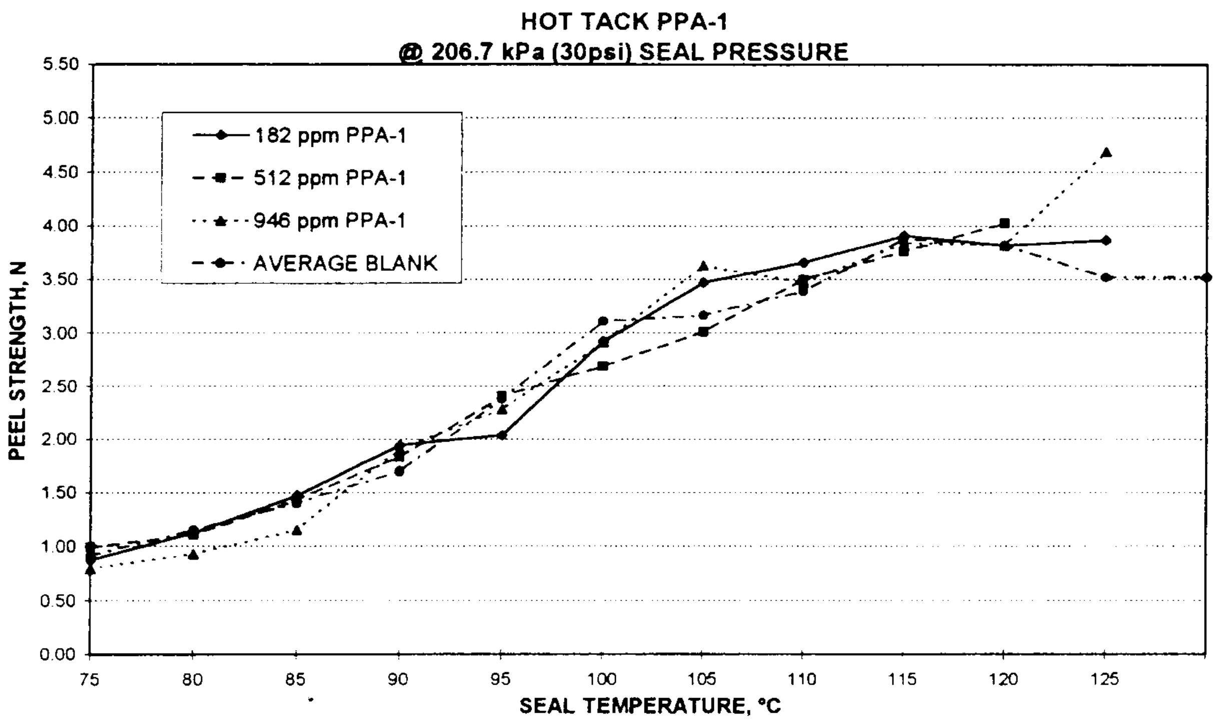
Processing Aid	Temperature, °C	Maximum Peel Strength, N (Pooled for all levels)	95% Confidence Interval (Pooled)
PPA-1	120	3.89	±0.13
PPA-2	115	3.97	±0.13
PPA-3	115	3.89	±0.13
Blank	115	3.88	±0.13

Table VIII. Average Peel Strength Based on Processing Aid Level

Processing Aid	Average Peel Strength, N			95% Confidence Interval (Pooled)
	Low Level	Middle Level	High Level	
PPA-1	2.65	2.64	2.68*	±0.07
PPA-2	2.78*	2.57	2.64	±0.07
PPA-3	2.52	2.71*	2.63	±0.07
Blank	First 2.54	Middle 2.51	Last 2.71*	±0.07

*Denotes maximum values.

Figure 1. Individual Hot Tack Curves for PPA-1 & PPA-2



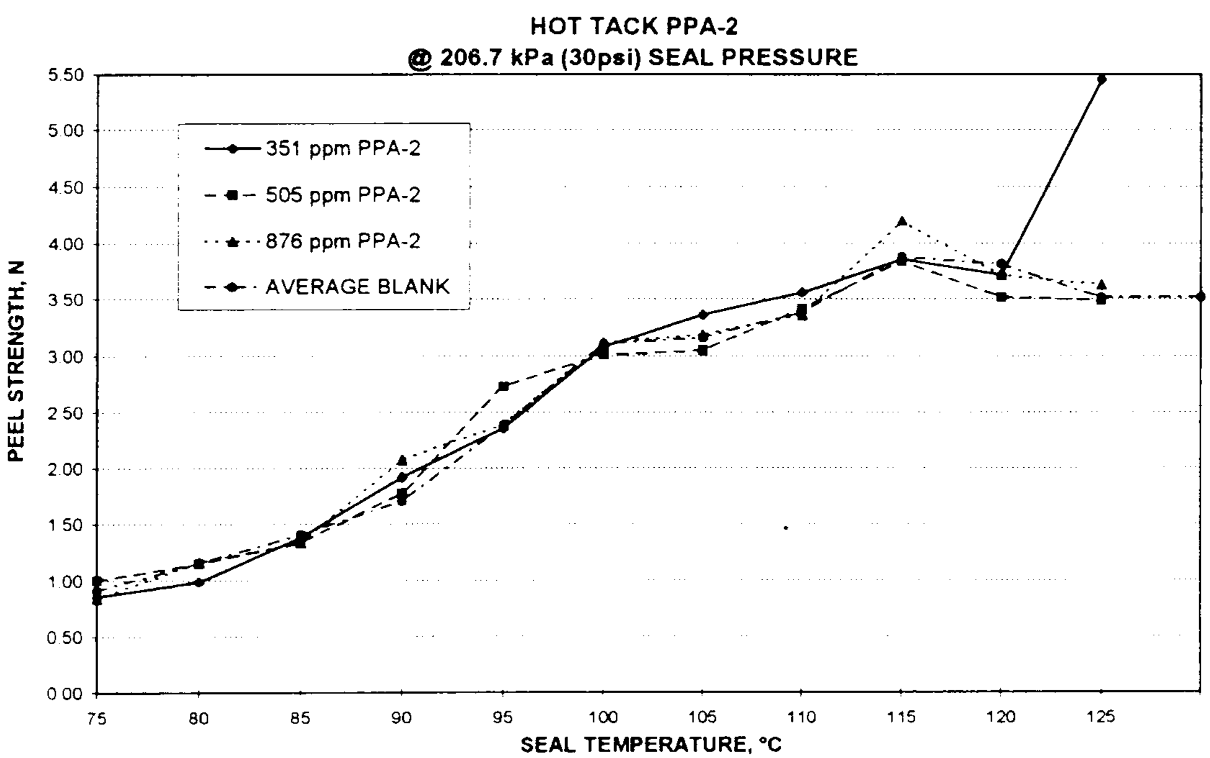
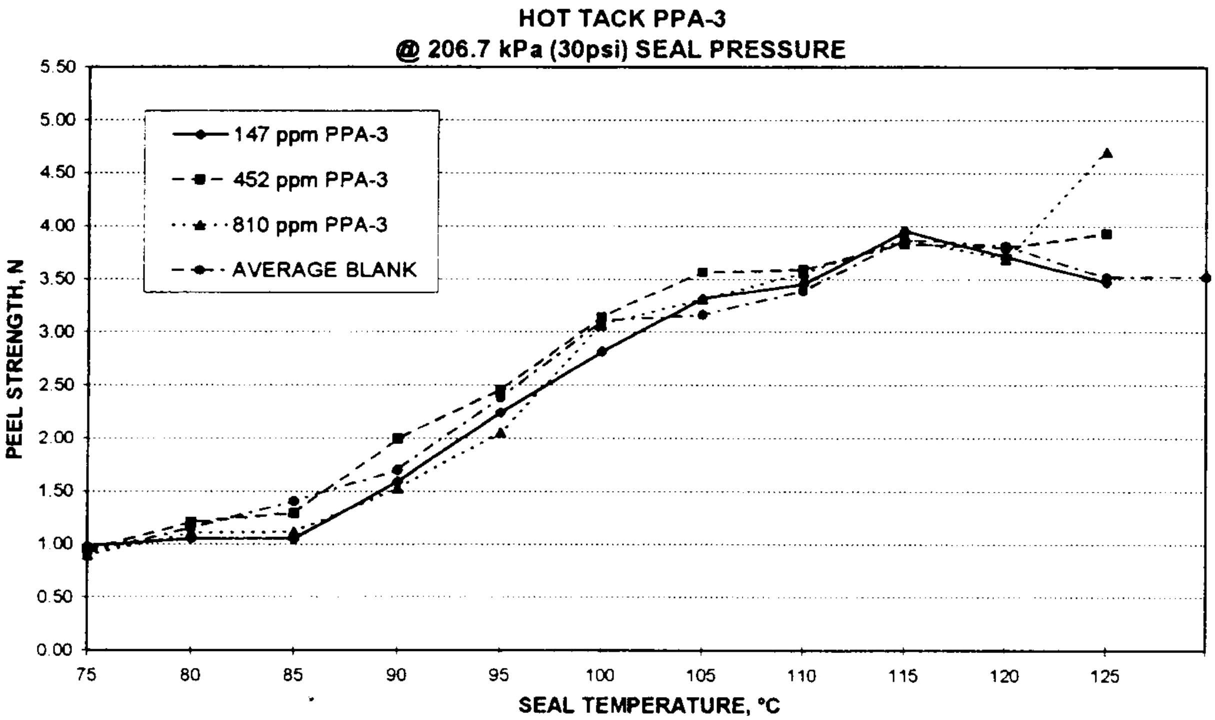


Figure 2. Individual Hot Tack Curves for PPA-3 & Blanks



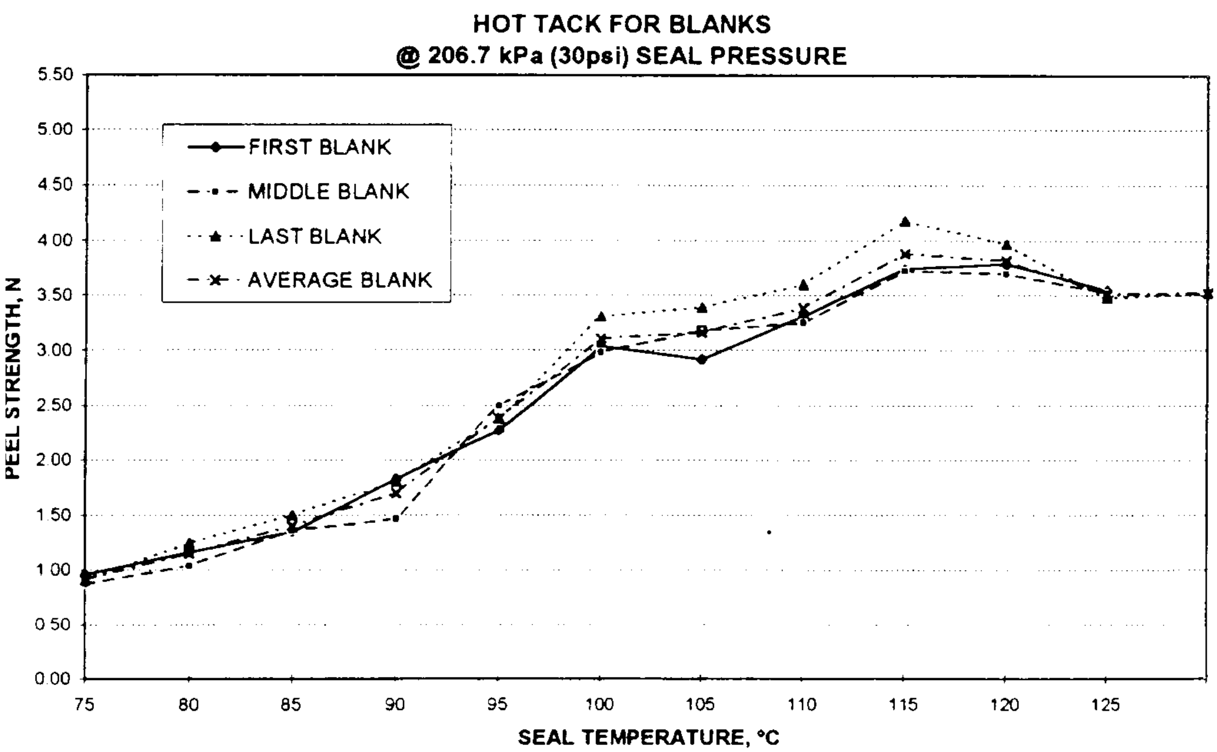
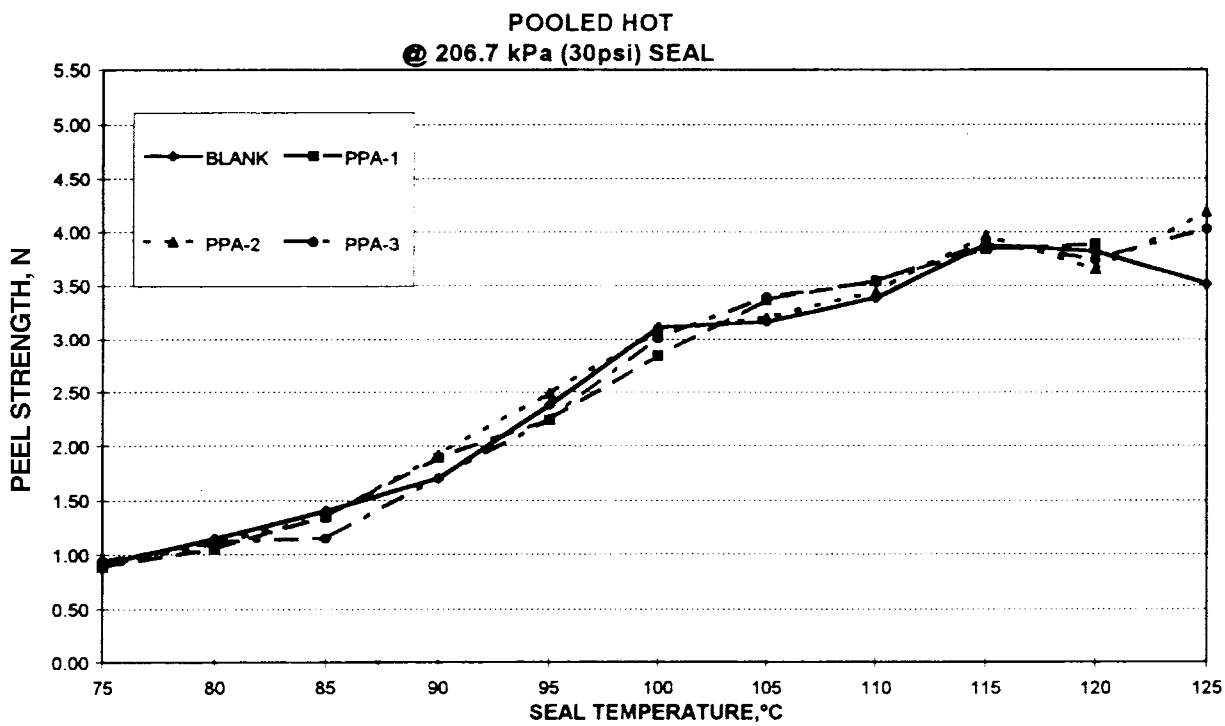
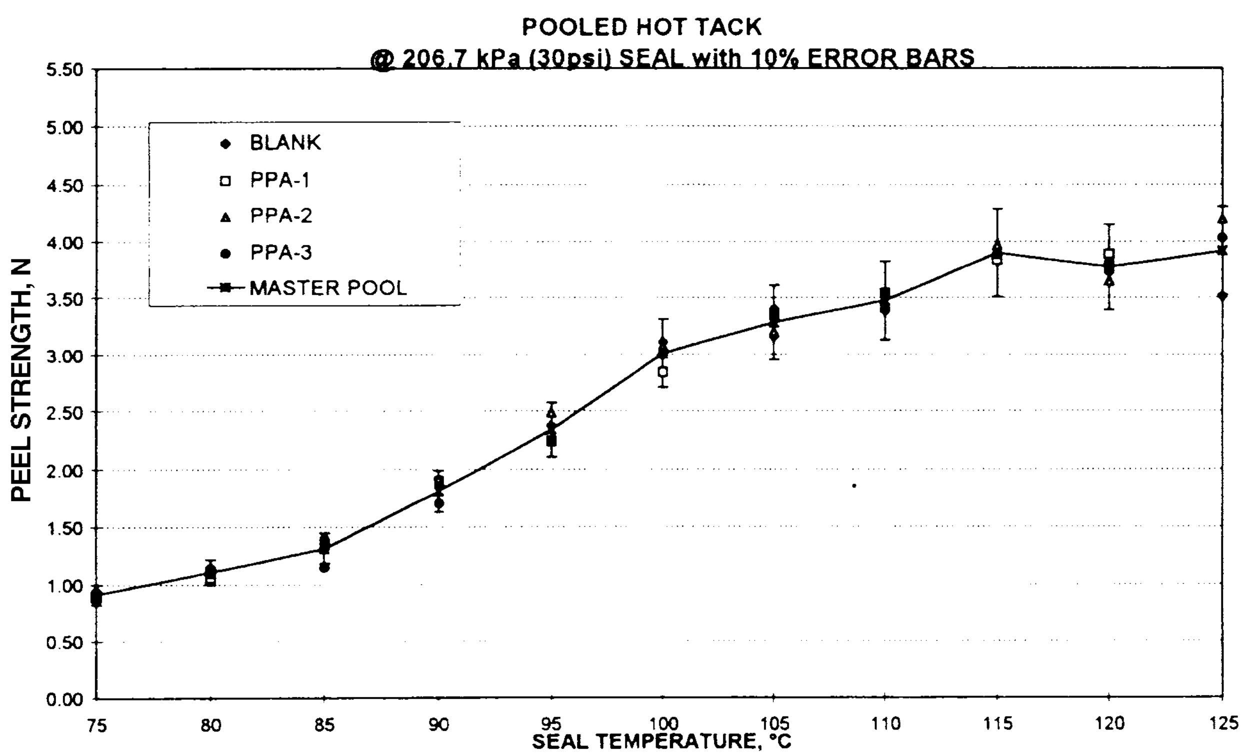


Figure 3. Pooled Hot Tack Curves





Pallamreddy, Praveen (pvpa)

From: kpca@orvax4.hou130.chevron.com[SMTP:kpca@orvax4.hou130.chevron.com]
Sent: Tuesday, May 19, 1998 9:37 AM
To: PVPA@chevron.com
Subject: TROUBLED LOT RESULTS

19-MAY-1998 Page 1

FINAL LOT ANALYSIS
SAMPLE RESULTS SHEET

Encompass V4.4

COMPUTER LOG NUMBER: 98ORPE051250
DATE LOGGED: 16-May-1998 21:39:00 SAMPLE TYPE: 5251/FP

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LOT NUMBER: H051090 PRODUCT ID: 5251
REACTOR/TRAIN: RX10 COMPOUNDER/EXTRUDER:
CONTAINER ID: ACFX97767

==

Test Code	Results	Limits	
MI	1.18 g/10mi	1.10	1.35
DEN	0.9296 gm/cc	.9280	.9310
HAZE	4.7		5.5
GLOSS	116	110	
G/32	CANCELLED	0	10
G/64	CANCELLED	0	10
X	0.0500 %	.0375	.0600
AF	0.1030 %	0.0850	0.1150
AP	0.0531 %	.0400	.0600
CV	0.6420 %	.5100	.6900
VA	4.2 %	3.7	4.5
CARB	0	0	1
SPEC	0	0	8
OXID	0	0	6
MET	0	0	2
COLOR	0	0	20
SPEC-BLD	0	0	8
OXID-BLD	0	0	6
CARB-BLD	0	0	1
MET-BLD	0	0	2
PF	0	0	0
FV_SMALL	186	0	140
FV_MEDIUM	22	0	40
FV_LARGE	2	0	5
DARK_GELS	0	0	50
SPORE	0	0	0
G/32	CANCELLED		

SALES WAIVER

FROM ____________________ FOR ___________________ CUSTOMER _______________

REMARKS

___

HOLD FOR ______________ DATE ___________ BY ___________ ENTERED _________

RELEASED AS ___________ DATE ___________ BY ___________ ENTERED _________

Pallamreddy, Praveen (pvpa)

From: jnth@orvax4.hou130.chevron.com[SMTP:jnth@orvax4.hou130.chevron.com]
Sent: Monday, May 18, 1998 1:06 PM
To: PVPA@chevron.com
Subject: TROUBLED LOT RESULTS

18-MAY-1998 Page 1

FINAL LOT ANALYSIS
SAMPLE RESULTS SHEET

Encompass V4.4

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REACTOR/TRAIN: RX10 COMPOUNDER/EXTRUDER:
CONTAINER ID: ACFX97767

==

Test Code	Results	Limits	
MI	1.18 g/10mi	1.10	1.35
DEN	0.9296 gm/cc	.9280	.9310
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AF	0.1030 %	0.0850	0.1150
AP	0.0531 %	.0400	.0600
CV	0.6420 %	.5100	.6900
VA	4.2 %	3.7	4.5
CARB	0	0	1
SPEC	0	0	8
OXID	0	0	6
MET	0	0	2
COLOR	0	0	20
SPEC-BLD	0	0	8
OXID-BLD	0	0	6
CARB-BLD	0	0	1
MET-BLD	0	0	2
PF	0	0	0
FV_SMALL	2	0	140
FV_MEDIUM	22	0	40
FV_LARGE	186	0	5
DARK_GELS	0	0	50
SPORE	0	0	0
G/32	CANCELLED		
G/64	CANCELLED		

SALES WAIVER

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REMARKS

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Issued: 1/99, 10/00

Printed in USA
98-0504-1110-1

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This paper was prepared for presentation at TAPPI '98. Published in the *1998 TAPPI Polymers, Laminations and Coatings Conference Proceedings*.

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98-0504-1110-1