



Producers of Specialty Chemicals

Struktol Company of America

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High Surface Area Silica
in
High Performance Tread
with
Next Generation Struktol Process Additives
(STRUKTOL[®] CY 48 and STRUKTOL[®] KK 49)
and
STRUKTOL[®] SCA 985
(Disulfide)

Struktol Rubber Lab Project 09006 Part 2

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Next Generation Struktol Process Additives

STRUKTOL® KK 49

Blend of fatty acid derivatives

DP 105C

Zn 10%

STRUKTOL® CY 48

Blend of fatty acid derivatives

DP 97C

Zn ~8%

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High Surface Area Silica in High Performance Tread with Next Generation Struktol Process Additives SCA985 (Disulfide)

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Outline

- Formulations
- Mix Spec
- Mix Data
- Physical Property Data
- Conclusions

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Formulations

First Pass	1	2	3	4	5
<u>INGREDIENT</u>	<u>MASS</u>	<u>MASS</u>	<u>MASS</u>	<u>MASS</u>	<u>MASS</u>
DURADENE 751B	103.5	103.5	103.5	103.5	103.5
TAKTENE 220	25	25	25	25	25
N220	10	10	10	10	10
SCA 985 (Disulfide)	5	5	5	5	5
ZNO	2.5	2.5	2.5	2.5	2.5
ST ACID	2	2	2	2	2
6PPD	2	2	2	2	2
SUNDEX 790	5	5	5	5	5
ZS P200MP	75	75	75	75	75
KK 49	0	4	6	0	0
CY 48	0	0	0	4	6
TOTAL	230	234	236	234	236

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Formulations

Second Pass	1	2	3	4	5
MB 1	230				
MB 2		234			
MB 3			236		
MB 4				234	
MB 5					236
TOTAL	230	234	236	234	236

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Formulations

Final Pass	1	2	3	4	5
MB 1	230				
MB 2		234			
MB 3			236		
MB 4				234	
MB 5					236
DPG	2.2	2.2	2.2	2.2	2.2
CBS	2.2	2.2	2.2	2.2	2.2
SULFUR	1.7	1.7	1.7	1.7	1.7
TOTAL	236.1	240.1	242.1	240.1	242.1

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Mix Spec

First Pass mix spec (65 rpm, fill factor 70%)

0 SECONDS ADD RUBBER

30 SECONDS ADD 1/3 SILICA

60 SECONDS ADD 1/3 SILICA

90 SECONDS ADD SILANE, OIL, CARBON BLACK, ADDITIVES AND REST OF SILICA

180 SECONDS OR 266F RAM LIFT

300 SECONDS OR 302F BRUSH AND SWEEP

420 SECONDS OR 320F DISCHARGE

ROTOR SPEED 65 RPM AT DISCHARGE 30 PSI

2nd pass mix spec for batch rotor speed started at 110 rpm, at 320F changed rotor speed to 60 rpm fill factor 70%)

0 SECONDS ADD all MB

320F AUTO RAM LIFT, CHANGE ROTOR SPEED TO 60 RPM

DISCHARGE 7 MINUTES AFTER ROTOR SPEED

Mix spec final pass for batch (65 % fill factor)

0 SECONDS ADD ALL RUBBER

15 SECONDS ADD CURES

60 SECONDS BRUSH AND SWEEP

150 SECONDS OR 226F DISCHARGE

ROTOR SPEED 77 RAM PRESSURE 30 PSI

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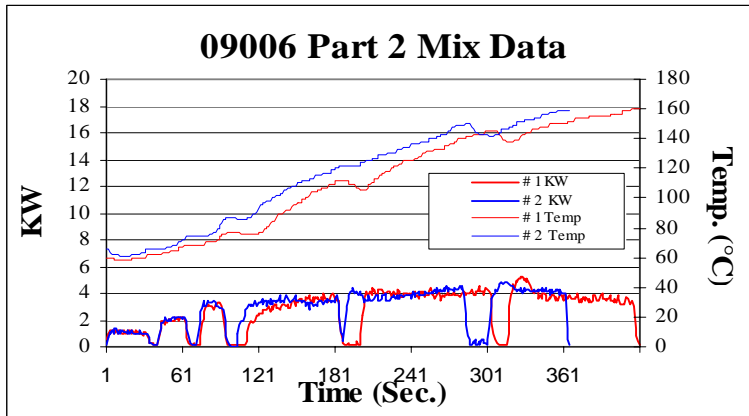
Mixing Observations

Compound	Temp (°C)	Probe Temp (°C)	Energy (WH)	First pass comments
BATCH 1 (Control)	160	177.5	321	Ram did not bottom out/Ok
BATCH 2 (KK 49) 4 phr	160	175.5	288	Ok
BATCH 3 (KK 49) 6 phr	160	174.5	234	Ok
BATCH 4 (CY 48) 4 phr	160	175.5	224	Ok
BATCH 5 (CY 48) 6 phr	160	174.4	224	Ok

Significant Reduction in mix energy with Struktol Additives

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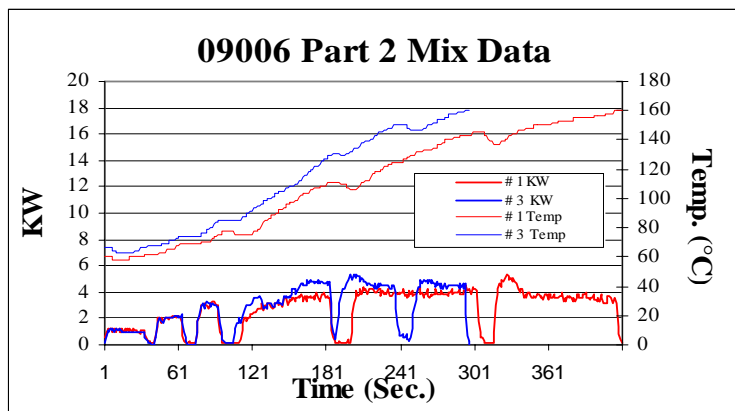
Mixing Observations



Mixing Time Reduced with Struktol Additives
Mixing Peaks Shifted with Struktol Additives

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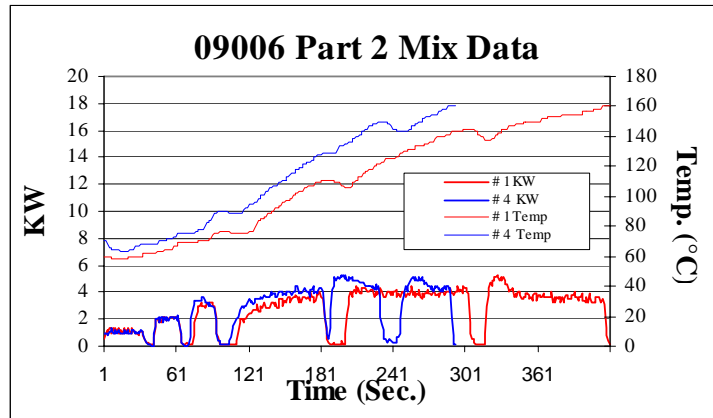
Mixing Observations



Mixing Time Reduced with Struktol Additives
Mixing Peaks Shifted with Struktol Additives

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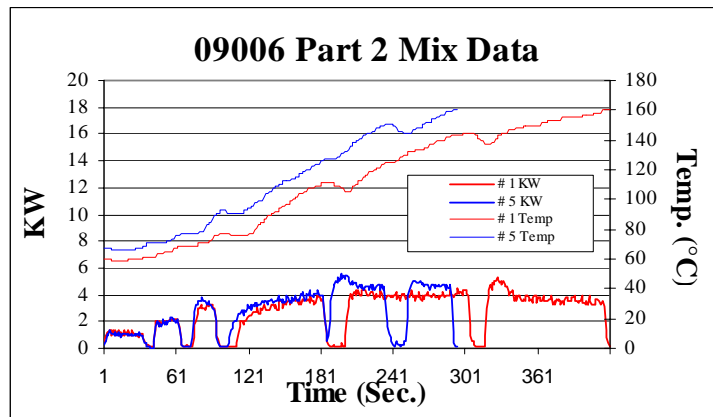
Mixing Observations



Mixing Time Reduced with Struktol Additives
Mixing Peaks Shifted with Struktol Additives

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Mixing Observations



Mixing Time Reduced with Struktol Additives
Mixing Peaks Shifted with Struktol Additives

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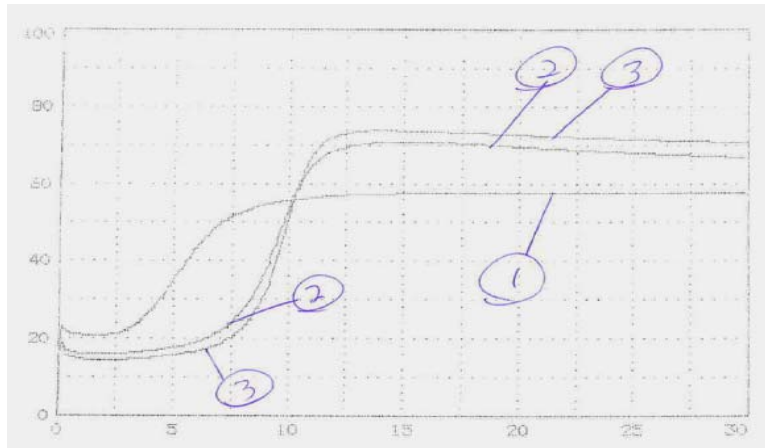
Mixing Observations

Compound	Temp (°C)	Probe Temp(°C)	Energy (WH)	Second pass comments	Final pass comments
BATCH 1 (Control)	166	177.5	537	Ok	Ok
BATCH 2 (KK 49) 4 phr	163	173.8	487	Ok	Ok
BATCH 3 (KK 49) 6 phr	160	169.0	447	Ok	Ok
BATCH 4 (CY 48) 4 phr	170	177.0	472	Ok	Ok
BATCH 5 (CY 48) 6 phr	163	173.5	444	Ok	Ok

Energy Consumption Reduced with KK 49 and CY 48

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Rheometer Curves



Reduced viscosity and increased cure state with KK 49

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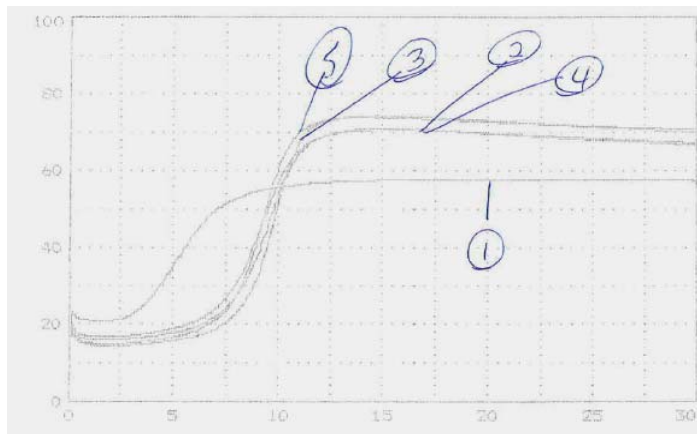
Rheometer Curves



Reduced viscosity and increased cure state with CY 48

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Rheometer Curves



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Rheometer Data

160°C; 3° arc; 100 range; 30 minutes – Tech Pro

Compound	Min Torque	Max Torque	Ts ₂	T ₅₀	T ₉₀	T-2 Reversion
BATCH 1 (Control)	20.80	57.64	3.33	5.58	8.75	-
BATCH 2 (KK 49) 4 phr	15.86	70.89	5.38	9.46	11.25	22.63
BATCH 3 (KK 49) 6 phr	14.41	73.80	5.96	9.75	11.17	24.29
BATCH 4 (CY 48) 4 phr	16.68	70.72	5.13	9.17	11.17	24.42
BATCH 5 (CY 48) 6 phr	14.82	74.20	5.46	9.25	10.83	22.92

Increased cure state and reduced minimum torque with KK 49 and CY 48

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Mooney Viscosity

ML (1+4) @ 100°C Aged Mooney

Compound	Init. Vis	ML(1+4)	Init. Vis 3 day	ML(1+4)	Init. Vis 14 day	ML(1+4)	Init. Vis 28 day	ML(1+4)
BATCH 1 (Control)	118.5	84.6	137.8	88.8	137.7	87.2	175.7	102.4
BATCH 2 (KK 49) 4 phr	98.3	72.3	108.2	76.5	114.1	75.9	126.6	82.9
BATCH 3 (KK 49) 6 phr	92.2	69.2	101.1	72.9	106.8	71.4	116.2	77.1
BATCH 4 (CY 48) 4 phr	104.2	77.4	114.4	81.5	124.3	81.7	135.6	90.2
BATCH 5 (CY 48) 6 phr	95.8	72.6	102.7	77.7	110.7	76.3	121.4	83.3

**Decreased viscosity for better processability for KK 49 and CY 48
Better viscosity control with time providing more stable stock storage**

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Mooney Scorch

ML @ 125°C

Compound	Min Torque	T ₅	T ₃₅	Cure Index
BATCH 1 (Control)	74.4	21.24	31.66	10.42
BATCH 2 (KK 49) 4 phr	60.8	46.37	-	-
BATCH 3 (KK 49) 6 phr	59.5	46.58	57.73	11.15
BATCH 4 (CY 48) 4 phr	68.4	45.07	59.02	13.95
BATCH 5 (CY 48) 6 phr	61.0	41.43	55.56	14.13

Scorch Safety Doubled with KK 49 CY 48

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Tensile Data

Unaged

Compound	Cure Time @ 160C	Shore A Duro	Tensile (MPa)	Elongation (%)	100% Mod. (MPa)	200% Mod. (MPa)	300% Mod. (MPa)
BATCH 1 (Control)	13	67	17.1	431	2.1	5.4	10.3
BATCH 2 (KK 49) 4 phr	13	67	21.4	395	3.2	8.2	15.0
BATCH 3 (KK 49) 6 phr	13	67	18.9	445	2.8	6.1	10.6
BATCH 4 (CY 48) 4 phr	13	67	19.8	343	3.4	9.3	14.9
BATCH 5 (CY 48) 6 phr	13	67	19.1	365	3.5	8.7	14.8

Increased Tensile and Modulus with KK 49 and CY 48

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Oven Aged Tensile

70 Hrs @ 100°C

Sample	Shore A	Pts. Chg	Tensile	Pct. Chg	Elong %	Pct. Chg	100% Mod. Mpa	Pct. Chg	200% Mod. Mpa	Pct. Chg	300% Mod. Mpa	Pct. Chg
BATCH 1 (Control)	76	9	16.4	-4.1	239	-44.5	5.2	147.6	12.8	137.0	-	-
BATCH 2 (KK 49) 4 phr	76	9	17.7	-17.3	298	-24.6	4.8	50.0	10.9	32.9	15.5	3.3
BATCH 3 (KK 49) 6 phr	76	9	17.0	-10.1	283	-36.4	5.3	89.3	11.5	88.5	-	-
BATCH 4 (CY 48) 4 phr	74	7	18.4	-7.1	276	-19.5	5.6	64.7	12.7	36.6	-	-
BATCH 5 (CY 48) 6 phr	77	10	16.1	-15.7	246	-32.6	5.7	62.9	12.8	47.1	-	-

Better retention of aged properties with KK 49 and CY 48

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Tear Strength

ASTM D – 624 Die C N/MM

Compound	23°C	100°C
BATCH 1 (Control)	39.7	46.7
BATCH 2 (KK 49) 4 phr	50.7	53.2
BATCH 3 (KK 49) 6 phr	50.1	52.2
BATCH 4 (CY 48) 4 phr	53.3	49.1
BATCH 5 (CY 48) 6 phr	51.7	47.8

Tear Strength better with KK 49

Tear Strength better at RT and equal at 100C with CY 48

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Heat Build Up

250 lb. Weight; 0.325" Throw; 45 minute test

Compound	Cure time 160C	Duro	Temp°C
BATCH 1 (Control)	52	66	153.2
BATCH 2 (KK 49) 4 phr	52	66	155.2
BATCH 3 (KK 49) 6 phr	52	67	152.7
BATCH 4 (CY 48) 4 phr	52	67	159.6
BATCH 5 (CY 48) 6 phr	52	67	153.7

Heat Build Up is equivalent

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Capillary Rheometer Data

100 C, 180 sec preheat Die L/D ratio: 15:1

90 entrance angle: 1.5 mm orifice

	Apparent Stress (Pa)	Apparent Viscosity (Pa-s)	Apparent Stress (Pa)	Apparent Viscosity (Pa-s)
Shear	500/s	500/s	1000/s	1000/s
BATCH 1 (Control)	279,070	558.17	324,870	324.85
BATCH 2 (KK 49) 4 phr	253,420	506.87	284,560	284.55
BATCH 3 (KK 49) 6 phr	242,430	484.89	274,180	274.17
BATCH 4 (CY 48) 4 phr	270,050	541.07	301,660	301.65
BATCH 5 (CY 48) 6 phr	251,590	503.21	281,510	281.50

**Reduced apparent viscosity for better extrudability
and less scrap/workaway**

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MER

Tension/Compression/Cylindrical / 1 HZ

Compound	Tan delta/ average of 3 / 23c	Tan delta/ average of 3 / 100c
BATCH 1 (Control)	.162	.104
BATCH 2 (KK 49) 4 phr	.166	.117
BATCH 3 (KK 49) 6 phr	.150	.124
BATCH 4 (CY 48) 4 phr	.152	.122
BATCH 5 (CY 48) 6 phr	.147	.128

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Spiral Mold Flow

Cured at 160°C

Compound	Cure	Weight 1	Weight 2	Weight 3	Average Weight
BATCH 1 (Control)	13	1.623	1.613	1.571	1.602
BATCH 2 (KK 49) 4 phr	13	1.886	1.892	1.845	1.874
BATCH 3 (KK 49) 6 phr	13	1.964	1.894	2.009	1.956
BATCH 4 (CY 48) 4 phr	13	1.877	1.867	1.872	1.872
BATCH 5 (CY 48) 6 phr	13	1.953	1.998	1.962	1.971

**Significantly increased flow for better processability
with KK 49 and CY 48**

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Conclusions

Struktol Additives KK 49 and CY 48

- **Reduced mix energy and mix time.**
- **Increased cure state.**
- **Better scorch resistance.**
- **Higher tensile and modulus.**
- **Better tear.**
- **Reduced viscosity for better processability/
extrudability (less scrap/workaway).**

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