TECHNICAL Bulletin

Subject: Butyl Rubber Compounds

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COMPOUNDING BUTYL RUBBER WITH THERMAL CARBON BLACK

Thermax® medium thermal carbon black N990 is manufactured by the thermal decomposition of natural gas. The thermal process provides a unique carbon black characterized by a large particle size and low structure. Thermax® is widely used in applications that require excellent heat, oil and chemical resistance and dynamic properties. The large particle size (low surface area) and low structure allow for low compression set, high rebound and low hysteresis, maintaining the inherent elastomeric properties of the rubber compound. These unique properties also help to maintain low viscosity, provide excellent dispersion and reduce heat build-up in processing. As a non-reinforcing black, thermal black is often blended with furnace carbon blacks and/or mineral fillers to achieve cost reduction and specific physical properties in the rubber compound.

Thermax® can be used in all polymers and is commonly used in elastomers such as IIR, NBR, EPDM, HNBR, ACM and ECO. High loadings of Thermax® are possible, while maintaining low viscosity, low compression set and high resiliency, thereby allowing manufacturers to reduce compound cost. Thermal blacks are often blended into high tensile stocks containing furnace blacks to give resilience and lower heat build-up.[1]

Butyl rubber is known for its special properties, including low rates of gas permeability, thermal stability, ozone and weathering resistance, vibration damping, higher coefficients of friction and chemical and moisture resistance.[2] For most butyl compounds used in the rubber industry, reinforcing fillers are specified to achieve smooth processing and appropriately high modulus and strength properties in the vulcanizate. Carbon black, together with an appropriate quantity of process oil, gives the best balance in processing and in the physical properties of the vulcanizate.[3] Applications where butyl rubber is filled with thermal black include body mounts, condenser packings, gaskets, tank linings, roll coverings, curing bladders, cable and hose.

Proper selection of carbon black grade is important for obtaining optimum compound properties. For example, fillers with high surface area should not be used with fluids that attack the polymer by absorption because such fillers tend to imbibe the chemical. Hygroscopic fillers should be avoided when resistance to water, salt solutions or weak acids and bases is a requisite.[4] Better compression set is realized with stocks loaded with large particle size fillers than with fine particle fillers.

[1] H. Nagano, Exxon Butyl Rubber Compounding and Applications, Exxon Chemical Publication, p. 19
[2] J.V. Fusco and P. Hous, "Butyl and Halobutyl Rubbers," in The Vanderbilt Rubber Handbook, 13th edition, 1990, p. 101
[3] Bayer-Polysar Butyl Website – Section 9.3, Compounding, p. 5
[4] ExxonMobil Chemical: Compounding for Chemical Resistance (website)

cancarb
1702 Brier Park Cres. N.W. • Medicine Hat, AB, Canada T1C 1T8
Phone 1.403.527.1121 • Fax 1.403.529.6093

Butyl is widely used in applications that require impermeability, such as curing bladders and ball liners. Because filler particles are impermeable, the permeability decreases with filler loading. A reduction of 33% is observed when SRF carbon black content is increased from 0 to 60 parts by weight[5]. However, thermal black can be used at higher loadings than all other carbon blacks due to its non-reinforcing properties arising from the low structure and large particle size. Therefore higher levels of impermeability are obtainable with high loadings of thermal black.

In Table 1 below, the effect of increasing the loading of N990 in butyl is demonstrated. The test compound and details are shown below.

Butyl Compound

Exxon Butyl 268	100.00
Carbon Black	As shown
Vanfre AP – 2	2.00
Stearic Acid	1
Zinc Oxide	5
Sulphur	2
Methyl Tuads	1
Captax	0.5

Compounds cured 20 minutes @ 171°C, tear strength measured on Die A samples, compression set measured after 22 hours @ 70°C, Mooney measured @ 132°C.[6]

Table 1: Increasing Loading of Thermax® N990 in Butyl Rubber

Thermax® N990	25	50	75	100	125
Mooney, t5/ML	18/46	16/48	14/52	12/58	11/63
Hardness	40	48	54	62	66
300% Modulus, MPa	2.1	2.9	3.6	3.9	3.9
Elongation (%)	420	530	540	490	460
Tensile Strength, MPa	4.0	6.1	5.4	4.4	4.2
Tear Strength, kN/m (pli)	9.7	15.8	16.7	20.2	19.4
Compression Set (%)	15	16	17	18	18

Source: The Vanderbilt Handbook, 13th edition, p. 461

Thermax® N990 Ultra Pure

For applications requiring resistance to high temperature, air and steam, butyl rubber is normally vulcanized by the polymethylol-phenol resin cure system. In this case and depending on the compound and application, a low sulphur/low pH carbon black may be preferred. Thermax® N990 Ultra Pure is a specialty grade of Thermax® characterized by low ash levels, a typical pH of 5 and typical sulphur levels of 3 ppm. Table 2 reports the typical properties of Thermax® N990 and Thermax® N990 UP.

[5]Bayer-Polysar Butyl Website – Section 9.5, p. 1
[6]The Vanderbilt Handbook, 13th edition, 1990, p.462

Table 2: Typical Properties of Thermax® N990 and N990 Ultra Pure

Properties	Thermax® N990	Thermax® N990 UP
Nitrogen Surface Area m^2/g	9.5	9.5
DBP, ml/100 g	38	38
Sulphur, ppm	100	3
pH	9.5	5
Ash (%)	0.1	0.01

Thermax® N990 and N990 UP were evaluated in a blend with N330 in a resin-cure butyl curing bladder compound. In Table 3 the differences in the processing properties arising from the different chemistry of the carbon black can be seen, in spite of equivalent physical properties.

Table 3: Thermax® N990 versus N990 UP in Butyl Curing Bladder Compound

	Thermax® N990	Thermax® N990 UP
Thermax® Loading, phr	90	90
N330 Loading, phr	20	20
Mooney Scorch @ 137°C, ASTM D 1646		
Minimum	52	53
tmin + 5 minutes	35.8	17.4
ODR @ 190°C, 1° arc		
t90, minutes	37.6	30.7
t95, minutes	46.3	40.1
Hardness	74	72
Tensile Strength, MPa	8.9	8.9
Tear Strength, kN/m	33.1	32.3

The shorter tmin + 5 scorch time and the faster t90 cure time for the N990 UP are attributed to the lower pH and may be of value to specific applications and processing conditions.

Case Study: Butyl Condenser Packings

Condenser packings are a very demanding application and require the following:

- good dynamic properties to allow for expansion of the electrolyte
- high sealing properties and gas impermeability, to prevent leakage of the electrolyte
- low sulphur and chloride levels, to prevent chemical reaction
- high insulating values

Exxon Butyl 268 is commonly used in this application due to the high levels of chemical resistance and gas impermeability provided by this polymer. Thermax® N990 is used in butyl condenser packings, but generally in a blend with SRF or FEF carbon blacks.

In this case study, a control compound with 45 phr of SRF and 20 phr of N990 is compared to one compound with 95 phr of N990 and a third compound with 66 phr of N990 and 20 phr SRF. The control formulation was taken from the Exxon Butyl Rubber, Compounding and Applications brochure (see endnotes). The filler loadings were calculated for 70 Shore A Hardness.

Test Formulations

	Control	#2	#3
Exxon Butyl 268	100	100	100
Thermax® N990	20	95	66
SRF N762	45	-	20
Translink #37	90	90	90
Stearic Acid	1	1	1
Zinc Oxide	3	3	3
SP 1045 Resin	20	20	20

Compound Data

Processing Properties			
Compound	Control	#2	#3
Carbon Black Loading – N990/N762	20/45	95/0	66/20
Specific Gravity	1.3955	1.4367	1.4236
Mooney Viscosity, 100°C			
Initial	100.47	97.65	101.07
@ 4 minutes	79.43	78.94	81.22
Mooney Scorch, ML, 125°C			
Minimum Torque	59.15	58.29	59.10
Ts1, minutes	14.25	12.00	13.25
Ts5, minutes	37.75	33.25	34.00
Ts10, minutes	68.00	59.00	60.00
Ts35, minutes	147.75	139.00	140.00

The viscosity of the three compounds is essentially similar, in spite of the higher loadings of carbon black in compounds #2 and #3. As shown below, the rheometer data indicates little difference in processing properties.

Rheometer, ASTM D 2084, 1° arc, 190°C			
Minimum, dN-m	6.3	5.9	6.2
Maximum, dN-m	31.3	32.4	32.0
tc50	18.9	19.2	19.1
tc90	28.8	29.7	29.5

Vulcanizate Properties, ASTM D 412, Originals			
Carbon Black N990/N762	20/45	95/0	66/20
Hardness	69	71	71
100% Modulus, MPa	3.52	3.58	3.70
200% Modulus, MPa	5.31	5.07	5.35
300% Modulus, MPa	6.26	5.31	5.78
Tensile Strength, MPa	6.65	5.02	5.60
Elongation (%)	426	485	442

The filler loadings were designed for a Shore A Hardness of 70, which has been achieved. Except for tensile strength, the compound properties are essentially equal and indicate that the carbon black can be loaded 46% more than the control compound for equivalent hardness. This would provide substantial cost savings. Compounds with thermal black generally have lower tensile strength due to the non-reinforcing properties of the large particle, low structure black.

Aged Properties, 168 hours @ 125°C			
Hardness	74 (+7.2%)	77 (+8.4%)	76 (+7.0%)
100% Modulus, MPa	4.19 (+19%)	3.87 (+8.1%)	4.17 (+12.7%)
200% Modulus, MPa	5.38 (+1.3%)	4.13 (-18.5%)	ND
300% Modulus, MPa	ND	ND	ND
Tensile Strength, MPa	5.44 (-18.1%)	4.68 (-6.7%)	5.02 (-10.3%)
Elongation (%)	209 (-51%)	197 (-59%)	181 (-59%)

Compression Set, ASTM D 395, Method B, 25% compression, 70 hours @ 125°C			
Compression Set (%)	24.1	24.8	24.5

Resilience by Vertical Rebound, ASTM D 2632, @ 23°C			
Rebound (%)	5	5	4

Rebound Resistance of Rubber, DIN 53512 (Zwick Rebound Tester) @ 23°C			
Rebound Resist (%)	6.5	6.5	6.4

Dynamic properties are maintained in spite of the higher loading of carbon black in compounds #2 and #3.

Volume Resistivity, ASTM D 257, 100 VDC, @ 23°C			
DC Resistance, Ohms, average of 3	2.26×10^{8}	3.20×10^{10}	2.16×10^{9}
*Volume Resistivity (ohms-cm), average of 3	7.61×10^{9}	1.12×10^{12}	7.55×10^{10}

Volume Resistivity (ohms-cm) = electrode area/sample thickness (cm) x volume resistance (Ohms)

The insulating properties of the compound are critical for this application. Due to the low level of agglomeration of the carbon particles in the N990, substantially higher volume resistivity is obtained in the compound with thermal black only. The resistivity increases proportionally to the increasing loading of N990.

The presence of sulphur and chloride in the carbon black is a concern for condenser packing manufacturers. Thermax® N990 has typical sulphur levels of 100 ppm and 30 ppm for chloride. This is reduced to average levels of 3 ppm for both S and Cl for the Thermax® N990 Ultra Pure grade.

Summary

The filler loading can be increased by roughly 45% over the control compound when using Thermax® N990 as the sole filler, while maintaining hardness, compression set and dynamic properties. This will provide substantial costs savings as the filler displaces the more expensive polymer. Significantly higher levels of volume resistivity are also achievable. As fillers such as carbon black are generally impermeable, the compounds with higher loadings will also have enhanced impermeability.