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# TECHNICAL BULLETIN

## Butyl Rubber Compounds

Thermax® N990 medium thermal carbon black 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 grade 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.

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 carbon 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 carbon black.

[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)
[5] Bayer-Polysar Butyl Website – Section 9.5, p. 1

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In Table 1 below, the effect of increasing the loading of Thermax® N990 in butyl is demonstrated. The test compound and details are as follows:

| Butyl Compound | phr |
|---|---|
| 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® Ultra Pure N990UP**

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® Ultra Pure N990UP 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® N990UP.

[6] The Vanderbilt Handbook, 13th edition, 1990, p.462

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**Table 2: Typical Properties of Thermax® N990 and Thermax® Ultra Pure N990UP**

| Properties | Thermax® N990 | Thermax® N990UP |
|---|---|---|
| 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 Thermax® Ultra Pure N990UP 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 Thermax® Ultra Pure N990UP in Butyl Curing Bladder Compound**

| | Thermax® N990 | Thermax® Ultra Pure N990UP |
|---|---|---|
| **Thermax® Loading (phr)** | **90** | **90** |
| N330 Loading (phr) | 20 | 20 |
| **Mooney Scorch @ 137°C, ASTM D1646** | | |
| Minimum | 52 | 53 |
| $t_{min}$ + 5 minutes | 35.8 | 17.4 |
| **Oscillating Disk Rheometer @ 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 $t_{min}$ + 5 scorch time and the faster t90 cure time for Thermax® 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 N762 and 20 phr of Thermax® N990 is compared to one compound with 95 phr of Thermax® N990 and a third compound with 66 phr of Thermax® N990 and 20 phr N762. 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.

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## Test Formulations (phr)

| | Control | #2 | #3 |
|---|---|---|---|
| Exxon Butyl 268 | 100 | 100 | 100 |
| **Thermax® N990** | **20** | **95** | **66** |
| N762 Carbon Black | 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, $M_L$, 125°C** | | | |
| Minimum Torque | 59.15 | 58.29 | 59.10 |
| Minutes to 1pt. rise | 14.25 | 12.00 | 13.25 |
| Minutes to 5pt. rise | 37.75 | 33.25 | 34.00 |
| Minutes to 10pt. rise | 68.00 | 59.00 | 60.00 |
| Minutes to 35pt. rise | 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.

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| | Control | #2 | #3 |
|---|---|---|---|
| **Rheometer, ASTM D2084, 1° arc, 190°C** | | | |
| Minimum (dN-m) | 6.3 | 5.9 | 6.2 |
| Maximum (dN-m) | 31.3 | 32.4 | 32.0 |
| $t_c50$ | 18.9 | 19.2 | 19.1 |
| $t_c90$ | 28.8 | 29.7 | 29.5 |
| **Vulcanizate Properties, ASTM D412, Originals** | | | |
| Carbon Black N990/N762 | 20/45 | 95/0 | 66/20 |
| Shore A 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 Thermax® black generally have lower tensile strength due to the non-reinforcing properties of the large particle, low structure carbon.

| | | | |
|---|---|---|---|
| **Aged Properties, 168 hours @ 125°C** | | | |
| Shore A 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 D2632, @ 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 D257, 100 VDC, @ 23°C** | | | |
| DC Resistance, Ohms, average of 3 | $2.26 \times 10^{8}$ | $3.20 \times 10^{10}$ | $2.16 \times 10^{9}$ |
| *Volume Resistivity (ohms-cm), average of 3 | $7.61 \times 10^{9}$ | $1.12 \times 10^{12}$ | $7.55 \times 10^{10}$ |

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

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

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® Ultra Pure N990 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.