

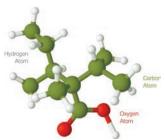
Excellent Rubber Adhesion with Cobalt Salts of Versatic[™] Acid 10





Versatic[™] Acid 10 is a synthetic, highly branched tertiary carboxylic acid which provides excellent rubber to steel adhesion properties when formulated as a Cobalt salt in passenger car, truck and bus radial (TBR) tire applications. The outstanding rubber adhesion attributes combined with the high product quality and consistency contributes significantly to the safety profile of radial tires. The initial adhesion between rubber and tire cord in steel-belted radial tires is superior and the adhesion properties are maintained during usage under high temperature, humidity and corrosive conditions. Versatic Acid 10 is not subject to oxidative degradation in contrast to unsaturated natural acids that require more expensive refrigerated transportation.

Characteristics and Structure



Acid value 318-330 mg KOH/g Appearance Clear liquid ≤30°C Melting point

Benefits

- Significant contribution is made to the **safety profile** of radial tires when formulated as a Cobalt salt.
- Superior initial adhesion between rubber and steel cord with excellent adhesion properties is maintained during usage under hot and humid conditions.
- The synthetically manufactured product has a high product quality and consistency.
- The saturated molecule has good storage stability and is not subject to oxidative degradation.

Adhesion

The adhesion properties of Versatic Acid 10 as a Cobalt salt in a typical rubber formulation for a steel-belted TBR tire have been measured versus a 20.5% Cobalt Neodecanoate commercial benchmark that is globally recognized as the industry standard. The Cobalt salt synthesis technology is proprietary to the Beijing Rubber Research and Design Institute and licensed for the Chinese market. It is based on Versatic Acids 10 and 5. All characterization and testing have been made according to standard methods.

Cobalt Salts Characterization

The characterization of the Cobalt salt synthesized from Versatic Acid 10 (Cobalt Versatate) is given in Table 1 and compared with the 20.5% Cobalt Neodecanoate commercial benchmark (Benchmark) and the market specifications. Cobalt content and softening point meet the market specifications.

Table 1: Cobalt Salt Characteristics

| Property | Cobalt Versatate | Benchmark | Market specifications |
|-----------------------|-------------------------------|-------------------------------|-------------------------------|
| Appearance | Brittle solid, blue-violet | Brittle solid, blue-violet | Brittle solid, blue-violet |
| Cobalt content (%) | 20.7 | 20.4 | 20.5 ± 0.5 |
| Softening point (°C) | 87 | 92 | 90 ± 10 |
| Heating loss test (%) | 0.2 | 2.4 | ≤1.0 |

Formulation and Mixing Process

The test parameters are given in Table 2 and are typical for TBR belt formulations.

- Natural rubber is mixed (80 rpm) at 80°C in the Banbury for 1 minute, the temperature is about 90°C.
- After rubber additives addition which includes Zinc Oxide, Cobalt salt, antioxidant and rubber adhesives, mixing is carried out for 1.5 minute, the temperature is about 110°C, and carbon black is added and mixed over 3.5 minutes.
- The temperature is increased at 15°C/min for 3.5 minutes until 150-170°C at which point the mixture is discharged from the Banbury.
- The formulated rubber is transferred to a rubber mixing mill: after adding sulfur and accelerator, it is processed 5 times at 90~95°C.

The cure curves in Figure 1 illustrate very similar curing behavior for both formulations.

The vulcanized rubber has been characterized: hardness, elongation at break, tensile strength, tension permanent deformation, tensile stress at a given elongation and tear strength were measured. Results below in Table 3 for the Cobalt Versatate are very similar to those for the benchmark sample.

Table 2: Rubber Formulation

| Component (in grams) | Cobalt Versatate | Benchmark |
|------------------------|------------------|-----------|
| Natural rubber | 100.0 | 100.0 |
| Carbon black | 53.5 | 53.5 |
| Zinc Oxide | 8.0 | 8.0 |
| Antioxidant | 3.5 | 3.5 |
| Sulfur and Accelerator | 6.4 | 6.4 |
| Benchmark Cobalt salt | _ | 1.2 |
| Cobalt Versatate | 1.2 | _ |
| Rubber adhesives | 5.7 | 5.7 |
| Total (grams) | 178.3 | 178.3 |

Figure 1: Mooney Viscosity Curves at 150°C Curing

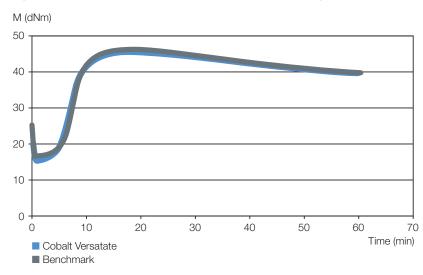


Table 3: Strength Test Results of Vulcanized Rubber Before Ageing

| Physical properties of vulcanized rubber (150°C, 20 min) | Cobalt Versatate | Benchmark |
|---|------------------|-----------|
| Shore hardness | 79 | 79 |
| Elongation at break (%) | 414 | 395 |
| Tensile strength (MPa) | 25 | 25 |
| Tensile stress at a given elongation M ₁₀₀ (MPa) | 6 | 6 |
| Tensile stress at a given elongation M ₃₀₀ (MPa) | 19 | 20 |
| Tension permanent deformation (%) | 20 | 20 |
| Tear strength (kN/m) | 56 | 57 |

Excellent Adhesion Performance

Initial adhesion at different curing conditions

To evaluate the robustness under different curing conditions, the H pull out force (N) has been determined for the below optimum (15 minutes curing at 150°C), optimum (30 minutes), slight over cure (40 minutes) and strong over cured samples (50 minutes). The rubber coating rate is greater than 95% (optical disc). As shown in Table 4, the variation trend versus curing time is very similar for the two samples.

Adhesion after ageing

The H pull out force (N) has also been determined after ageing under various conditions:

- Heat ageing (100°C for 24, 48 and 98 hours)
- Steam-heat ageing (60°C; 90% humidity for 24, 48 and 72 hours)
- Salt ageing (20% NaCl for 2 and 7 days)

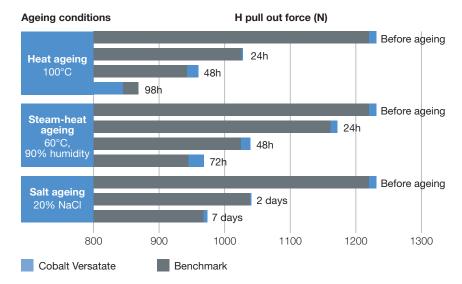
The adhesion results obtained were compared with the initial results. See Figure 2 for the results. The variation trend of the H pull out force of both cured rubber are the same whatever the ageing conditions are. The rubber coating rate is greater than 95% (optical disc), before and after ageing. The results demonstrate the good performance of the Cobalt salt based on Versatic Acid 10 as a rubber to steel tire adhesion promoter.

Table 4: H Pull Out Force Under Different Curing Conditions

| | H pull out force (N) | |
|-----------------------|----------------------|-----------|
| Curing time (minutes) | Cobalt Versatate | Benchmark |
| 15 | 907 | 869 |
| 30* | 1231 | 1220 |
| 40 | 1178 | 1153 |
| 50 | 983 | 990 |

^{*}Optimum curing conditions

Figure 2: H Pull Out Force of Vulcanized Rubber Before/After Ageing



Conclusions

- Versatic Acid based Cobalt salt technology complies with the requirements of the global and local chemical industry standards.
- When using Versatic Acid 10 in a typical belt formulation for TBR, good rubber processing performance is obtained with very similar mechanical properties to the commercial Cobalt salt benchmark sample.
- Rubber to steel adhesion stays at a high level after ageing under high temperature, humidity and corrosive conditions.

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