

Recycling of GTR with VESTENAMER®

The recycling of waste rubber is an important issue when it comes to use resources efficiently. Most rubber producers recycle already at least small amounts of their rubber waste, e.g., by adding ground rubber waste to fresh compounds. However, this way is restricted to very small amounts and does not solve the problem of disposing many thousand tons of rubber waste, particularly from tires (GTR = Ground Tire Rubber). The recycling of GTR is an important approach towards resource efficiency by making a putative waste a valuable raw material.

For reprocessing GTR a material is necessary to bind the ground rubber. Such a binder should have the following properties:

- act as crosslinking agent
- coating of the rubber granules
- improve processing
- Easy to handle
- Non toxic

Processing with VESTENAMER®

The material meeting these requirements is VESTENAMER® trans-polyoctenamer (TOR). Due to its melting range at 54°C, it functions as a plasticizer at processing temperatures. This property enables a very good coating of the ground rubber and a reduction of compound viscosity. Subsequent vulcanization converts VESTENAMER® into a true elastomer.

Compounds based on ground rubber and VESTENAMER® can be prepared in the traditional way by mixing in an internal mixer, when available. For a much easier reprocessing GTR is coated with TOR, oil and curing additives in a simple powder blender, similar to the mixing technology of thermoplastics.

This process consists of three steps:

1. Preparation of a solution of TOR in mineral oil
2. Mixing and coating GTR with the TOR solution and curing chemicals
3. Forming and vulcanization of this GTR blend



Preparation of the TOR solution

When solving TOR in mineral oil at temperatures between 80 and 120°C the VESTENAMER® granules are converted in a well dispersible form, and with the oil the hardness of the final product can be adjusted. An appropriate blend is 80 wt% paraffin or naphthenic oil and 20 wt% VESTENAMER® 8012. At ambient temperatures, this blend is wax-like and can be stored. At temperatures above 60°C, it is liquid.

Coating of GTR

In the second step, GTR is mixed in a low-speed powder blender at approximately 800–1000 min⁻¹ with curing chemicals, i.e., sulfur and accelerators on the one hand, and with the TOR-oil solution on the other hand. First the GTR and the chemicals are pre-blended for one minute. Then the 80–100°C warm TOR-oil solution is either sprayed or injected into the mixer. Table 1 lists a complete standard formulation, whereas in Table 2 the mixing sequence is shown.

Table 1: Standard formulation of GTR recycling

Material	phr
GTR	100
VESTENAMER® solution 20%	15
ZnO	1.5
Stearic acid	0.5
Sulfur	0.8
CBS	0.8
TMTD	0.2

Table 2: Mixing sequence for powder blender

Material	Unit	Value
GTR/additives/curing system	min	0
VESTENAMER® solution 20%	min	1
Dump	min	4
Wall temperature	°C	20–80
Speed	min ⁻¹	800–1000

Depending on the provenience and the grinding method of the waste rubber—cryogenic or warm—small adjustments to this standard formulation might be favorable. The curing system should crosslink the VESTENAMER® itself as well as achieve the bonding to the GTR granules.

The free flowing properties or friability of the dry blend achieved this way depend mainly on the grinding method of the tire rubber. The adsorption ability of cryogenically ground rubber is rather poor. This might afford an additional cooling step of the blend before dumping. The same applies for larger particle sizes (>4mm) of the ground rubber. At ambient temperatures ground rubber with particle sizes <2mm, however, adsorbs the oil much better and yields normally a free flowing powder that can even be stored for some time.

Production of finished products

The coated GTR compounds are processed to finished articles preferably by compression

molding. For this the "dry blend" is filled into the mold either directly from the powder blender or from a silo. As the bulk density of the compounds is low, we recommend telescopic molds. By varying the compression or the filling factor of those molds, either massive or porous products can be obtained. This will not cause great problems, as most articles produced from ground rubber waste will have simple geometric forms, e.g., sheets, mats or blocks. Compounds with a higher amount of the oil-VESTENAMER® solution can even be molded by injection molding.

Physical properties

The particle size and surface structure of the ground rubber primarily determine the physical properties. Principally it can be stated that the smaller the particle size is the better are the properties. Cryogenically ground rubber with low structure and a smooth particle surface is inferior to ambient ground rubber with a rough particle surface and high structure. Due to these influences, Table 3 only lists a range for each physical property.

The properties have been determined from test slabs according to the above-mentioned standard formulation. They were vulcanized for 15 minutes at 160°C. As expected the physical properties are not very high. The hardness of the ground rubber more or less determines the hardness of the product.

Compounds from GTR are in general less homogeneous compared to fresh rubber compounds. Consequently, the properties measured on test specimen cut from such more or less inhomogeneous test slabs can vary to a great extent whereas the finished products have quite acceptable usage properties.

Table 3: Physical Properties

Properties	Unit	Value
Hardness	Shore A	64–74
Tensile strength	MPa	4.5–8.5
Elongation at break	%	150–300
Modulus 100 %	MPa	2.0–3.0
Rebound resilience	%	50–60

Production of VESTENAMER® coated ground rubber waste

Production of the VESTENAMER® / oil blend

Mineral oil is heated up to about 100°C in a vessel with stirrer and VESTENAMER® granules are added. The mixture is stirred until the granules are completely dissolved. The dissolving time is 15 to 60 minutes, depending on the oil / VESTENAMER® ratio. VESTENAMER® / oil blends with up to 30% rubber are pourable at higher temperatures; at ambient temperatures, they have a wax-like consistency.

Coating ground rubber waste with VESTENAMER®

Ground rubber waste (e.g. from tires) is mixed in a powder blender (e.g. Diosna, Hentschel) with curatives and with the hot (100°C) oil / VESTENAMER® blend. Blending conditions:

- Speed 500–1000 rpm
 - Blender temperature 80°C
 - Mixing time 3 min
- The resulting free-flowing compound has a temperature of about 60°C.

It can either be processed directly, e.g., by compression molding, or it can be added to conventional compounds.

Rubber recycling Standard Recipe

The test specimens were prepared by compression moulding of the powdered dry blend.

Table 4: Standard formulation of GTR recycling

Material	phr
GTR	100
VESTENAMER® solution 20%	15
ZnO	1.5
Stearic acid	0.5
Sulfur	0.8
CBS	0.8
TMTD	0.2

Table 7: Mixing of the compound

Mixer	Powder blender
Speed	500 rpm
Mixing Temperature	23 °C
Mixing Time	3 min.
Appearance of Compound	Powder dry blend

Table 5: Press Cure 20 min. 180 °C

Properties	Unit	Value
Hardness	Shore A	68
Tensile strength	MPa	4.7
Elongation at break	%	170
Modulus 100 % Elong	MPa	2.8
Rebound resilience	%	37
Compr. Set 70h 23 °C	%	20
Abrasion (DIN 53516)	mm ³	282
Density	g/m ³	1.18

Table 6: Hot air aging, 7d 100 °C

Properties	Unit	Value
Hardness	Shore A	77
Tensile strength	MPa	4.5
Elongation at break	%	82

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