



Ecorr[®] Reclaim in compounds

In the technical brochures for Ecorr[®] RBR and Ecorr[®] RNR a detailed overview is given of different applications in which a rubber reclaim can be used. Each virgin compound responds different when a part of the virgin rubber is replaced by rubber reclaim. Not only the physical properties but also the processing properties, such as vulcanisation behaviour, are influenced. If a reclaim is used in a compound, it is possible and / or necessary to optimise the total compound recipe. During the years, Rubber Resources has developed different techniques to minimise these effects. In this brochure examples are given of compounds in which the different parts of virgin rubber are replaced by rubber reclaim.



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Ecorr® RNR Natural Rubber Reclaim in truck tyre tread compound

Rubber Resources Ecorr® RNR reclaim can be added on top of an existing compound, with adjustment to the curing system. Also it can be used as a partial substitute for the natural and / or SBR rubber, with adjustment to rubber hydrocarbon content. It is important to notice that the rubber content of a rubber reclaim is about 50% and therefore 1 part of virgin rubber has to be replaced by 2 parts of rubber reclaim. In table 1 an example is given in which a truck tyre tread compound is used as a reference compound. Two types of natural rubber reclaim,

Ecorr® RNR B01 and Ecorr® RNR B11, are added on top of this compound with adjustment of the curing system. The substitution of virgin rubber with a reclaim is done for Ecorr® RNR B01. As can be seen from the examples, it depends on the Ecorr® RNR type and the method of addition to a compound. For concentrations up to 15 phr of reclaim, no difference in the properties of a truck tyre tread compound due to the compounding methods is observed. For higher concentrations compounding on top results in better properties, though the compound price reduction is lower.

	Reference compound	Ecorr® RNR B01 on top of reference compound	Ecorr® RNR B01 partial substitute of natural rubber	Ecorr® RNR B11 on top of reference compound
NR	100	100	97,5 94,8 89,6 84,4 73,5	100
Carbon black	50	50	50	50
Low PCA Oil	5	5	5	5
Zinc oxide	5	5	5	5
Stearic Acid	2	2	2	2
IPPD	1	1	1	1
TMQ	2	2	2	2
Par. Wax	2,5	2,5	2,5	2,5
Reclaim B01	0	5 10 20 30 50	5 10 20 30 50	5 10 20 30 50
TBBS	1,5	1,54 1,58 1,66 1,73 1,89	1,50	1,54 1,58 1,67 1,75 1,91
Sulphur	1,5	1,54 1,58 1,66 1,73 1,89	1,50	1,54 1,58 1,67 1,75 1,91

Table 1 Recipes for substitution or addition of reclaim in an existing compound.

Influence on physical properties

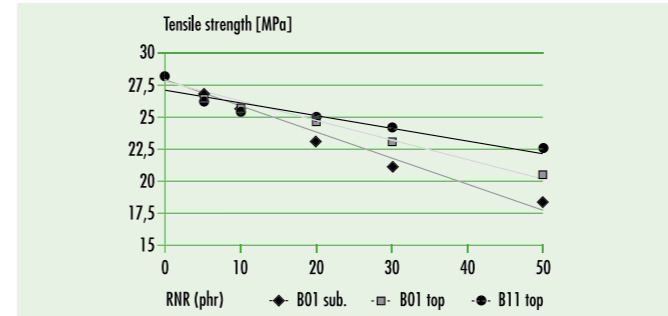


Figure 1

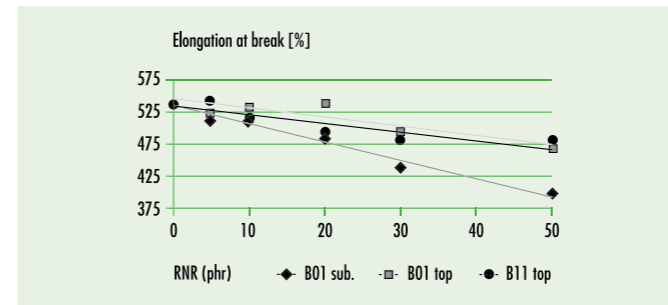


Figure 2

Figure 1 Comparison of tensile strength between addition of Ecorr® RNR B01 and B11 on top of an existing compound and substituting B01 reclaim for RHC (see table 1). Up to 10 phr, no difference between types of reclaim and way of compounding can be seen. At higher concentrations, adding on top results in higher tensile strengths.

Figure 2 Comparison of elongation at break between addition of Ecorr® RNR B01 and B11 on top of an existing compound and substituting B01 reclaim for RHC (see table 1). Here the same is true as for the tensile strength: Up to 10 phr no difference can be seen, above that, adding reclaim on top of the compound gives better results.

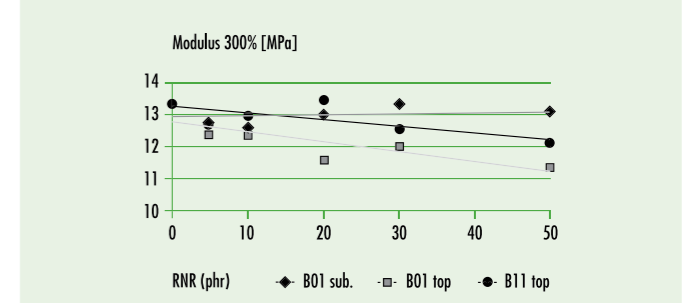


Figure 3

Figure 3 Comparison of modulus (300%) between addition of Ecorr® RNR B01 and B11 on top of an existing compound and substituting B01 reclaim for RHC (see table 1). Substitution for RHC gives more stable values.

Figure 4 Comparison of hardness between addition of Ecorr® RNR B01 and B11 on top of an existing compound and substituting B01 reclaim for RHC (see table 1). When adding B01 or B11 on top of the reclaim it provides comparable values as for the reference compound.

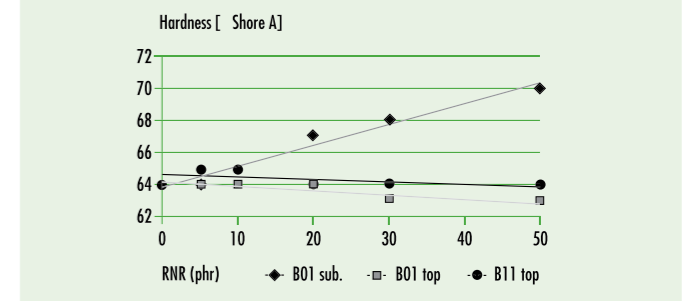


Figure 4

Figure 5 Comparison of Abrasion between addition of Ecorr® RNR B01 and B11 on top of an existing compound and substituting B01 reclaim for RHC (see table 1). Adding B11 on top of a compound results in the lowest increase.

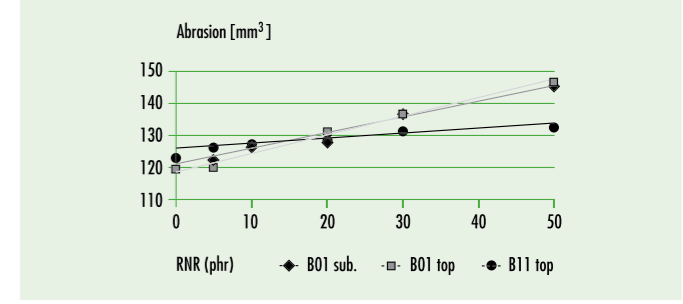


Figure 5



Figure 6 Comparison of tear strength between addition of Ecorr® RNR B01 and B11 on top of an existing compound and substituting B01 reclaim for RHC (see table 1). Here, adding on top results in the best values.

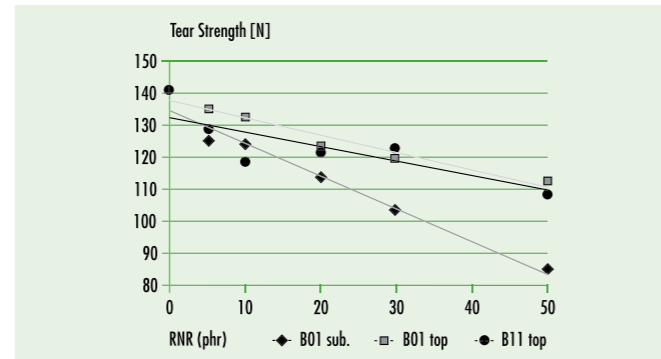


Figure 6

Figure 7 Comparison of the compression set between addition of Ecorr® RNR B01 and B11 on top of an existing compound and substituting B01 reclaim for RHC (see table 1). As expected adding on top results in the lowest values.

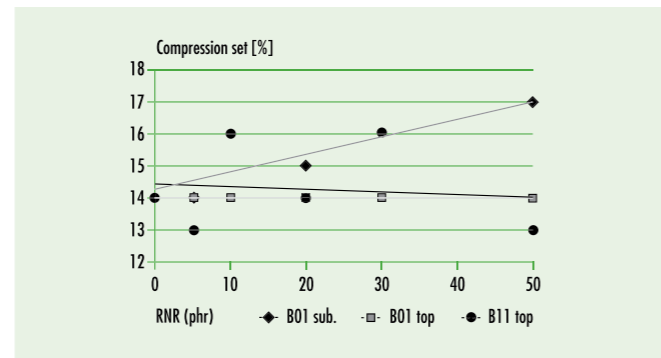


Figure 7

Figure 8 Comparison of rebound resilience between addition of Ecorr® RNR B01 and B11 on top of an existing compound and substituting B01 reclaim for RHC (see table 1). On top adding of reclaim has almost no influence on the rebound resilience.

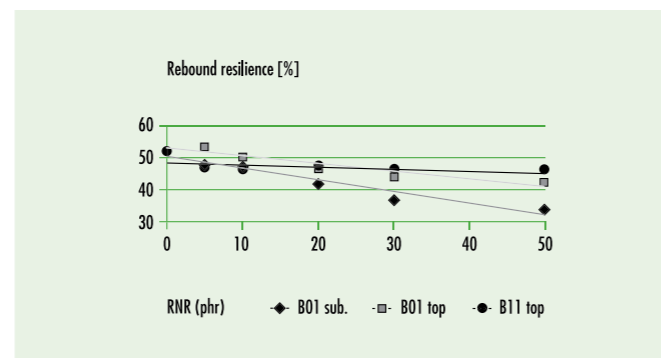


Figure 8

Figure 9 (After ageing: 14 days at 70 °C). Comparison of Tensile Strength (MPa) between addition of Ecorr® RNR B01 and B11 on top of an existing compound and substituting B01 reclaim for RHC (see table 1). Addition of reclaim improves ageing behaviour because the tensile strength drops less than the reference compound.

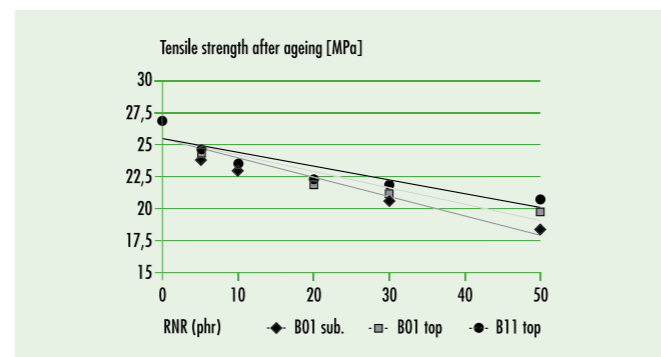


Figure 9

Ecorr® RNR Natural Rubber Reclaim in tyre tread compound

Besides the substitution of SBR rubber by a reclaim, also other changes to the compound recipe are possible and/or necessary. For example, introduction of reclaim generally leads to shorter vulcanisation times (decrease in ts2 and t90). In table 2, two possibilities are given to compensate this behaviour. First 0,5 phr of a Pre-Vulcanisation-Inhibitor (PVI) is added. It can be seen that this leads to an increase of ts2 and t90. On the other hand, by using this PVI, also the price of the total compound is increased. A more efficient way of increasing the vulcanisation time is by leaving out extra parts of carbon black and processing oil, for these materials are also present in the reclaim.

The last column shows the advantages of this method:

1. More comparable vulcanisation behaviour to the virgin reference compound.
2. An increase of several physical properties such as tensile strength, abrasion, tear strength.
3. Lower compound price due to further savings on carbon black and processing oil.

It must be noted that these are just two examples of compound optimisation. The most efficient method differs for each compound.

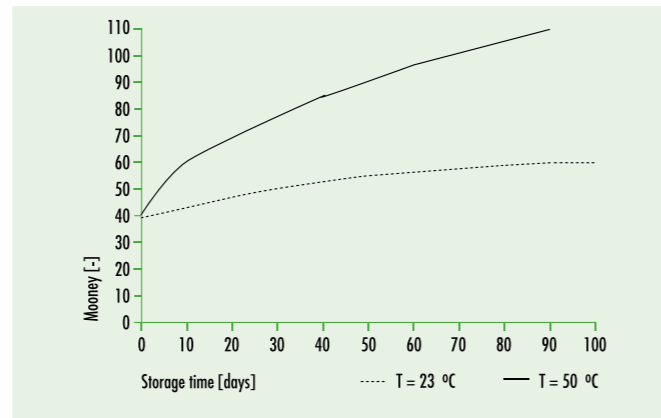
Table 2 In the first column the recipe and the properties of a tyre tread compound are given. In the second, the influence of interchanging 10 parts of the SBR rubber by 20 parts of B01 reclaim (n 50% rubber) is noted. In the last two columns two possible optimisation steps are given with the effect on the compound properties.

	Virgin Compound	+20 phr B01	+PVI	-PVI -15 phr CB -10 phr oil
SBR 1500	80	70	70	70
BR 1220	20	20	20	20
B01	-	20	20	20
ISAF N234	75	75	75	60
Low PCA oil	25	25	25	15
Stearic acid	1	1	1	1
Struktol 40 MS	3	3	3	3
TMQ	2	2	2	2
IPPD	2	2	2	2
Antilux 654	1.5	1.5	1.5	1.5
ZnO	5	5	5	5
TBBS	1.5	1.5	1.5	1.5
SU-109	2.4	2.4	2.5	2.4
PVI	-	-	0,5	-
Mooney	71	75	73	71
ML(1+4) @ 100 °C				
Rheo @ 160 °C	11	14	13	12
ML	68	69	69	71
MH	4'39"	3'51"	5'26"	4'54"
ts2	7'22"	6'02"	8'57"	8'21"
t50	10'20"	8'40"	11'25"	10'53"
t90				
Modulus (MPa)				
100%	1.7	2.3	2.1	2.0
200%	2.9	5.2	4.9	4.6
300%	6.9	9.3	9.0	8.4
400%	8.6	13.3	12.9	12.4
500%	11.7			
Tensile strength (MPa)	17.4	14.7	14.8	15.5
Elongation at break (%)	577	456	456	480
Hardness (° Shore A)	71	74	74	75
Density (g/cm3)	1.17	1.18	1.18	1.16
Din Abrasion (mm3)	81	106	82	75
Tear Delt				
⊥ [N]	62	60	57	65
// [N]	57	56	56	59

Storage hardening of Ecorr® RNR Natural Rubber Reclaim

The phenomenon of the hardening of natural rubber during storage is well known in the rubber industry. Due to the high proportion of natural rubber in tread reclaim, the hardening behaviour of reclaim during storage is similar to that of natural rubber.

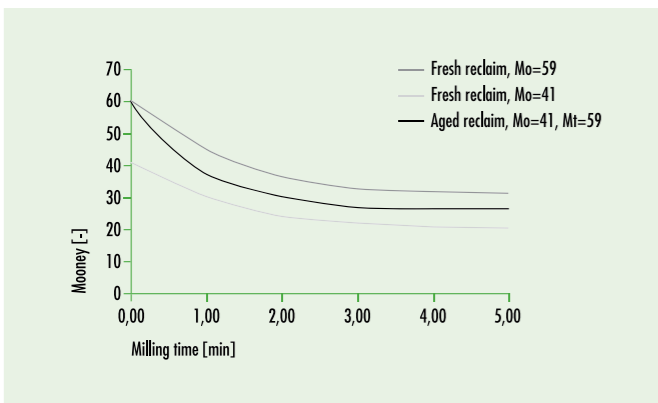
Figure 10 Increase in Mooney as a function of storage time and temperature (M₀ = 40)



The Mooney viscosity of tread reclaim increases with time due to physical and chemical processes. The increase is dependent on storage time and temperature (see figure 10). It can be calculated with the following empirical equation:

- M_t = Mooney after t days of storage
- M₀ = Mooney at the day of production
- t = storage time in days
- a = 0.04 for T = 23 °C
- a = 0.15 for T = 50 °C

$$M_t = M_0 * (1 + a * t^{0.55})$$



Increases in viscosity are largely reversible with a small amount of milling. As shown in figure 11, the viscosity of the aged reclaim decreases sharply during the first minute of milling; further the decline in viscosity is analogous to that of fresh reclaims. Afterwards, the aged reclaim behaves like fresh reclaim, having a much lower Mooney viscosity. Reclaim with an increase in Mooney viscosity of approximately 20 points can be masticated easily with a 5% increase in total mixing energy. The processing behaviour and the product properties of compounds with aged vs. fresh reclaim (having the same Mooney viscosity at the day of production) do not differ significantly.

Figure 11 Mooney as a function of time on a mill for fresh and aged reclaims.



Multiple reclaiming with Ecorr® RNR Natural Rubber Reclaim

Multiple reclaiming occurs when scrap rubber containing reclaim is itself reclaimed. Reclaim as a partial substitute is generally used in concentrations not higher than 20%. In this concentration range only slight differences in properties are caused by multiple reclaiming (see figure 8).

The tensile strength of a compound containing a percentage of reclaim can be described as follows:

$$T = T_0 (1 - \alpha) + T_1 (\alpha - \alpha^2) + \sum_{n=2}^{\infty} T_n \alpha^n$$

- T = Tensile strength of the compound containing a % reclaim
- T₀ = Tensile strength of the compound without reclaim
- T_n = Tensile strength of the reclaim after n reclaiming steps
- α = Percentage of reclaim in the compound

This equation is based on a linear relationship between the compound's reclaim content and the tensile strength of the reclaim as they relate to the resulting tensile strength of the compound. Figure 12 shows the effect of multiple reclaiming on the tensile strength of a compound containing reclaim, both calculated and measured values. The calculation of the tensile strength of this natural rubber compound containing 20% (= a) reclaim being reclaimed for the first time (n=1) will yield a tensile strength of 21,1 MPa:

$$T = 24,4 \text{ MPa} (1 - 0,2) + 7,9 \text{ MPa} * 0,2 = 21,1 \text{ MPa}$$

The tensile strength of the same compound containing recycled material which has undergone up to three reclaiming steps is 21,0 MPa:

$$T = 24,4 \text{ MPa} (1 - 0,2) + 7,9 \text{ MPa} (0,2 - 0,2^2 - 0,2^3) + 6,6 \text{ MPa} * 0,2^2 + 6,0 \text{ MPa} * 0,2^3 = 21,0 \text{ MPa}$$

Figure 12 The influence of multiple reclaiming on the properties of a compound

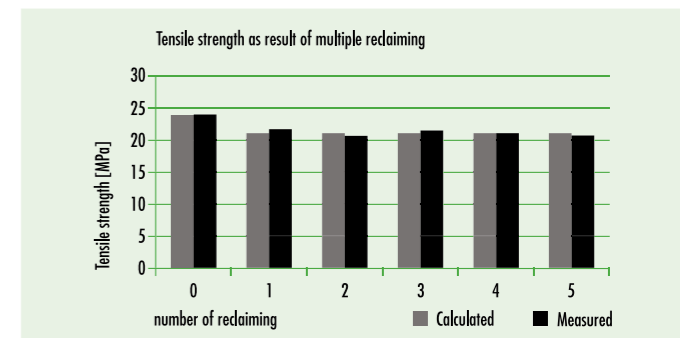


Figure 12



Ecorr® RBR70 butyl rubber reclaim in inner-liner compounds

Introduction

Most of the Ecorr® butyl reclaim is utilised by the tyre industry in BIIR- and CIIR-inner-liner applications. This part provides information on the influence of Ecorr® RBR70 butyl rubber reclaim on the most important properties of BIIR- and CIIR-inner-liner compounds.

The most important properties of an inner-liner include:

1. Low air permeability: butyl- and halobutyl rubber are characterised by very low gas permeability.

Chlorine or bromine is introduced in the allyl position to the double bond in order to increase the reactivity of the butyl rubber and to make it co-vulcanisable with NR and SBR.

2. Good ageing- and fatigue-resistance: as a result of crack initiation and crack growth, air can penetrate between the plies of the tyre causing ply separations. In the case of truck tyres, superior ageing performance is required due to higher running temperature and the need for good retreadability.

3. Good cured adhesion to NR: due to the low air permeability of the inner-liner compound, the formation of trapped air is possible. This trapped air can cause blisters between the inner-liner and the carcass ply, particularly at the end of the curing cycle when pressure in the bladder is reduced but vulcanisation is

not yet complete. During consumer use of the tyre, layer separations might result from these blisters.

4. Economic considerations: compound price as well as the possibility of reducing the inner-liner's thickness need to be considered.

The advantages of Ecorr® RBR70 butyl reclaim in an inner-liner compound:

The use of Ecorr® RBR70 butyl reclaim in bromobutyl- and chlorobutyl-inner-liner compounds provides several technical and cost benefits: The compound price can be reduced through the incorporation of butyl reclaim. Increasing the load of butyl reclaim reduces air permeability.

This allows for a reduction in thickness of the inner-liner resulting in additional cost savings and in a reduction in tyre weight. Flex fatigue resistance is improved. In bromobutyl-compounds, ageing resistance and green strength are improved. An increasing concentration of butyl reclaim improves air venting between bladder and inner-liner during vulcanisation.

Compound recipes

The following data were derived using two different compounds, one bromobutyl rubber based, and the other chlorobutyl rubber based. The two compounds are typical in tyre inner-liner applications. Mixing was performed in a 2,5 litre Homrich-laboratory mixer.

table3 The bromobutyl inner liner compound

Mixing step I (batch temperature: 135 °C)							
BIIR X 2	100,0						
Ecorr® RBR70	---	5,0	15,0	20,0	25,0	30,0	
Carbon black N 660	60,00						
Koresin resin	4,00						
Struktol 40 MS	7,00						
Paraffinic oil	8,00						
Stearic acid	2,00						
Maglite D	0,15	0,15	0,16	0,17	0,17	0,17	0,17
MBTS	1,50	1,50	1,60	1,65	1,70	1,75	
Mixing step II (Batch temperature: 90 °C)							
ZnO	3,00	3,10	3,20	3,30	3,40	3,50	
Sulphur	0,50	0,50	0,55	0,55	0,55	0,60	
Compound data							
Rheometer (ODR; 160 °C)							
t _{g2}	3'50"	3'20"	3'10"	3'05"	3'00"	2'55"	
t ₉₀	17'05"	17'20"	17'10"	17'10"	17'20"	17'55"	

table4 The chlorobutyl-inner liner compound

Mixing step I (batch temperature: 135 °C)							
CIIR 1066	70,0						
SMR20	30,00						
Ecorr® RBR70	---	5,0	10,0	15,0	20,0	25,0	30,0
Carbon black N 660	60,00						
Koresin resin	4,00						
Struktol 40 MS	7,00						
Paraffinic oil	8,00						
Stearic acid	2,00						
Mixing step II (Batch temperature: 90 °C)							
ZnO	3,00	3,08	3,17	3,25	3,33	3,41	3,50
MBTS	1,50	1,54	1,58	1,62	1,67	1,71	1,75
Sulphur	0,50	0,51	0,53	0,54	0,56	0,57	0,58
Compound data							
Rheometer (ODR; 160 °C)							
t _{g2}	3'10"	3'20"	4'25"	4'05"	4'25"	4'25"	4'10"
t ₉₀	9'30"	17'20"	11'20"	11'10"	11'30"	11'50"	11'30"

Bromobutyl rubber has a high cure rate and short scorch time. For a better process safety the BIIR-compound contains MgO and MBTS. Both chemicals are added during the first mixing step. The ZnO and sulphur should be added during the second mixing step.



Influence of Ecorr® RBR70 on the properties of the BIIR- and CIIR-compounds

Lower air permeability:

The addition of Ecorr® RBR70 butyl rubber reclaim results in a reduction in air permeability. This is one of the most important advantages for inner-liner applications.

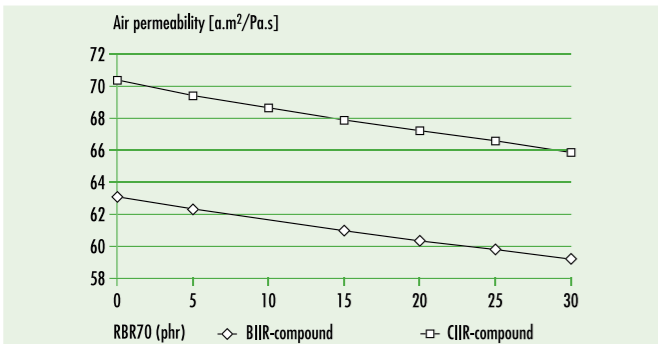


Figure 13 Influence of Ecorr® RBR70 butyl rubber reclaim on the permeability of a BIIR- and a CIIR- inner-liner compound (65 °C).

Improved ageing resistance of the BIIR-compound:

Ageing resistance is improved by the addition of increasing amounts of Ecorr® RBR70 butyl rubber reclaim, especially in the case of the BIIR compound. Figure 14 and 15 illustrate the effect on tensile strength and elongation at break after ageing at 125 °C for 72 hours in hot air.

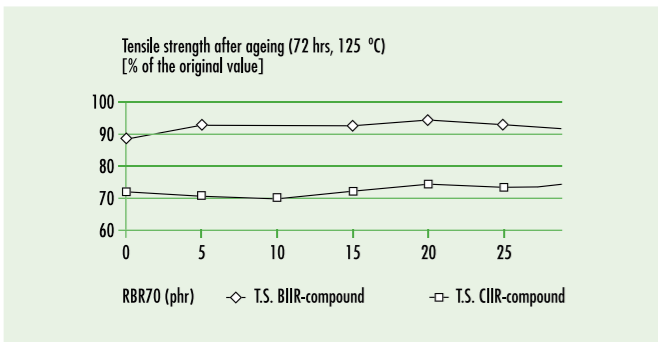


Figure 14 Influence of Ecorr® RBR70 butyl rubber reclaim on the tensile strength of a BIIR- and a CIIR- inner-liner compound after ageing (ASTM D 412).

Figure 17 Influence of Ecorr® RBR70 butyl rubber reclaim on the flex fatigue resistance of a CIIR- inner-liner compound (De Mattia, ISO 132).>>

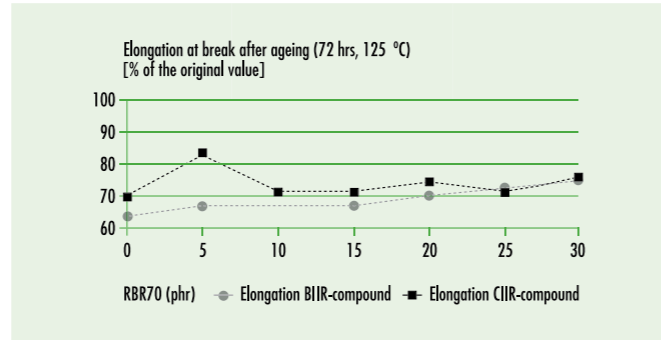


Figure 15 Influence of Ecorr® RBR70 butyl rubber reclaim on the elongation at break of a BIIR- and a CIIR- inner-liner compound (ASTM D 412).

Improved flex life after ageing:

Just the same as static properties, also dynamic properties can be improved by compounding with Ecorr® RBR70 butyl rubber reclaim (after ageing at 120 °C, 168 hours). The charts below illustrate the effect on the flex fatigue behaviour of BIIR- and CIIR compounds.

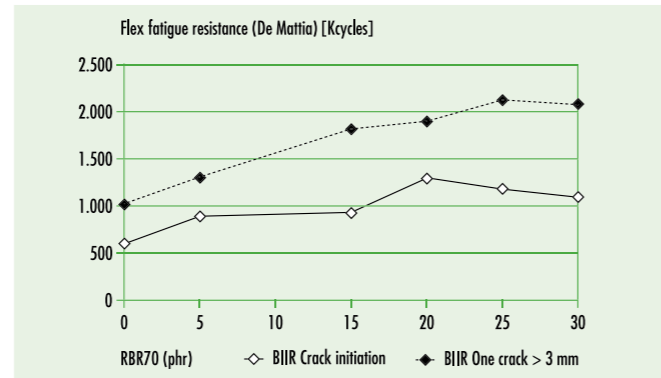
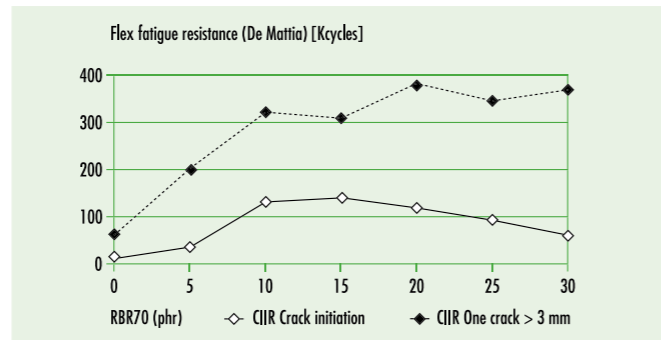


Figure 16 Influence of Ecorr® RBR70 butyl rubber reclaim on the flex fatigue resistance of a BIIR- inner-liner compound (De Mattia, ISO 132).



Improved cured adhesion to NR:

The addition of Ecorr® RBR70 butyl rubber reclaim results in stronger adhesion of halobutyl compounds to NR compounds in the cured state. This effect is more applied in the case of bromobutyl compounds, whose cured adhesion is stronger in comparison to the CIIR compounds.

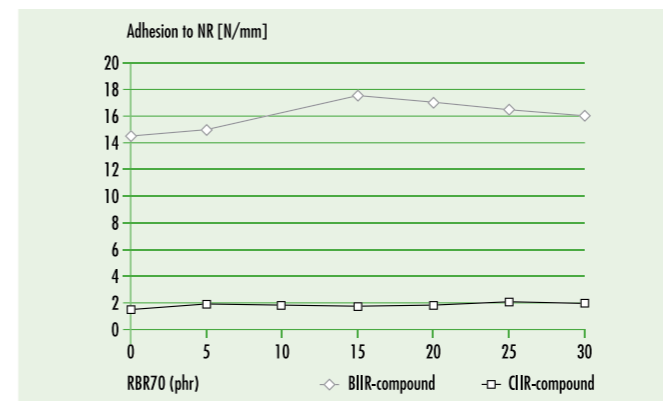
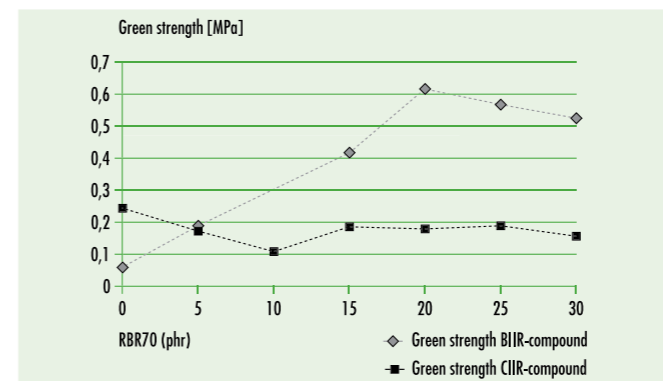


Figure 18 Influence of Ecorr® RBR70 butyl rubber reclaim on the adhesion of a BIIR- and a CIIR- inner-liner compound.

Improved green strength of BIIR-compounds:

The green strength of BIIR compounds is significantly increased by the addition of up to 20 phr Ecorr® RBR70 butyl rubber reclaim. With loadings higher than 20 phr green strength remains on this high level. Figure 19 also illustrates the influence on the green strength of a CIIR compound.



Cost savings:

Cost savings are achieved through lower compound costs, and due to the reduction of the inner-liner's thickness. The latter can be achieved by adjusting the permeability/ thickness ratio. The precise cost saving depends on the current price for material costs and compounding.

Other mechanical properties:

For the influence of Ecorr® RBR70 butyl rubber reclaim on the tensile strength and elongation at break see figures 20 and 21; for the effect on modulus and hardness see figures 22 and 23.

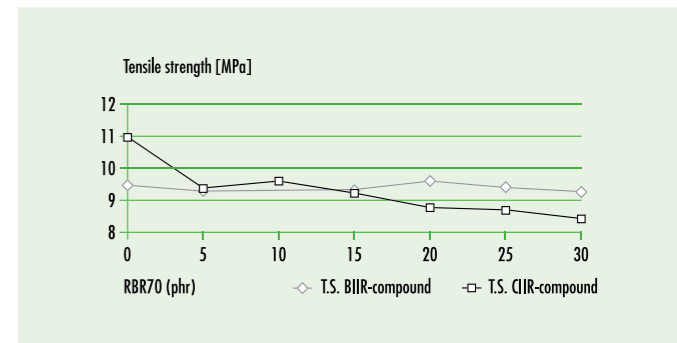


Figure 20 Influence of Ecorr® RBR70 butyl rubber reclaim on the tensile strength of a BIIR- and a CIIR- inner-liner compound (ASTM D 412).

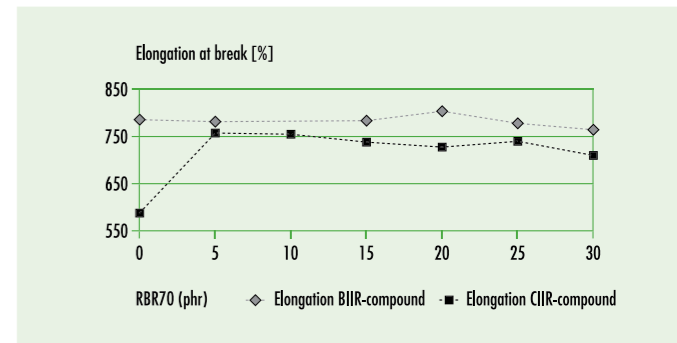


Figure 21 Influence of Ecorr® RBR70 butyl rubber reclaim on the elongation at break of a BIIR- and a CIIR- inner-liner compound (ASTM D 412).

<< Figure 19 Influence of Ecorr® RBR70 butyl rubber reclaim on the green strength of a BIIR- and a CIIR- inner-liner compound.

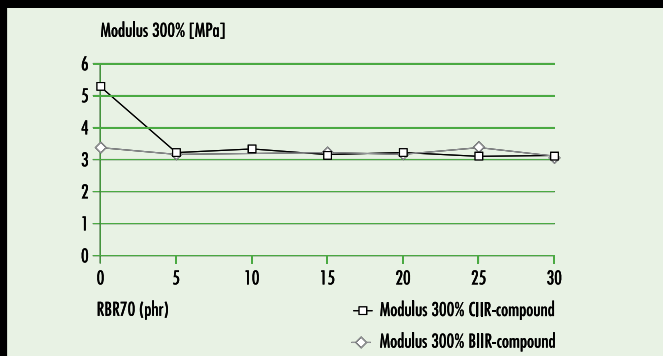


Figure 22 Influence of Ecorr® RBR70 butyl rubber reclaim on the modulus of a BIIR and CIIR- inner-liner compound (ASTM D 412).

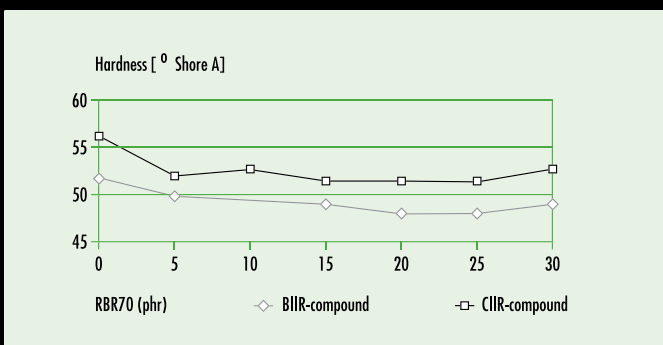


Figure 23 Influence of Ecorr® RBR70 butyl rubber reclaim on the hardness of a BIIR and CIIR- inner-liner compound (° Shore A, ASTM D 2240).

Influence on processability

No significant negative influence on the processability of the compound has been observed.

The following influences should be mentioned:

1. Tack is not significantly influenced.
2. Die-swell is reduced, but mill shrinkage is increased slightly.

Storage and handling of Butyl reclaim

1. During storage the temperature should be cool (< 23°C, preferably < 15°C), dry (relative humidity lower than 65%), dust free and moderately ventilated.
2. Reclaim should be protected from light, in particular from direct sunlight and strong artificial light with a high ultraviolet content.
3. Reclaim should be protected from circulating air by a protective packaging or by storage in airtight containers. Ozone concentration in the storage room should be below 10 ppm. Therefore the storage area should not contain any equipment capable of generating ozone.
4. Reclaim should be stored under conditions free from compression, tension or other possible sources of deformation.
5. Although the mooney increase for butyl reclaim is very low, the reclaim inventory should be rotated on a 'first in, first out' basis.

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