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EFFECT OF TALC ON ROLLING RESISTANCE IN TREAD COMPOUNDS

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Effect of Talc on Rolling Resistance in Tread Compounds

by

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ABSTRACT

The effect of talc on the rolling resistance in both carbon black and precipitated silica reinforced tire-tread compounds was evaluated. The results indicate that 5 phr of Mistron® Vapor R talc can be used as a dispersion aid to reduce the mixing time by 20% of the tire-tread stock *without influencing the dynamic properties at -30°, 0°, and 60°C or the mechanical properties*. Talc does not have a significant influence on the cure or viscosity in either system. Although talc has a slight effect on abrasion resistance of black compounds, it did not change the wear performance in precipitated silica reinforced vulcanizates. Talc did not affect the adhesion between the tread compound and the carcass.

INTRODUCTION

Talc is a natural occurring mineral. Pure talc is hydrous magnesium silicate consisting of a brucite sheet sandwiched between two silica sheets as illustrated in Figure 1. Talc has platy morphology as shown in Figure 2. Special micronizing techniques are used to preserve the aspect ratio of talc used in the rubber industry as this is critical to reinforcement, rheology, and permeability resistance.

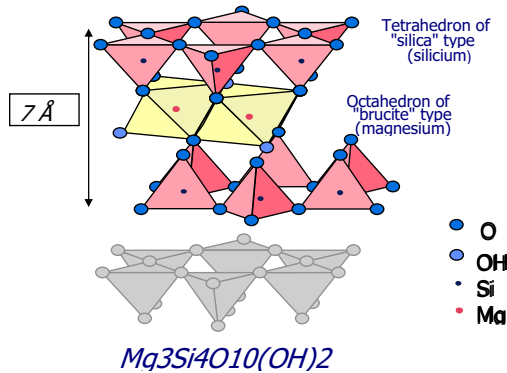


Fig.1 – Talc structure.

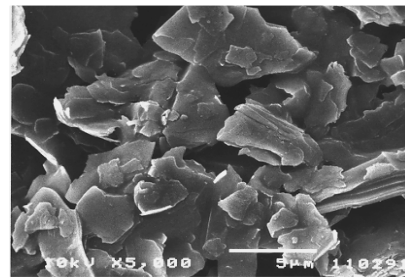


Fig. 2 – SEM of platy talc.

Although talc is used in inner liners^{1,2} and white side-walls², it has not been used in tread compounds. This was investigated in the late 1980's by Gonzalez-Herandez, Rueda and Anton in natural rubber truck tire tread.² It was observed that wear and heat build-up were adversely affected with 30% replacement of N330 with talc. The loss of wear performance was confirmed in the laboratory using Lambourne Abrasion Resistance at 25% slip ratio on a tread compound containing 20 phr of talc by Mouri and Akutagawa.³ In addition to this deficiency, talc is perceived as a filler by some rubber chemist. This is simply not true as demonstrated in Table I for a black side-wall compound where 40% of the carbon black is replaced with talc on an equal volumetric basis or as shown in the work of Gonzalez-Herandez, et al.²

Table I
Talc as a Reinforcement

N330	40	24
Talc	0	24
Wt. % N330 replaced	0	40
Tensile Strength, MPa	16.4	17.2
Elongation, %	586	642
100% modulus, MPa	2.10	1.64

The addition of talc also increases tear resistance and insensitivity to flaws of carbon black compounds due to crack diversion and the reduction of the stress concentration at the crack tip.⁴ This is demonstrated in the following table on tensile samples that were precut with a razor blade to a depth of 10 percent of their thickness and by the difference in topography of the failure surfaces in Figure 3.

Table II
Effect of Talc on Cut Sensitivity

N550	225	180	135
Talc	0	67.5	135
<i>Wt % N650 replaced</i>	0	20	40
Tensile Strength, MPa	5.70	9.33	8.53
Elongation @ break, %	121	236	304
Energy to Break, J	1.11	3.98	4.58

The combination of tear resistance and insensitivity to flaws results in increased toughness and durability.⁴ This should improve the cut and chip performance in tires.

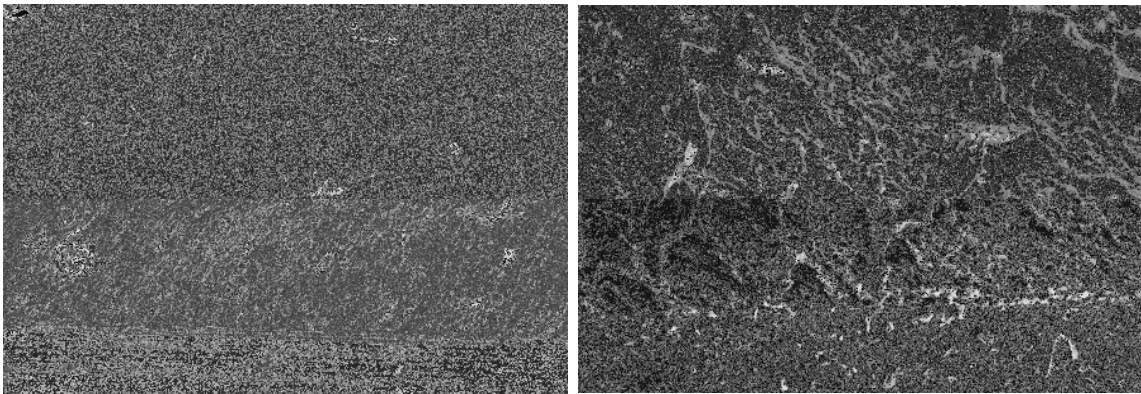


Fig. 3 – SEM of tear surface of tensile specimens without talc (left) and with talc (right) which were nicked on the edge with a razor blade.

The planar surfaces of talc are hydrophobic and are preferentially wetted by organic substances such as rubber. This translates to rapid incorporation of talc in rubber compounds as demonstrated in Table III.

Table III
Mill Mixing Study in SBR

Mineral Pigment	phr	Time (min)
Mistron Vapor R	100	6
Hard Clay	100	18
Precipitated Silica	50	24

This feature combined with the observation that talc functions as a homogenizing/compatibilizing agent⁵ in TPO as shown in Figure 4 fostered the concept of talc as a dispersion aid for reinforcing fillers in tire-tread compounds.

Due to the effect of talc on wear in tire treads, only 5 phr was used to determine if talc would improve the dispersion of carbon black and precipitated silica in tire-tread compounds. The results indicate that ultra-fine microcrystalline talc (Mistron® Vapor R), which has been used in the rubber industry for over 40 years, could be used to reduce the mixing time by 20% of the tire-tread stock without influencing dispersion or mechanical performance.⁶ Although generally accepted mixing procedures were followed in this work, there appeared to be a significant difference in the $\tan \delta$ at 60°C for the precipitated silica-reinforced compound due to the addition of talc.

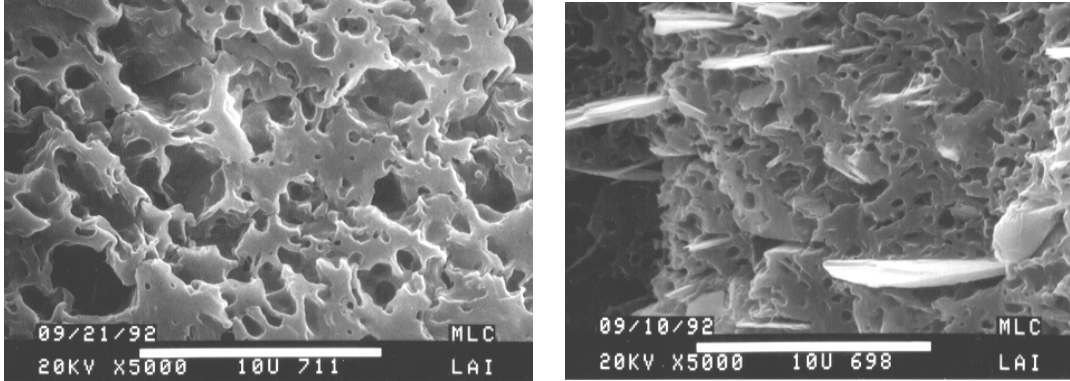


Fig. 4 – SEM of freeze-fractured surfaces of 50/50 blends of EPDM and PP after hexane extraction showing a significant size reduction of the holes with the addition of talc (right) vs. without talc (left).

This paper reviews the work done on talc as a dispersion aid⁶ including its effect on wear resistance, fatigue resistance, Mooney viscosity and cure. New data indicating that talc does not influence the rolling resistance ($\tan \delta$ at 60°C) of a “green” tire will also be presented. In addition, the influence of talc on the viscosity at shear rates encountered in production for extrusion is reported. The effect of talc on adhesion, which is critical in tire performance, is presented.

The production of ultra-fine talc requires significantly less energy than needed to manufacture carbon black or precipitated silica. For example, it has been estimated that the energy required is 20% of that used for precipitated silica.³ In addition, there is the potential with talc to reduce the power needed to mix a batch.⁶

EXPERIMENTAL

ARDL (Akron Rubber Development Lab) prepared a standard black tread compound as shown in Table IV according to mixing specifications outlined in Table XX in the Appendix. The mixing times for compounds 2b and 4b were reduced by 20% as shown in the last row of Table IV.

Table IV
Black Tread Formulations and Mixing Times

I.D.	1b	2b	3b	4b
RSS-1	80	80	80	80
Taktene 221PBD	20	20	20	20
N220	70	70	70	70
Mistron® Vapor R talc	-	-	5	5
Sundex 8125	12	12	12	12
<i>Mixing Time, min</i>	<i>13</i>	<i>10.5</i>	<i>13</i>	<i>10.5</i>

Note: cure system (phr) = stearic acid 2.5; ZnO 6; 6PPD 2.5; sulfur 1.5; and CBS 1.5

The Phillips dispersion was measured using an Olympus SZ60 Zoom Stereo Microscope at 30x magnification on vulcanized samples which were cut with a razor blade.

The following tests were conducted according to ASTM standards:

- Cure rheometry (ASTM D 2084-95)
- Mooney viscosity (ASTM D 1646-00)
- Tensile properties (ASTM D 412-98a)
- DeMattia flex (ASTM D 813)
- DIN abrasion (DIN 53516)
- Melt viscosity (ASTM D5099-08, Method A)
- Adhesion Peel test (ASTM D429 – 03e1)

In regards to the adhesion test, the standard 180° peel test results were extremely variable in spite of using fiber backing on the sample. The use of a thin metal plate in the bottom sheet and the initial peak values corresponding to 90° pull provided more consistent results.

The dynamic properties were determined in compression using a MTS Elastomer Test System at 10 Hz and a strain level of 7.5% with an amplitude of ± 2.5%.

The same experiment was performed using a “green” tire recipe. The mixing conditions are outlined in Table XXI of the Appendix. The recipe is presented in the following table. The mixing time was reduced by 20% from compounds 2g and 4g from 13 to 10.4 minutes as documented in Table V.

Table V
Green Tread Formulations and Mixing Times

ID	1g	2g	3g	4g
SSBR Buna VSL 5025-1	103.12	103.12	103.12	103.12
Taktene 220PBD	25	25	25	25
Zeosil 1165	80	80	80	80
Si 69	6.4	6.4	6.4	6.4
Mistron® Vapor R talc	-	-	5	5
Sundex 8125	5.13	5.13	5.13	5.13
<i>Mixing Time, min</i>	<i>13</i>	<i>10.4</i>	<i>13</i>	<i>10.4</i>

Note: cure system (phr) = stearic acid 2; ZnO 2.5; DPG 1.5; 6PPD 1.9; sulfur 1.1; and CBS 2

RESULTS AND DISCUSSION

Dispersion Rating

The addition of 5 phr of talc improved the dispersion of carbon black as shown in the following table and in Figure 5. For example, at a recommended mixing time of 13 minutes the Phillips dispersion rating was 9 for compound 3b vs. 6 for the control compound 1b. With 20% reduction in mixing time, the dispersion rating of compound 4b with talc is equal to the control compound.

Table VI
Dispersion Ratings for Carbon Black Reinforced Compounds

I.D.	1b	2b	3b	4b
MVR conc., phr	0	0	5	5
Mixing Time	T	T-20%	T	T-20%
Dispersion Rating	6	5 (with a size 1 agglomerate)	9 (with size 6 agglomerates)	6

- Notes: 1) Phillips dispersion rating standards range from 1 (poor) to 10 (excellent).
2) Mixing time T = 13 minutes.

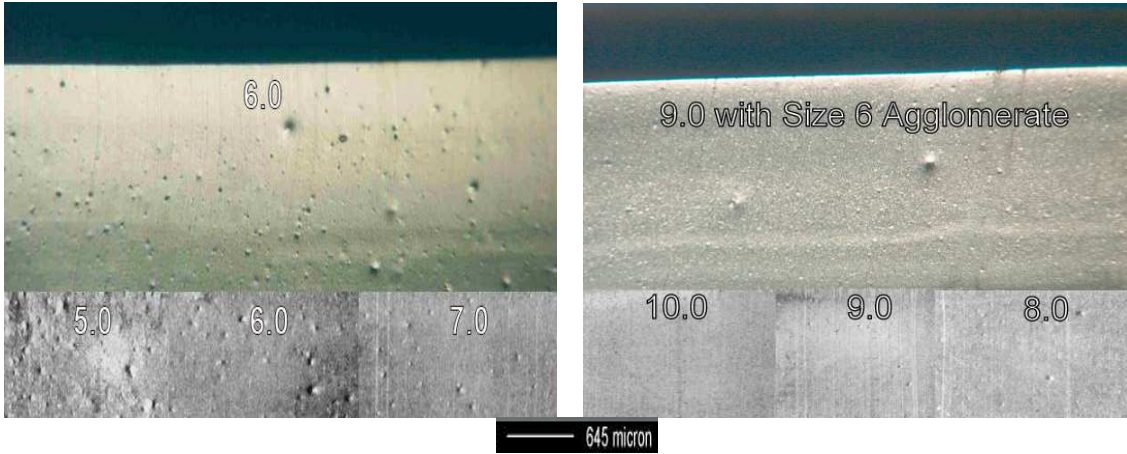


Fig. 5. — Reflected light image of specimens 1b (left) and 3b (right) at 30X with embedded Phillips Rating Charts.

Similar results were achieved with the “green tire” compound as shown in the following table and Figure 6. The Phillips dispersion rating was 6 for compound 3g containing talc vs. 5 for the control compound 1g at the recommended mixing time of 13 minutes.

Table VII
Dispersion Ratings for Precipitated Silica Reinforced Compound

I.D.	1g	2g	3g	4g
MVR conc., phr	0	0	5	5
Mixing Time	T	T-20%	T	T-20%
Dispersion Rating	5 (with a size 1 agglomerate)	5	6	5

Notes: 1) Phillips dispersion rating standards range from 1 (poor) to 10 (excellent).
2) Mixing time T = 13 minutes.

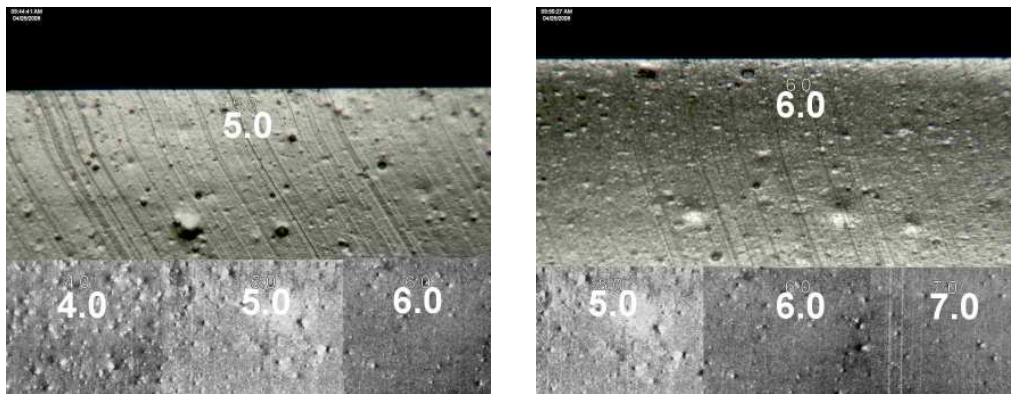


Fig. 6 — Reflected light image of specimens 1g (left) and 3g (right) at 30X with embedded Phillips Rating Charts. Note: bumps in 3g are actually shallow indentations.

The observations indicate that talc functions as a dispersion aid in both carbon black and precipitated silica reinforced compounds *to improve dispersion and reduce mixing time.*

Rheology and Cure

The addition of talc did not affect the Mooney viscosities as shown in the following table; however, a reduction in the mixing time and/or the degree of dispersion results in an increase in Mooney. Talc does not influence the cure characteristics as shown in Table IX.

Table VIII
Mooney Viscosity (CML 1+4 @100°C) for Black Tread Compounds

I.D.	1b	2b	3b	4b
Initial Viscosity, MI	120.90	140.10	125.00	127.80
Viscosity @ 4 minutes ¹	81.71	85.99	80.80	84.79

Note: Alpha Technologies MV 2000 viscometer per ASTM D 1646-00

Table IX
Cure Data for Black Tread Compounds

I.D.	1b	2b	3b	4b
ODR Cure Data¹ (3° arc @ 166°C)				
Maximum Torque, M _H , N-m	8.78	8.81	8.99	8.80
Minimum Torque, M _L , N-m	1.51	1.60	1.45	1.56
Cure Time, t ₅₀ , min.	3.22	3.29	3.20	3.23
Cure Time, t ₉₀ , min.	3.83	3.90	3.77	3.80
Scorch Time, t _{S1} , min.	2.14	2.19	2.15	2.16

Note: Tech Pro ODR per ASTM D 2084-95

In the case of the “green” tire compounds, the effect of mixing time on Mooney viscosity is significant. This could be attributed to high filler-filler interaction with reduced mixing time. With the addition of talc, the difference in viscosity due to mixing time is considerably less suggesting that talc reduces the filler-filler interaction of silica. It is proposed that the higher viscosity of 3g vs. 1g is related to improved dispersion of silica and/or reduction in the effects of mastication on the polymer due to talc.

Table X
Mooney Viscosity (CML 1+4 @100°C) for Green Tread Compounds

I.D.	1g	2g	3g	4g
Initial Viscosity, MI	98.2	167.8	111.4	119
Viscosity @ 4 minutes ¹	60.1	103	66.9	71.6

Note: Alpha Technologies MV 2000 viscometer per ASTM D 1646-00

Talc does appear to retard the cure slightly as determined by the t₉₀ as shown in the following table. The increases are not considered to be critical to production output. Talc did not influence the scorch time.

Table XI
Cure Data on Green Tread Compounds

I.D.	1g	2g	3g	4g
ODR Cure Data (3° arc @ 166°C)				
Maximum Torque, M _H , lb _f -in	72.3	80.2	76.3	76.1
Minimum Torque, M _L , lb _f -in	15.1	19.1	17.1	18.3
Cure Time, t ₅₀ , min.	5.69	5.18	4.95	5.21
Cure Time, t ₉₀ , min.	19.88	16.24	20.81	22.87
Scorch Time, t _{S2} , min.	2.29	2.23	2.25	2.26

Note: Tech Pro ODR per ASTM D 2084-95

In regards to processing, the viscosity of the “green” compounds was evaluated after each pass using a capillary rheometer. The apparent viscosity vs. shear rate data is presented in the following table.

Table XII
Apparent Viscosity vs. Shear Rate at 100°C

Pass Number			1		2		3	
Shear Rate, sec ⁻¹			100	1000	100	1000	100	1000
ID	Mixing Time	Talc, phr	Apparent Viscosity, Pa-sec					
1g	T	0	7297	1088	6947	925	5551	762
2g	T-20%	0	8634	1401	8113	1195	6599	865
3g	T	5	8361	1347	7980	1155	6476	861
4g	T-20%	5		-	8539	1305	6949	974

Notes: 1) L/D = 20/1 for the capillary
2) data is not corrected using the Rabinowitsch relationship or for entrance effects

Inspection of Table XII indicates that the viscosity is reduced after each pass. This is attributed to the additional thermo-mechanical exposure. In the cases where the mixing time is reduced, the compounds exhibit higher melt viscosity. This is consistent with reduction in mastication.

The viscosity increased with the addition of talc which was unexpected and has not been observed in other compounds. This increase could be attributed to better dispersion of the precipitated silica and/or reduction in the effects of mastication on the polymer due to talc. Although the increase in viscosity with talc ranges from 5 to 15%, this is not considered to be detrimental to the extrusion of the tread stock.

Tensile Properties

The effects of talc and mixing time on tensile properties at 23°C are shown in Table XIII for carbon black tread compounds. With the exception of the 50% and 100% moduli, talc does not affect the tensile properties. The mixing time also has no effect on the tensile properties which was unexpected but may simply reflect the insensitivity of the tensile test.

Table XIII
Mechanical Properties for Black Tread Compounds

Run ID		1b	2b	3b	4b
MVR conc., phr		0	0	5	5
Mixing Time		T	T-20%	T	T-20%
Tensile Strength, MPa.	Mn	25.36	25.32	24.92	25.17
	Std	0.621	0.517	0.724	0.262
Elongation @ break, %	Mn	442	450	434	444
	Std	10.8	12.0	13.6	6.1
50% Modulus, MPa.	Mn	2.12	2.21	2.43	2.43
	Std	0.063	0.046	0.054	0.034
100% Modulus, MPa.	Mn	4.10	4.17	4.50	4.47
	Std	0.126	0.091	0.134	0.083
300% Modulus, MPa.	Mn	17.99	17.78	18.18	17.93
	Std	0.184	0.352	0.346	0.148
Shore A Hardness		75	75	75	75

Notes: 1) Cure time and temperature for 4 samples were the same (4.5 minutes @ 166°C) per ASTM D182-94
2) Testing per ASTM D 412-98a. 3) abbreviations: Mn = mean or average; Std = standard deviation.

The effects of talc and mixing time on tensile properties at 23°C are shown in Table XIV for the “green” tire compound.

Table XIV
Mechanical Properties for Green Tread Compounds

Run ID		1g	2g	3g	4g
MVR conc., phr		0	0	5	5
Mixing Time		T	T-20%	T	T-20%
Tensile Strength, MPa.	Mn	20.76	19.19	22.19	21.55
	Std	1.287	1.388	1.684	0.737
Elongation @ break, %	Mn	391	300	370	361
	Std	13.5	13.1	18.0	7.1
50% Modulus, MPa.	Mn	1.30	1.52	1.55	1.57
	Std	0.016	0.028	0.012	0.040
100% Modulus, MPa.	Mn	2.23	3.07	2.92	2.99
	Std	0.043	0.088	0.027	0.085
300% Modulus, MPa.	Mn	13.56	-	16.23	16.35
	Std	0.338	-	0.325	0.384
Shore A Hardness		61	64	62	65

Notes: 1) Cure temperature was 166°C with cure times of 21 minutes for 1g, 18 for 2g, 22 for 3g and 24 for 4g per ASTM D182-94
2) Testing per ASTM D 412-98a. 3) abbreviations: Mn = mean or average; Std = standard deviation.

Inspection of the data indicates no statistical significant difference in tensile strength or elongation at break with the addition of talc at the recommended mixing time. There is a slight increase in modulus with talc. The mixing time has essentially no effect on the tensile properties when talc is present. On the other hand, without talc in the compound mixing time has a significant effect on both tensile strength and elongation at break. This may be attributed to poor dispersion of the precipitated silica.

DIN Abrasion

As indicated by the results in Table XV, it appears that there is a slight reduction in the wear performance even at 5 phr of talc in the black tread compound. In addition, mixing time also affects abrasion resistance.

Table XV
DIN abrasion on Black Tread Compounds

Run ID	1b	2b	3b	4b
MVR, phr	0	0	5	5
Mixing Time	T	T-20%	T	T-20%
Mean, cc	90	85	109	99
Std.Dev.	3.1	2.1	1.5	3.5

Note: 40 rpm and 10N load

On the other hand, talc does not affect the wear resistance of the precipitated silica compound as shown in the following table.

Table XVI
DIN abrasion on Green Tread Compounds

Run ID	1g	2g	3g	4g
MVR, phr	0	0	5	5
Mixing Time	T	T-20%	T	T-20%
Mean, cc	116	-	119	122
Std. Dev.	3.5	-	4.9	5.4

Note: 40 rpm and 10N load

Adhesion

The adhesion of “green” tread which is presented in Table XVII indicates no loss in adhesion with the addition of talc.

Table XVII
90° Peel Adhesion on Green Tread Compounds

Run ID	1g	2g	3g	4g
MVR, phr	0	0	5	5
Mixing Time	T	T-20%	T	T-20%
Mean, cc	123	140	143	140
Std. Dev.	10	16	18	15

Dynamic Properties

A comparison of the $\tan \delta$ at different temperatures for 1b (carbon black control) to 4b (5 phr of talc with a 20% reduction in mixing time) is shown in the following table. The differences are not statistically significant. This observation is consistent with the findings of Mouri and Akutagawa who used rebound resilience at room temperature to screen various mineral fillers for rolling resistance in black tread compound.³

Table XVIII
Tan δ vs. Temperature

Temperature, °C		-30	0	60
Run ID		Tan δ		
1b	Mn	0.5820	0.3560	0.2915
	Std	0.0141	0.0064	0.0120
4b	Mn	0.5909	0.3791	0.2912
	Std	0.0213	0.0143	0.0018

A comparison of the $\tan \delta$ at different temperatures for the “green” tire compounds in Table XIX indicates that talc does not affect the $\tan \delta$ at -30, 0, or 60°C.

Table XIX
Tan δ vs. Temperature

Temperature, °C		-30	0	60
Run ID		Tan δ		
1g	Mn	0.1359	0.7297	0.1488
	Std	0.0053	0.0068	0.0036
3g	Mn	0.1233	0.7431	0.1515
	Std	0.0053	0.0086	0.0128
4g	Mn	0.1324	0.7386	0.1468
	Std	0.0032	0.0056	0.0046

Notes: compression buttons cured 35 minutes at 166°C

The storage modulus and $\tan \delta$ are significantly affected by the efficiency of the silane coating on the precipitated silica.⁷ As the silane is added directly to the rubber batch, i.e., insitu, the batch temperature must reach 165°C in order for the silane to couple to the silica via a condensation reaction in order to prevent strong filler-filler interactions.⁷ It is believed that this was not achieved in our initial study⁶ and accounted for the significant difference in $\tan \delta$ at 60°C. This led to the conclusion that talc could not be used as a dispersion aid in “green” tread compounds. The results reported in Table XIX correspond to compounds where the batch temperatures reach 165°C.

CONCLUSIONS

The use of talc as a dispersion aid in “green” tire tread compound to increase the speed of incorporation and improve dispersion of the precipitated silica does not affect the rolling resistance as determined by $\tan \delta$ at 60 °C. Talc did not alter the ice or wet traction as indicated by the $\tan \delta$ at -30 and 0°C, respectively. The addition of talc caused a slight retardation in the cure and an unexpected increase in viscosity. Talc addition had no influence on the tensile strength or elongation at break but increased the modulus. Without talc in the batch, a reduction in mixing time significantly lowered both the tensile strength and elongation at break. Talc did not affect abrasion resistance or adhesion.

As in the “green” tire tread compound, the mixing time of black tread-compounds can be reduced by 20% with the addition of 5 phr of Mistron® Vapor R talc to the formulation without affecting the cure, viscosity, tensile or dynamic properties of the compound. The addition of talc has a slight effect on abrasion resistance but improves DeMattia flex fatigue.

In conclusion, talc can function as a dispersion aid in both carbon black and precipitated silica reinforced compounds to improve dispersion in order to reduce mixing time *without altering* the dynamic properties related to ice and wet traction and *rolling resistance*.

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APPENDIX

Table XX

Mixing Specifications for Black Tread Compound

I.D.	1b and (3b)	I.D.	2b and (4b)
Time	1st Pass	Time	1st Pass
0.0	½ rubber + ½ black + stearic acid + 6PPD + (MVR) + ½ rubber	0.0	½ rubber + ½ black + stearic acid + 6PPD + (MVR) + ½ rubber
1.5	½ black + oil	1.0	½ black + oil
3.0	sweep down	2.0	sweep down
4.0	sweep down	3.0	sweep down
5.0	sweep down	4.0	sweep down
6.0	Dump @ 160°C	4.8	Dump @ 160°C
	2nd Pass		2nd Pass
0.0	½ masterbatch + ZnO + ½ masterbatch	0.0	½ masterbatch + ZnO + ½ masterbatch
2.0	sweep down	1.0	sweep down
3.0	sweep down	2.0	sweep down
4.0	Dump	3.2	Dump
	3rd Pass		3rd Pass
0.0	½ masterbatch + cure + ½ masterbatch	0.0	½ masterbatch + cure + ½ masterbatch
1.0	sweep down	1.0	sweep down
2.0	sweep down		
3.0	Dump @ 104°C	2.5	Dump @ 104°C

Notes: 1) Mixing done in a BR Banbury as follows: 1st pass at 77 rpm without cooling water to rotors and chamber; 2nd pass at 60 rpm without water; and 3rd pass at 50 rpm with cooling water. 2) The batches were dumped on a two-roll mill at 54°C and milled as follows: After the 1st and 2nd Banbury pass, the band was cut 3 times and end rolled 3 times. After 3rd pass, the band was cut 5 times and end rolled 5 times followed by one minute on mill to set grain before sheeting off the compound. Milled material was allowed to cool at room temperature for 2 hours between Banbury passes. 3) bold () indicate additive added to specific run, i.e., **(3b)** contains **(Mistron Vapor R)**

Table XXI

Mixing Specifications for Green Tread Compound

I.D.	1g and (3g)	ID	2g and (4g)
Time	1st Pass	Time	1st Pass
0.00	polymer	0.00	polymer
0.50	1/3 silica	0.50	1/3 silica
1.50	1/3 silica	1.00	1/3 silica
2.50	1/3 silica + rest of ingredients with exception of ZnO+ (MVR talc)	1.50	1/3 silica + rest of ingredients with exception of ZnO + (MVR talc)
4.00	sweep	2.50	sweep
5.00	sweep	3.50	sweep
6.00	Dump (@ 165°-177°C)	4.80	Dump (@ 165°-177°C)
	2nd Pass		2nd Pass
0.00	Masterbatch + ZnO	0.00	Masterbatch + ZnO
1.50	Sweep	1.50	Sweep
4.00	Dump (@ 138°-143°C)	3.20	Dump (@ 138°-143°C)
	3rd Pass		3rd Pass
0.00	1/2 masterbatch	0.00	1/2 masterbatch
0.50	curatives + 1/2 masterbatch	0.50	curatives + 1/2 masterbatch
1.50	sweep	1.50	sweep
3.00	Dump (@ 104°C)	2.40	Dump (@ 104°C)

Notes: 1) Mixing done in a BR Banbury as follows: 1st pass at 77 rpm without cooling water and steam combined to control temperature; 2nd pass at 77 rpm without water; and 3rd pass at 77 rpm with cooling water.

2) The batches were dumped on a two-roll mill as follows: After the 1st and 2nd Banbury pass, the batch was simply sheeted out with maximum possible nip thickness. After 3rd pass, the band was cut 3 times. Milled material was allowed to cool at room temperature for 2 hours between Banbury passes.

3) bold () indicate additive added to specific run, i.e., **(3g)** contains **(Mistron Vapor R)**