Paper 93

EFFECT OF TALC ON THE DISPERSION OF CARBON BLACK

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Effect of Talc on Dispersion of Carbon Black

ABSTRACT

The effect of talc on the dispersion of various grades and blends of carbon blacks was evaluated. The addition of talc improved the dispersion of semi-reinforcing grades of blacks and blends thereof. The effect was independent of structure and mixing procedure. On the other hand, the talc did not appear to effect the degree of dispersion of high surface-area reinforcing blacks. This response was, however, influenced by the choice of elastomers. A theory is proposed to explain the effect of talc on dispersion.

The synergism of talc with various grades and blends of carbon black was demonstrated by only minor changes in mechanical properties. This observation indicates that talc is not simply a filler in rubber compounds but rather a functional mineral.

INTRODUCTION

Carbon black is the most commonly used reinforcing filler in the rubber industry today. The grade of carbon black is characterized by surface-area and structure. The choice of carbon black is based on mechanical requirements, rheological considerations, and elastomer choice. For example, the higher the surface area of the carbon black, the more reinforcing it is. This is consistent with the fact that the interaction between carbon black and the polymer is mainly of a physical nature and is attributed to Van der Waals forces.[1] The structure of the carbon black also influences the rheological behavior of the unvulcanized material as well as the tensile properties.

Both reinforcement and rheology are affected by the degree of dispersion of the carbon black. In other words, the better the dispersion the better the processing and mechanical properties. Dispersion is dependent upon the shear stress imposed on the material during mixing. Shear stress is a function of viscosity of the material and shear rate. The viscosity of a compound is dependent upon the temperature. Therefore, higher temperatures in the mixer results in lower viscosity and lower shear stresses. Shear rate is a function of fluid velocity (rotor speed) and position (channel vs. tip). This would imply that higher rotor speed should improve dispersive mixing. The temperature of the batch, however, increases proportionally to the square of the rotor speed. Therefore, lower rotor speeds may be more beneficial to dispersion; however, this must be balanced against the mixing time. Dispersive mixing requires sufficient time for each carbon black agglomerate to encounter sufficient shear force in order to rupture. This time can obviously be reduced by mixing at higher speeds. Such is the dilemma.

It was observed that the addition of 5 phr of ultra-fine talc improved the dispersion of carbon black in tire tread compounds.[2] This allowed mixing times to be reduced by 20 percent without affecting the mechanical performance.[2] This reduction in mixing time has been confirmed commercially.

It has also been shown that the partial replacement of carbon black and/or oil with talc has little, if any, effect on mechanical properties and improves processibility.[3] This is attributed to synergism between talc and carbon black. Although talc exhibits synergy with clay and silica, it has less affect on hardness. This feature allows the compounder to increase the filler content to reduce the volume percentage of polymer and/or the oil.

In this paper, we studied the effect of talc on the dispersion of various grades and blends of carbon black. The influence of surface-area and structure of the carbon black were examined.

The talc used in this study was Mistron® Vapor R which is an ultra-fine microcrystalline talc. Special micronizing techniques are used to preserve the aspect ratio of this product which is critical to its functions in rubber such as improved processing.

EXPERIMENTAL

ARDL (Akron Rubber Development Lab) prepared rubber compounds with various compositions using a BR Banbury. The mixing sequence and conditions are provided within the text.

The following types of carbon blacks and blends were used:

1) different surface areas with the same structure (Group 1)
2) different structures with the same surface-area (Group 2)
3) reinforcing blends
4) reinforcing/semi-reinforcing blends
5) semi-reinforcing blends

In addition, a blend of two semi-reinforcing blacks was used to evaluate standard vs. upside down mixing to determine the effectiveness of talc on dispersion.

The Phillips dispersion was measured using an Olympus SZ60 Zoom Stereo Microscope at 30x magnification interfaced with a Polaroid DMC-ES digital camera on vulcanized samples which were cut with a razor blade. The un-aged mechanical properties were determined per ASTM D 412-98a. The samples were cured according to data from ODR rheometer per ASTM D 2084-95.

RESULTS and DISCUSSION

It has been documented that the partial substitution of Mistron® Vapor talc for carbon black improves processing and performance.[3] One such processing benefit is improved dispersion of reinforcing filler which provides the option of reducing mixing time to improve output.

This study is divided into several segments to evaluate the effect of talc on the dispersion of various grades and blends of carbon black. In the first two segments, those carbon blacks labeled Group 1 (G1) and Group 2 (G2) in Figure 1 were used. In Group 1, the surface areas range from 40 to 120 m^2/gm with the same structure. In Group 2, the reverse is true, i.e., the surface area is constant at 80 m^2/gm and the structure varies by a factor of two.

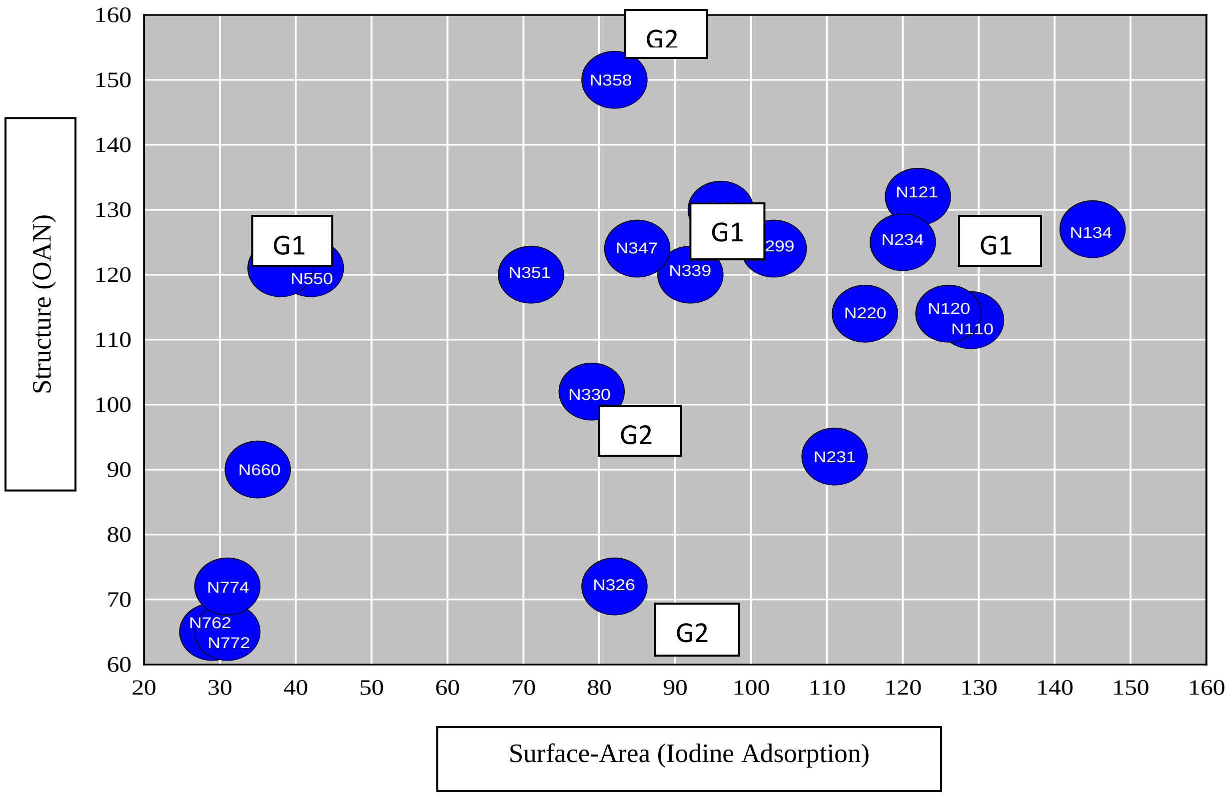


Fig.1.– Structure & Surface-Area chart for grades of carbon black (provided by Sid Richardson Carbon and Energy)

Surface-Area

In this segment of the study, the effect of talc on the mechanical properties and dispersion of carbon black with different surface-areas was evaluated. These carbon blacks which compose Group 1 are N550, N339, and N234 with surface-areas ranging from 40 to 120 m^2/gm. Their structure ratings are approximately the same.

As shown in Table I, twenty parts of talc was simply added to the control compounds. All of the compounds were one-pass mixed in a BR Banbury with a sweep at 82ºC and dropped at 104ºC. The ram pressure was 40 psi.

Table I
Formulation used in the evaluation of surface-area

Dutral 4038 EPDM	100	100	100	100	100	100
N234	82	82				
N339			93	93		
N550					100	100
Mistron® Vapor talc		20		20		20
Sunpar 2280	60	66*	60	66*	60	66*

Note: Cure package (in phr) ZnO 5; stearic acid 1.5; RM sulfur 1.5; ZDBC 1.5; MBTS 1.5; TMTD 0.5
* Sunpar 2280 oil was adjusted for all compounds containing talc to achieve equal hardness

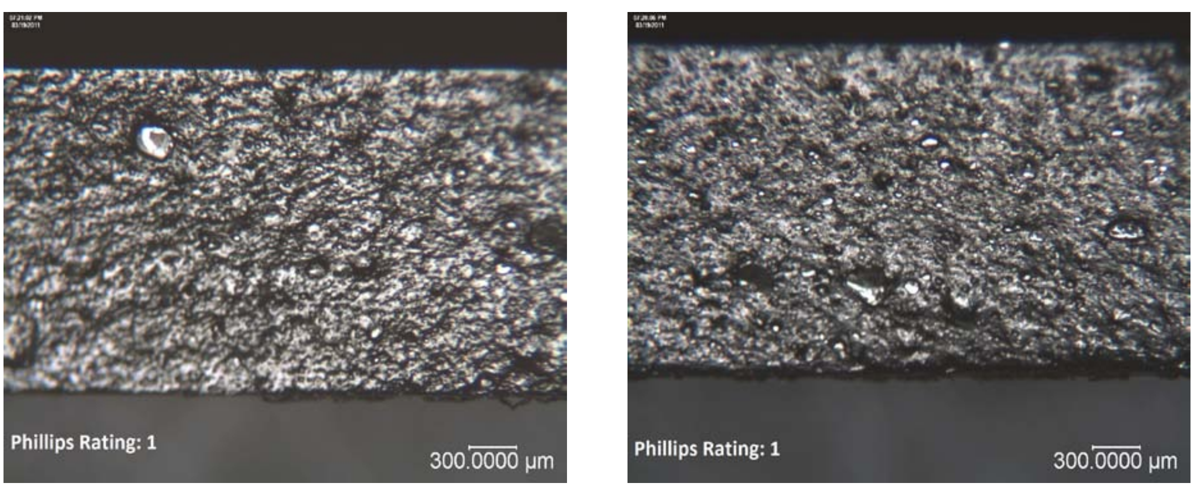
The comparison of mechanical properties as shown in Table II indicates that the addition of talc results in an increase in the elongation at break and a loss in modulus above 50 percent elongation. These results were anticipated based on previous studies.[3] The addition of talc also influenced the dispersion of N550 but had no affect on the higher surface-area blacks. The dispersion appears to be inversely proportional to the surface area.

Table II
Tensile properties and dispersion rating

Carbon black grade	N234	N234	N339	N339	N550	N550
Compound I.D.	control	Talc	control	Talc	control	talc
Tensile Strength, MPa	13.66	13.38	15.31	15.03	13.45	14.00
50% Modulus, MPa	1.42	1.59	1.54	1.71	1.84	2.01
100% Modulus, MPa	2.39	2.31	2.62	2.66	3.50	3.41
300% Modulus, MPa	8.66	7.31	9.86	8.64	9.21	8.72
Elongation @ break, %	423	468	427	464	448	504
Durometer, points	70	70	70	69	73	73
Phillips Rating	1	1	2	2	4	6

Note: Phillips dispersion rating 1 (worst) to 10 (best)

Inspection of the Phillips micrographs in Figures 2 and 3 indicate that the shear stresses during mixing were inadequate to disperse the high surface-area carbon blacks. This could be attributed to particle-particle attraction forces of these blacks and/or their small particle size.[5] Higher shear stresses may be required to disperse the agglomerates of high surface-area blacks in this elastomer.



Without Talc With Talc

Fig. 2. – Phillips micrographs of N234 compound with and without talc

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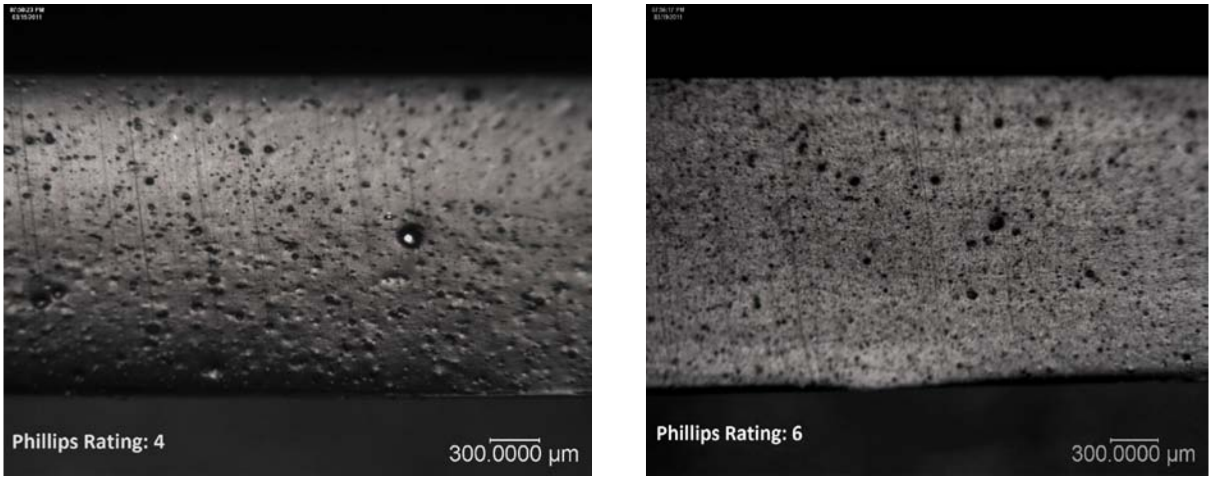


Without Talc With Talc

Fig.3.– Phillips micrographs of 550 compound with and without talc.

Structure

In the second phase of this study, the effect of talc on the dispersion of carbon blacks with equal surface areas but different structure was evaluated. The carbon blacks of Group 2 (N326, N330, and N358) have a surface area of approximately 80 m^2/gm. The structure, however, varies by a factor of two. The compounds were mixed using the same criteria as Group 1.

Table III
Formulations used in the evaluation of structure

Dutral 4038 EPDM	100	100	100	100	100	100
N326	102	102				
N330			93	93		
N358					77	70
Mistron® Vapor talc		20		20		20
Sunpar 2280	60	62*	60	66*	60	66*

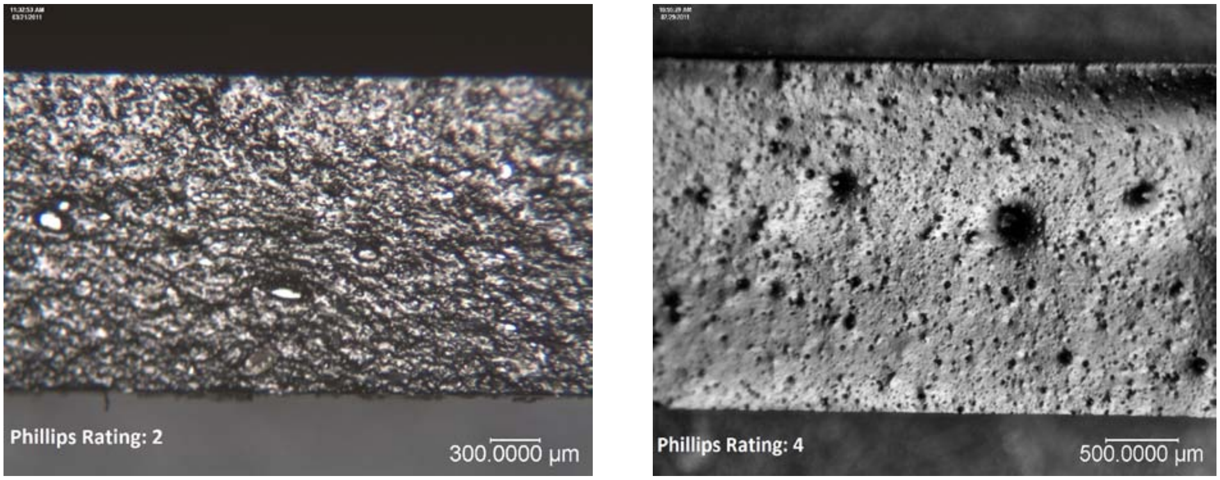
Note: Cure package ZnO (in phr) 5; stearic acid 1.5; RM sulfur 1.5; ZDBC 1.5; MBTS 1.5; TMTD 0.5.
* Sunpar 2280 oil was adjusted for all compounds containing talc to achieve equal hardness.

Inspection of Table IV reveals that the addition of talc improves dispersion of all three carbon blacks as measured by the Phillips dispersion rating (see Figures 4 and 5). This would indicate that structure is not a significant factor. It is, however, interesting to note that the carbon blacks in Group 2 have a surface area which is halfway between that of N550 at 40 m^2/gm and N234 at 120 m^2/gm. This would suggest that there is a critical surface-area above which talc has little, if any, influence on dispersion. These results may have been compromised due to the addition of oil.

Table IV
Tensile properties and dispersion rating

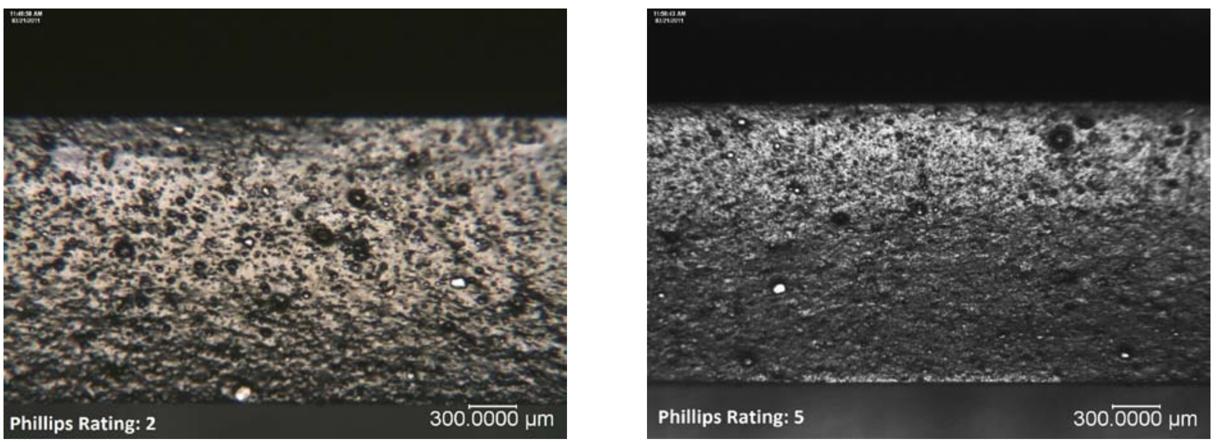
Carbon black grade	N326	N326	N330	N330	N358	N358
	control	Talc	control	talc	control	Talc
Tensile Strength, MPa	12.08	15.75	12.68	14.10	14.25	17.14
50% Modulus, MPa	1.81	1.59	1.63	1.82	1.73	1.59
100% Modulus, MPa	2.66	2.38	2.74	2.86	3.10	2.61
300% Modulus, MPa	7.53	7.23	9.37	9.06	10.94	8.90
Elongation @ break, %	436	501	396	438	375	495
Durometer, points	71	69	71	72	69	68
Phillips Rating	2	4	1	2	2	5

In regards to mechanical properties, the increase in tensile strength would not normally be anticipated. It is proposed that the higher values reported in Table IV are due to improved dispersion. The tensile modulus results for the N326 and N358 compounds are typical, but those for N330 are not.



Without Talc   With Talc

Fig.4.– Phillips micrographs of N326 compound with and without talc.



Without Talc   With Talc

Fig.5.– Phillips micrographs of N358 compound with and without talc.

Blends

Even though there are more than 20 ASTM grades of carbon black as well as numerous specialty grades, rubber compounders may still need to blend different grades in order to achieve the desired properties of the rubber compound. To evaluate the effect of talc on the dispersion of carbon black blends, we mixed the following: (a) two reinforcing blacks, (b) a reinforcing black with a semi-reinforcing black, and (c) two semi-reinforcing blacks.

Reinforcing Black Blends. For the reinforcing carbon black blends, we chose N220 and N330. A blend of NR/SBR/BR was used in this evaluation. The formulations are listed in Table V. Both compounds were one-pass mixed. The polymer was broken down for one minute and then all of the other raw materials were added to the mixer. A sweep was conducted at 99ºC, and the batch was dropped at 104ºC. The batches were cooled down and sheeted out on a two-roll mill. This simplified mix specification was used to accentuate any dispersion differences.

Table V
Formulation for blends of reinforcing blacks

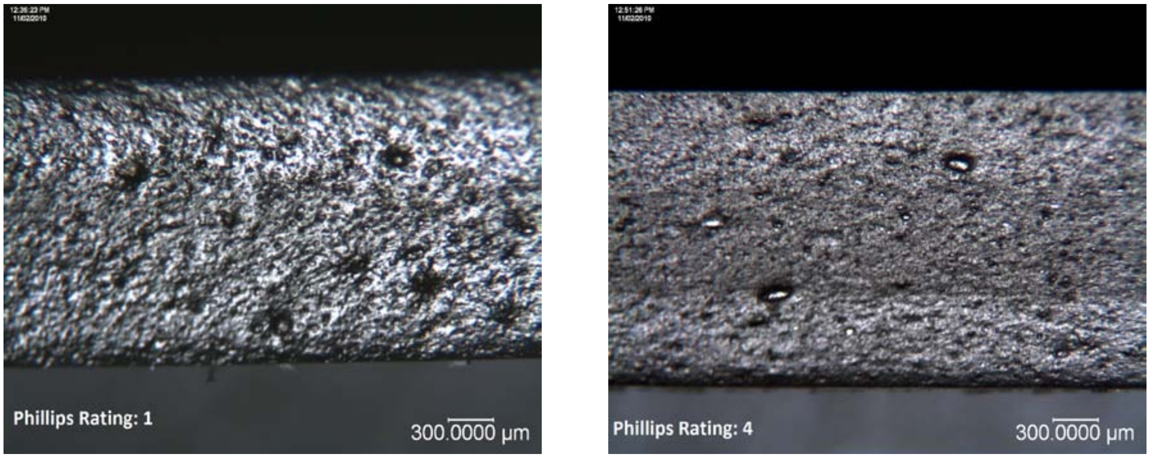
Components	Control	Control + talc
SIR 20	25	25
Budene 1208	37	37
SBR 1502	38	38
N220	15	15
N330	47	45*
Mistron® Vapor talc	-	30
Aromatic Oil	32	32

Notes: cure package (in phr) – ZnO 5; stearic acid 1.5; sulfur 2; CBTS 1.0; DOTG 0.25
*N330 was adjusted to achieve the same durometer

The mechanical properties and Phillips dispersion are shown in the following table. The Phillips micrographs are presented in Figure 6. The effect of talc on the dispersion of this blend of reinforcing blacks is impressive with an increased rating from 1 to 4. The improvement in tensile strength can be attributed to better dispersion and the synergism of talc with carbon black. The higher elongation at break with talc is typical.

Table VI
Tensile Properties and Phillips Rating on blends of reinforcing blacks

Property	Control	Control + talc
Tensile Strength, MPa	14.54	15.45
100% Modulus, MPa	2.06	2.41
300% Modulus, MPa	6.65	6.43
Elongation @ break, %	562	617
Durometer, points	60	60
Phillips dispersion rating	1	4



Without Talc With Talc

Fig.6.– Phillips micrographs of N220/N330 blends with and without talc.

Reinforcing and Semi-Reinforcing Black Blends. For the reinforcing and semi-reinforcing carbon black blends, we chose an EPDM compound and used N330 for the reinforcing black and N550 as the semi-reinforcing carbon black. Both compounds were one-pass mixed using upside down sequence. A sweep was performed at 82ºC and the batches were dropped at 104ºC. The batches were cooled down and sheeted out on a two-roll mill. This simplified mixing procedure was used to highlight any dispersion differences. The formulas are listed in Table VII.

Table VII
EPDM formulation for blends of reinforcing and semi-reinforcing blacks

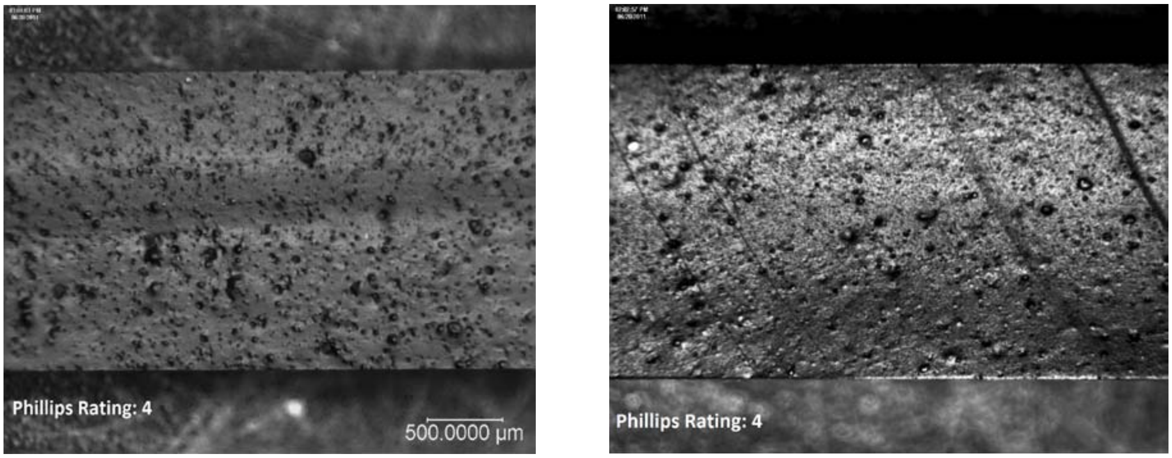
Component	Control	Control + talc
Dutral 4038 EPDM	100	100
N330	50	50
N550	50	50
Mistron® Vapor talc	-	20
Paraffinic Oil	60	66*

Notes: cure system (in phr) – ZnO 5; stearic acid 1.5; RM sulfur 1.5; ZDBC 1.5; MBTS 1.5; TMTD 0.5
*oil was added to the compound containing talc to achieve equal hardness.

The mechanical properties and the Phillips dispersion results are listed in Table VIII. Although the addition of talc improved the tensile properties, it did not affect dispersion. This is not surprising as N330 failed to disperse in the same elastomer in our previous experiment. This suggests that non-dispersed aggregates are primarily N330.

Table VIII
Tensile Properties and Phillips Rating on blend of reinforcing/semi-reinforcing blacks

Mechanical property	Control	Control + talc
Tensile Strength, MPa	13.76	14.29
50% Modulus, MPa	1.68	1.88
100% Modulus, MPa	3.02	3.21
300% Modulus, MPa	9.83	9.54
Elongation @ break, %	409	444
Durometer, points	71	71
Phillips rating	4	4



Without Talc With Talc

Fig.7.– Phillips micrographs of N330/N550 blends with and without talc.

Semi-Reinforcing Black Blends. For the semi-reinforcing carbon black blends, we chose a blend of N550 and N774. The EPDM compounds shown in Table IX were mixed upside down in one pass. A sweep was performed at 88ºC, and the batches were dropped at 104 ºC. The batches were cooled down and sheeted out on a two-roll mill.

Table IX
EPDM formulation semi-reinforcing black blends

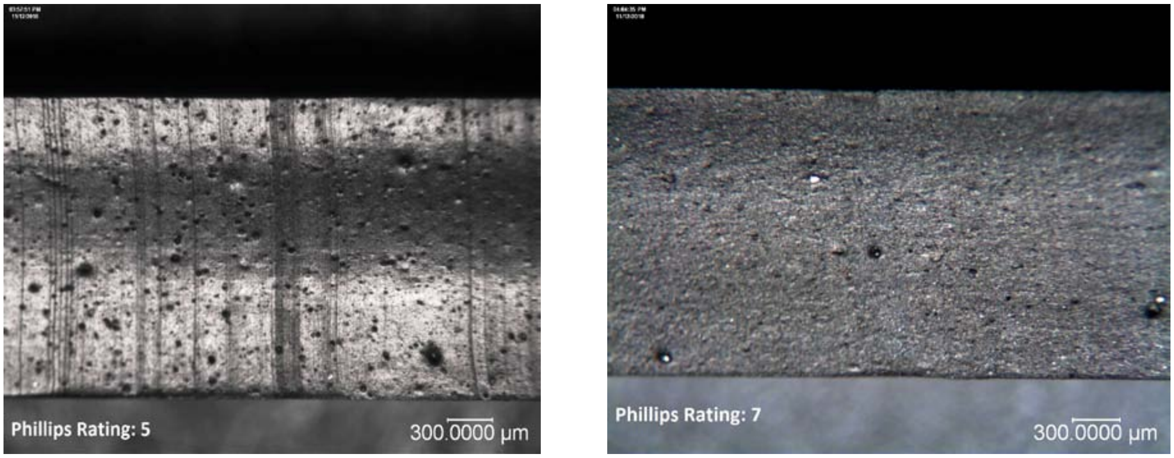
Component	Control	Control + talc
EPDM oil extended	130	130
N550	40	40
N774	35	35
Mistron® Vapor talc	-	60
Paraffinic Oil	10	20*

Note: cure package (in phr) – ZnO 5; stearic acid 1.5; accelerated 0.5; RM sulfur 1.5; MBTS 1.0
*additional oil was added to the talc compound to obtain the same durometer

The high loading of talc (60 phr) lowers the tensile strength by approximately 5 percent. The lower modulus at higher elongation is expected as well as the higher elongation at break. Talc does improve the dispersion of the blend as indicated in the last row in Table X. This was anticipated based on the previous observations on carbon blacks with low surface areas. The Phillips micrographs are shown in Figure 8.

Table X
Tensile Properties and Phillips Rating on blend of semi-reinforcing blacks

Mechanical Property	Control	Control + talc
Tensile Strength, MPa	15.79	14.80
100% Modulus, MPa	2.60	2.73
200% Modulus, MPa	5.71	4.78
300% Modulus, MPa	8.47	6.92
Elongation @ break, %	498	585
Durometer, points	65	66
Phillips dispersion rating	5	7



Without Talc With Talc

Fig.8.– Phillips micrographs of N550/N774 blends with and without talc.

Mixing Procedures

In this segment, tests were run to determine the effect of different mixing procedures on the dispersion of semi-reinforcing black blends. The formulas and mix variation are shown in the following table.

Table XI
Formulations and mixing procedures

Raw Material, phr	Control 1	Control 2	Control 2 + talc	Control 3	Control 3 + talc
EPDM oil extended	130	130	130	130	130
N550	40	40	40	40	40
N774	35	35	35	35	35
Mistron® Vapor talc	-	-	60	-	60
Paraffinic Oil	10	10	20*	20	20*
Mix Variation	2-Pass Standard	1-Pass Standard	1-Pass Standard	1-Pass Upside Down	1-Pass Upside Down

Note: cure system (in phr) – ZnO 5; stearic acid 1.5; Accelerator 0.5; RM sulfur 1.5; MBTS 1.0
*oil was adjusted to equalize the durometer

The Control 1 and 2 compounds differ only in mixing procedures as shown in Table XI. For example, Control 1 was a two-pass standard mix while Control 2 was only a one-pass. The effect of talc was evaluated using a one-pass standard as well as a one-pass upside down mix. The mixing specifications are shown in Appendix A.

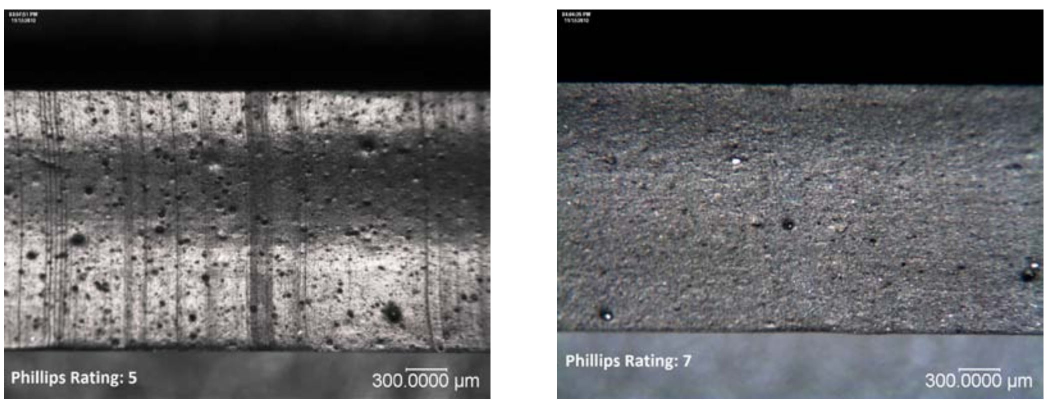
Table XII
Mechanical properties and Phillips dispersion vs. mixing technique

Mechanical properties	Control 1 (2-Pass Standard)	Control 2 (1-Pass Standard)	Control 2 + talc (1-Pass Standard)	Control 3 (1-Pass Upside Down)	Control 3 + talc (1 Pass Upside Down)
Tensile Strength, MPa	17.03	16.77	13.74	15.79	14.08
100% Modulus, MPa	2.32	2.47	2.73	2.60	2.73
200% Modulus, MPa	5.46	5.75	4.78	5.71	4.78
300% Modulus, MPa	8.35	8.70	6.94	8.47	6.92
% Elongation	546	550	552	498	585
Durometer, points	62	62	67	64	66
Phillips rating	5	5	6	5	7

The effect of talc and mixing procedures are shown in Table XII. The following observations can be made:

1. there is no difference between single and two-pass standard mixing on properties or dispersion
2. upside down mixing has no effect on properties or dispersion
3. talc improves the dispersion of semi-reinforcing black blends in upside down mixing.

In addition, all three control compounds were rated a 5 on the Phillips Dispersion scale regardless of the mix procedure. Although the addition of talc improved the dispersion as shown in Figure 9, no improvement in mechanical properties was observed. It is, however, note-worthy that even at 60 phr of talc that the tensile properties were not degraded. This observation indicates that talc is not simply a filler.



Without Talc   With talc

Fig.9.– Phillips micrographs of comparing control with and without talc mixed upside down.

THEORY

It is known that talc reduces the heat generation in rubber processing.[4] This decreases the rate at which the temperature of the compound increases during mixing. The lower temperatures in the talc-filled compounds results in higher viscosity and, thus, higher shear stresses which are necessary for dispersive mixing. This is demonstrated by the slope of the temperature traces in Figure 10 and tabulated in Appendix B. The rate of temperature rise (as indicated by the slope in Appendix B) for all the talc-filled compounds is less than the corresponding controls. For example, the temperature of the N550 control increased at a rate of 89ºC/min vs. 56ºC/min for N550 with talc. In addition to above, the initial viscosities of the talc-filled compounds are higher which further contributes to the dispersion.

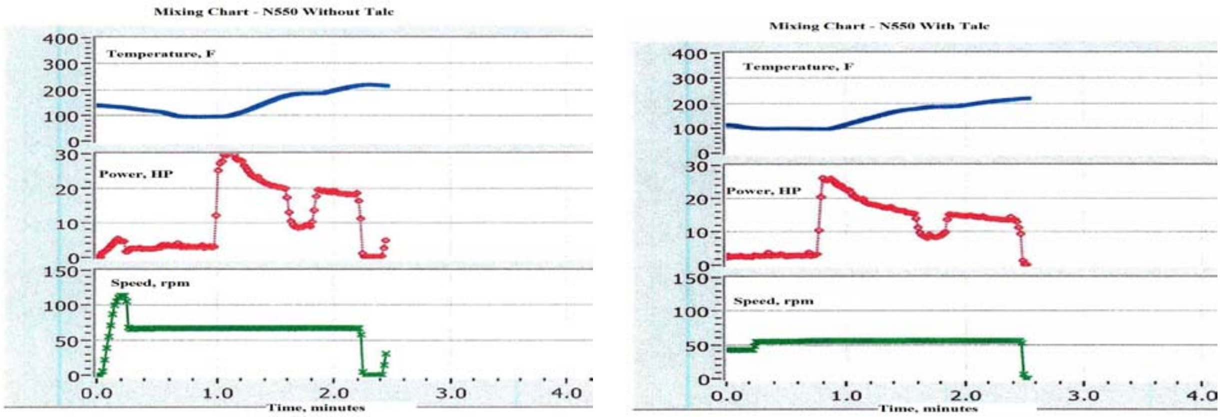


Fig. 10.–Comparison of mixing traces of N550 with and without talc.

It is proposed that the presence of ultra-fine platy talc increases the dispersion. This is supported by the higher tan δ observed for un-vulcanized talc-carbon black compound in Figure 11.[4] Furthermore, this is consistent with the RPA results published by Alpha Technologies indicating that higher tan δ values of uncured compounds correspond to a higher state of mix and hence better dispersion.[6] It is also hypothesized that talc prevents the re-agglomeration of carbon black in the rubber compound.

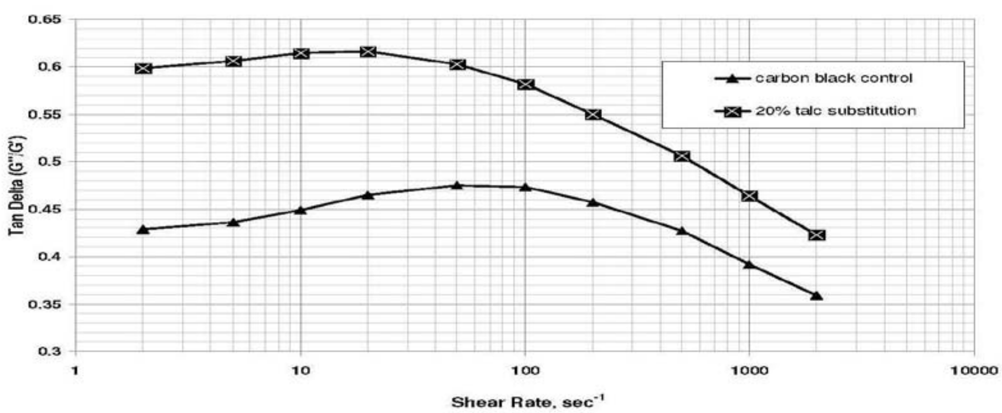


Figure 11.–RPA sweeps at 7% strain and 100°C for a un-vulcanized EPDM compound.

In regards to the minimal effect of talc on high surface-area blacks, the shear stresses were simply inadequate to disperse these blacks. This can be attributed to particle-particle attraction forces of these blacks and/or their small particle size.[5] Other contributing factors might be the formulation, mixing conditions and tolerances in the mixer.

The lower torques (see Appendix B) encountered with talc-filled compounds may be the result of talc lubricating the metal surfaces of the mixer during the initial phase of mixing. This is apparent from the plateau or pause in the torque after the ram is lower (see Figure 10).

In an attempt to better understand the improved dispersion of carbon black with talc, the following additional studies are being considered: partial substitution instead of addition, effect of talc concentration, effect of elastomer system and oil loading.

CONCLUSIONS

This study has shown that the addition of talc aids in the dispersion of semi-reinforcing blacks with surface areas of less than or equal to 80 m^2/gm and blends thereof. The dispersion of median surface-area carbon blacks, however, appears not to be dependent on structure. On the other hand, the addition of talc does not affect the degree of dispersion of high surface-area reinforcing blacks in EPDM but exhibited a significant effect in a NR/SBR/BR blend. This indicates that elastomer choice can be an important factor. Finally, the mixing procedure has no affect on the dispersion rating of semi-reinforcing blends.

It is proposed that the improvement in carbon black dispersion with the addition talc is due to the reduction in heat generation during processing. This results in higher viscosity and increased shear stresses. In addition, it is hypothesized that talc increases the spacing of carbon black aggregates and prevents their re-agglomeration.

The synergism of talc with various grades and blends of carbon black is demonstrated by minor changes in mechanical properties even at loadings of 60 phr. This observation indicates talc is not simply a filler in rubber compounds, but rather a functional mineral.

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APPENDIX A

Mixing Specifications

Type	Instructions
Standard 2-Pass	Polymer was broken down for 1 minute, N550 was then added, at 1:45 minutes all other raw materials were added. The mixer was swept at 127ºC, and the batch was dropped at 149ºC. The compound was cooled and remixed sandwiching the cure between the masterbatch. The batch was swept at 88ºC and dropped at 104ºC. Ram pressure 40 psi.
Standard 1-Pass	The polymer was broken down for 1 minute; N550 was then added; at 1:45 minutes the rest of the raw materials were added; the batch was swept at 88ºC and dropped at 104ºC. Ram pressure 40 psi.
Upside Down 1-Pass	All powder and oil raw materials were added to the mixer with the polymer being the last ingredient. The batch was swept at 88ºC and dropped at 104ºC. Ram pressure 40 psi

APPENDIX B

Mixing Data

I.D.	Max Torque	Temp @ max torque	Torque @ sweep (82ºC)	Time to sweep (min)	Slope (ºC/min)	Total mix time (min)	Power (kW-hr)	Phillips Dispersion Rating
N550	30	38	20	0.5	89	1.1	0.3442	4
N550 + talc	26	38	16	0.8	56	1.6	0.3668	6
N358	30	38	24	0.4	111	1.0	0.3500	2
N358 + talc	27	38	20	0.5	89	1.0	0.3280	5
N339	26	38	16	0.6	74	1.6	0.3825	2
N339 + talc	25	38	16	0.6	74	1.4	0.4114	2
N234	27	38	19	0.5	89	1.1	0.3732	1
N234 + talc	24	38	16	0.6	74	1.8	0.3999	1
220/330	20	82	15*	0.5	22	1.1	0.4585	1
220/330+talc	17	77	14*	1.1	15	2.0	0.5515	4

Note: * torque at 93ºC sweep temperature; dump temperature 104ºC for all compounds in the above table