

Comparative Study of Carbon Black and Silica as Reinforcing Fillers in Natural Rubber Compounds

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Abstract—This work evaluates the performance of carbon black and precipitated silica as reinforcing fillers in a natural-rubber compound, comparing the industry-standard carbon black system against the increasing use of silica in “Green Tire” technology. Because silica is polar and hydrocarbon rubber is not, silica-filled compounds require a silane coupling agent to achieve adequate filler–rubber interaction and dispersion. Three formulations were prepared: P1 (100% carbon black, N220), P2 (a 50:50 carbon black–silica blend with silane), and P3 (100% silica with silane). Rheometric testing at 150 °C showed that maximum torque fell progressively from P1 to P3, indicating lower crosslink density as silica content increased, while cure delay (Ts2, Tc90) lengthened in the same order. Physical testing showed that elongation at break rose with increasing silica content (P3 > P2 > P1) while 100/200/300% modulus, hardness, and abrasion resistance all declined. Rebound resilience was marginally higher for the silica-containing compounds. The reduced crosslink density associated with silica’s acidic surface chemistry is identified as the principal cause of these property shifts. Matching crosslink density across formulations is recommended for a fairer comparison of the two filler systems in future work.

I. INTRODUCTION

Rubber compounding draws on a wide range of ingredient classes - vulcanizing agents, accelerators and activators, antidegradants, processing aids, reinforcing fillers, inert fillers, and special-purpose materials - selected according to the physical properties, processing behaviour, and cost targets of the finished product [1]–[6]. Among these, reinforcing fillers exert the greatest influence on the mechanical performance of the vulcanizate [7].

Carbon black has long been the default reinforcing filler in rubber compounding, chemically active

toward the polymer and well understood in terms of particle size, structure, and surface activity [8]–[13]. Precipitated silica (SiO₂), by contrast, has grown in importance because of its ability to reduce rolling resistance and improve wet grip in tyre treads - the basis of “Green Tire” technology [14]–[17]. Unlike carbon black, silica’s surface is dominated by polar silanol groups, which makes it poorly compatible with non-polar hydrocarbon rubbers [18]–[23], slows cure, and hinders dispersion [24]–[28]. Bifunctional organosilane coupling agents (e.g., Si69/X-50S) are used to bridge this incompatibility: one functional group reacts with the silanol groups on the silica surface [29],[30], the other reacts with the rubber during vulcanization, forming a covalent filler–rubber bond that restores reinforcement [31]–[35].

The silica-silane system widens what is often called the “magic triangle” of tyre performance - rolling resistance, wet grip, and abrasion resistance - properties that are otherwise difficult to improve simultaneously with carbon black alone [36]. Because the silanization reaction (siloxane bond formation with cleavage of ethanol) occurs during mixing, the internal mixer effectively becomes a chemical reactor rather than a simple homogenizing device, and mixing conditions must be tightly controlled [37]–[39]. This study was undertaken to quantify how progressively replacing carbon black with silica (with proportional silane addition) changes rheometric, mechanical, and abrasion behaviour of a natural rubber compound, and to identify the underlying causes of the differences observed [40]–[44].

II. MATERIAL AND METHODS

1. Material

The base polymer was natural rubber (NR, RSS-2). The reinforcing filler system was varied across three compounds using N220 carbon black and precipitated silica, with a silane coupling agent (X-50S) introduced in proportion to the silica loading. Common compounding ingredients - process oil, zinc oxide (ZnO), stearic acid, and the antidegradant 6-PPD - were held constant across all formulations. The vulcanization system consisted of sulfur and the

accelerator CBS (N-cyclohexyl-2-benzothiazole sulfenamide).

2. Compound recipe

Three formulations were compounded to compare filler systems at equal total loading philosophy: P1 used carbon black only, P2 used a 50:50 carbon black-silica blend, and P3 used silica only, with silane dosed proportionally to silica content in P2 and P3. The master-batch (M-1) and final batch (F, including sulfur and CBS) formulations, in parts per hundred rubber (phr), are given in Table 1.

Table 1. Formulation of the carbon black / silica compounds (phr).

Ingredient (phr)	P1	P2	P3
NR (RSS-2)	100.00	100.00	100.00
N220 (carbon black)	40.00	18.00	—
Precipitated silica	—	20.00	36.00
X-50S (silane/carbon carrier)	—	4.00	7.20
Process oil	5.00	5.00	5.00
ZnO	4.00	4.00	4.00
Stearic acid	1.50	1.50	1.50
6-PPD	1.00	1.00	1.00
Master-batch total (M-1)	151.50	153.50	154.70
Sulfur	1.80	1.80	1.80
CBS	1.30	1.30	1.30
Final batch total (F)	154.60	156.60	157.80

3. Mixing sequence

All compounds were prepared by a two-step internal-mixer process using a 1.6 L Banbury mixer. In the first stage, the polymer was masticated and the fillers, oil, and remaining master-batch ingredients (ZnO, stearic acid, 6-PPD) were incorporated to produce the master-batch (M-1); no curatives were included at this stage to avoid scorch. In the second stage, sulfur and the accelerator CBS were added to the cooled master-batch to produce the final batch (F), which was discharged at a controlled temperature. The final batch was sheeted out on a two-roll mill. Because the silica-containing compounds (P2, P3) required additional homogenization to match target Mooney viscosity, these compounds were given a re-mill pass.

For silica-silane systems, mixing temperature is a critical variable: the mixing chamber must be held hot enough to drive the silanization reaction (release of ethanol) to completion while remaining cool enough, together with adequately high shear, to avoid premature scorch and to prevent re-agglomeration of the silica network. A processing window of roughly 145–155 °C is generally recommended for Si69-type silanes, with somewhat higher tolerance (up to ~165 °C) for disulfide silanes.

4. Vulcanization

Vulcanization was effected using a conventional sulfur/CBS accelerator system common to all three formulations, so that differences in cure behaviour

between compounds could be attributed to filler chemistry rather than to changes in the curative package. Silica's acidic surface silanol groups are known to adsorb basic accelerator species, retarding the vulcanization reaction relative to carbon black; this effect was evaluated by monitoring cure characteristics on a rheometer at two conditions — 190 °C for 3 minutes (a rapid industrial-style scan) and 150 °C for 1 hour (an extended scan capturing scorch, cure, and reversion behaviour) — per ASTM D5289.

5. Testing

The following standard test methods were used to characterize the raw compounds and their vulcanizates:

- Mooney viscosity and scorch — Mooney shearing-disk viscometer, ASTM D1646 (viscosity at 100 °C; scorch monitored at 125 °C, t₅ and t₃₅).
- Cure characteristics — rotorless (rheometer) cure meter, ASTM D5289, at 190 °C/3 min and 150 °C/1 hr.
- Hardness — Shore A durometer, ASTM D1415/D2240; five measurements per specimen.

- Tensile properties (modulus, tensile strength, elongation at break) — universal testing machine, ASTM D412 Method A, dumbbell specimens.
- Abrasion resistance — DIN abrasion tester, DIN 53516 / ASTM D5963 (equivalent to DIN ISO 4649); volume loss recorded after 84 drum revolutions under 10 N ± 0.2 N load.
- Rebound resilience — rebound pendulum, ASTM D1054, median of three post-conditioning strokes.
- Specific gravity — determined on cured specimens for each formulation.

6. Physical properties

The physical properties evaluated on the cured vulcanizates were: Shore A hardness; 100%, 200%, and 300% tensile modulus; tensile strength and elongation at break; rebound resilience at 100 °C; DIN abrasion loss and abrasion resistance index; and specific gravity. These properties, together with the rheometric and Mooney data, formed the basis for comparing the reinforcing efficiency of carbon black versus silica in the natural rubber matrix.

III. RESULT AND DISCUSSION

Rheometric properties (190 °C/3 min and 150 °C/1 hr) are summarized in Table 2 and Table 3.

Table 2. Rheometric properties at 190 °C / 3 min.

Property (190 °C/3 min)	Unit	P1	P2	P3
Maximum torque	lb-in	12.09	8.80	8.95
Ts2	min	0.44	0.72	0.72
Tc90	min	0.73	1.11	1.11

Table 3. Rheometric properties at 150 °C / 1 hr.

Property (150 °C/1 hr)	Unit	P1	P2	P3
Min. torque	lb-in	1.76	1.63	1.59
Max. torque	lb-in	15.78	11.28	11.19
Ts1	min	4.61	7.12	6.65
Ts2	min	5.12	8.29	9.53
Tc10	min	4.97	7.28	7.02
Tc15	min	5.20	7.97	8.76
Tc25	min	5.48	8.71	10.55

Property (150 °C/1 hr)	Unit	P1	P2	P3
Tc50	min	6.20	9.78	12.71
Tc90	min	9.45	13.68	19.23
Final torque @ 60'	lb-in	13.39	9.49	10.24
MH – ML	lb-in	14.02	9.65	9.60
Reversion	%	17.05	18.55	9.90
CRI	min	23.09	18.55	10.31

Maximum torque at 150 °C fell sharply from P1 to P2 and P3, pointing to a lower crosslink density in the silica-containing compounds. Scorch and cure times (Ts2, Tc90) lengthened progressively from P1 through P3, and the same trend was reproduced by Mooney scorch (t5, t35) at 125 °C (Table 4).

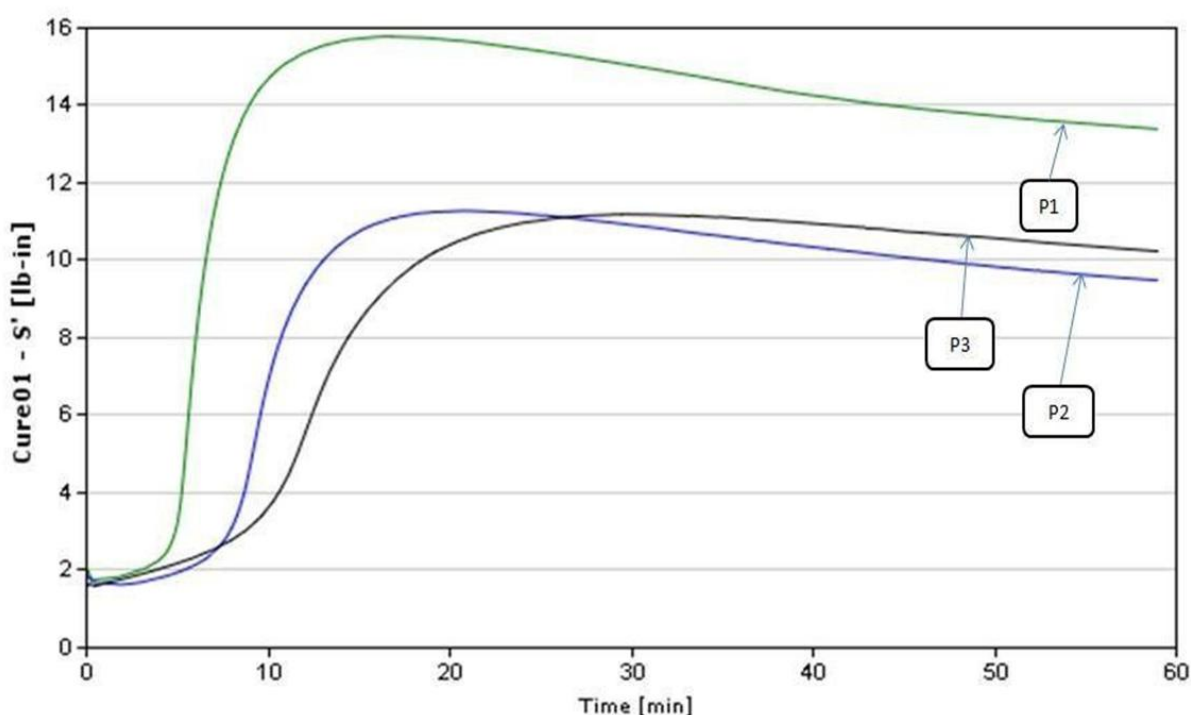


Fig.- Rheo graph

This slower, silica-dose-dependent cure is attributed to the acidic character of silica's surface silanol groups, which adsorb and deactivate part of the basic accelerator (CBS), reducing its effective reactivity.

Table 4. Mooney scorch and Mooney viscosity.

Property	Unit	P1	P2	P3
Mooney scorch @125°C (t5)	min	30.13	39.75	51.13
Mooney scorch @125°C (t35)	min	32.90	50.78	69.97
Mooney viscosity ML(1+4)@100°C	MU	51	53	52

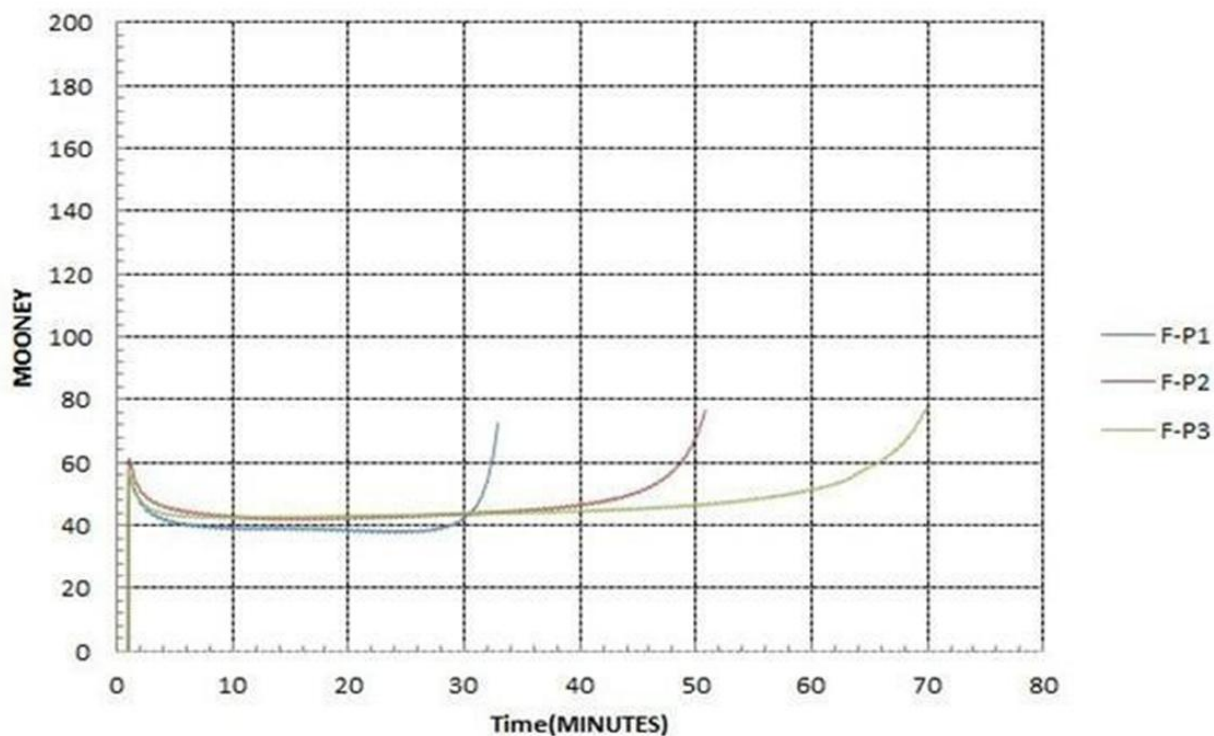


Fig- Mooney Scorch

Physical test results are summarized in Table 5. Hardness dropped from 61 Shore A (P1) to 51 Shore A for both P2 and P3. Elongation at break rose from 569% (P1) to 647% (P2) and 720% (P3), while 100/200/300% modulus and tensile strength all decreased across the same series — consistent with lower crosslink density reducing the network's resistance to deformation. Rebound resilience at 100 °C was marginally higher for P2 and P3 (66% and 65%) than for P1 (64%); the literature suggests silica-silane compounds should show a larger rolling-

resistance benefit than this, but the low crosslink density of the present silica compounds limited the improvement. Abrasion resistance declined from P1 to P3 (volume loss 106, 131, and 152 mm³; abrasion resistance index 99, 80, and 69, respectively), again consistent with the lower modulus of the silica-rich compounds. Specific gravity increased slightly with silica content (1.085, 1.099, 1.114 for P1, P2, P3), reflecting silica's higher density relative to carbon black at the loadings used.

Table 5. Physical properties of the cured compounds.

Property	Unit	P1	P2	P3
Hardness	Shore A	61	51	51
Elongation at break	%	569	647	720
100% modulus	kgf/cm ²	20.83	14.37	12.81
200% modulus	kgf/cm ²	49.16	33.78	24.76
300% modulus	kgf/cm ²	96.44	67.51	46.59
Tensile strength	kgf/cm ²	268.18	255.91	235.42
Rebound resilience @100°C	%	64	66	65

Property	Unit	P1	P2	P3
Abrasion volume loss	mm ³	106	131	152
Abrasion resistance index	%	99	80	69
Specific gravity	—	1.085	1.099	1.114

Filler dispersion, assessed by the carbon/filler dispersion index, remained high across all three compounds (99.84%, 99.75%, and 98.67% for P1, P2, and P3, respectively), indicating that the observed property differences arise primarily from crosslink density and filler–rubber bonding chemistry rather than from poor mixing or macro-dispersion of the fillers.

IV. INDUSTRIAL IMPLICATIONS

These results have direct relevance to compound design for tyre and general rubber-goods manufacturing. The silica-silane route is the basis of “Green Tire” technology because it can, in principle, widen the traditional trade-off triangle between rolling resistance, wet grip, and abrasion resistance. However, the present data show that unless the crosslink density of a silica compound is brought up to match that of a carbon-black control — typically through adjustment of silane dosage, mixing energy/temperature profile, or accelerator/sulfur ratio — much of that potential benefit (particularly the rolling-resistance gain expected from rebound resilience) will not be realized, while penalties in modulus, hardness, and abrasion resistance still appear.

From a processing standpoint, the requirement for a re-mill pass to bring silica compounds to target Mooney viscosity, together with the narrow mixing-temperature window (roughly 145–155 °C) needed to complete silanization without inducing pre-scorch, means silica-silane compounds carry a higher processing cost and require tighter process control than conventional carbon-black compounds. Manufacturers substituting silica for carbon black should budget for adjusted mixing cycles, possible additional mixing stages, and cure-system re-optimization (e.g., accelerator level or type) to compensate for accelerator deactivation by silica’s acidic surface, rather than assuming a like-for-like substitution at the existing cure package.

For applications where abrasion resistance and stiffness are the governing performance requirements (e.g., heavy-duty treads, industrial rubber goods), the carbon-black-rich formulation remains preferable unless crosslink density in the silica system is corrected. Where rolling resistance, wet traction, or flex-fatigue behaviour dominate the specification, silica-silane systems remain attractive provided the cure system is re-balanced.

V. FUTURE SCOPE

Because silica’s acidic surface partially deactivates the accelerator and suppresses crosslink density relative to carbon black, future work should focus on matching crosslink density across carbon black and silica compounds — for example, through adjusted accelerator/sulfur levels, alternative accelerator chemistries less sensitive to silica acidity, or optimized silane dosage and mixing temperature — before repeating the physical and rheometric comparison. A crosslink-density-matched comparison would allow the intrinsic reinforcing potential of silica versus carbon black to be evaluated independently of cure-system interference, and would give a more industrially representative assessment of the rolling-resistance and wet-grip benefits expected from silica-silane “Green Tire” technology.

VI. CONCLUSION

Replacing carbon black with silica (with proportional silane addition) in a natural rubber compound reduced maximum cure torque and slowed cure (Ts2, Tc90) in proportion to silica content, indicating a lower crosslink density that is attributable to the acidic nature of silica adsorbing and deactivating the accelerator. This lower crosslink density, in turn, explains the higher elongation at break and lower modulus, hardness, and abrasion resistance observed as silica content increased (P3 > P2 > P1 for elongation; P1 > P2 > P3 for modulus, hardness, and abrasion resistance). Rebound resilience was only

marginally improved in the silica-containing compounds, less than the literature would suggest for a well-optimized silica-silane system, again consistent with insufficient crosslink density. The study concludes that crosslink density, governed largely by silica's interaction with the accelerator, is the primary variable controlling the performance differences between carbon black and silica reinforcement in this formulation series.

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Author Contributions

The first author, Chandra Kant Sinha, and the corresponding author, Dr. Neetesh Kumar Dehariya, contributed substantially to the conceptualization and experimental design of the study, execution of experiments, data collection and analysis, interpretation of the results, and manuscript preparation, review, and editing.

The second author, Dr. Bhupendra Singh Ken, contributed to the conceptualization of the study and provided valuable intellectual input, critical review, and revisions to the manuscript throughout the review process.

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