– 고무용 신소재 –

카본 · 실리카의 복합물 "Carbon Shungit"

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Carbon Shungit은 러시아 및 인도의 고무소재 연구소가 러시아에서 생산된 특이한 물리 화학적 구조를 가진 화학물질을 가공하여 세계에서 최초로 개발한 고무용 복합 충진제로써 타이어 및 각종 고무제품에 사용되는 신소재이다.

이 소재는 개발자가 발표한 별첨 기술 자료에서와 같이 결정성 규산염 입자와 무정형 탄소가 각각 약 70% 및 30%의 비율로 결합되어 있고, 물리 화학적으로 그 결합이 안정되어 있으며 또한 비용이 저렴하기 때문에 생산고자 하는 고무제품의 용도에 따라 사용되고 있는 고가의 카본블랙 및 백카본의 전량 또는 부분적 대체품으로 이용되고 있다. 본 소재 및 고무배합물의 특성은 별첨 연구논문에 설명되어 있다.

참 고 : Carbon Shungit 에 대하여 문의 사항이 있으시면 필자의 E-mail : hanmah@chol.com 로 연락바랍니다.

Evaluation of Shungit -A Non-Petro Performance Filler in Tyre Compounds and Studies with Silica filler

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Abstract

Shungit are natural composite materials having unusual structure, consist of uniformly distributed highly dispersed crystalline silicate particles in amorphous carbon matrix. It's main component is full-erene like Shungit carbon (upto 80%).

Studies with Shungit, a non petro based performance carbon filler, in additional quantity to Carbon black in typical automotive tyre carcass, bead & apex and tread compounds as well as replacement of silica filler have been carried out and reported here.

Rheological / Rheometric studies, Physico-mechanical properties determination, Dynamic Mechanical analysis & Rubber Process Analyzer studies were carried out with different compounds. Compound mixing was carried out in Brabender Plasticoder. Technological properties of rubber compound with Shungit were considerably improved with respect to Mooney viscosity and time of premature vulcanization start (Scorch Safety). DMA analysis shows lower tan delta value with Shungit containing compound. RPA studies also indicate lower heat development and tan delta.

Results indicate that Shungit can replace, fully or partly, Silica in tyre tread compounds containing Carbon black.

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1. What is Shungit?

Shungit are natural composite materials having unusual structure, consist of uniformly distributed highly dispersed crystalline silicate particles in amorphous carbon matrix (Ref. 1). There is strong bond between carbon and silicate components. Shungit are characterized by high density, chemical stability and electrical conductivity. Shungit in finely ground state can partly replace carbon black and Silica in rubber compound (Ref. 2, 3 & 4).

Shungit consists of ~ 30% globular, X-ray amorphous and metastable carbon and of ~70% highly dispersive silicate particles. According to X-ray characteristics Shungit carbon is a non-crystalline form of carbon with a graphitic structure. Fine

Table 1. Basic properties of Shungit

	Characteristics	Property
1	Appearance	Fine black powder free of foreign matter
2	Carbon content % Min.	20
3	Loss of mass at 105°C, % Max.	2.5
4	Ash % Max	75
5	Bulk density, g / dm^3	250-350(0-10), 300-450 (0-20)
6	Aqueous extract pH	3.5-6.5
7	Sieve residue, % on mesh (Max.) 325	0.05
8	DBP absorption, ^{3}/100g	25-35

Table 1.1. Composition of Shungit

SiO_2	TiO_2	Al_2O_3	FeO	Mg	CaO	Na_2O	K_2O	S	C	H_2O Crys
57.0	0.2	4.0	2.5	1.2	0.3	0.2	1.5	1.2	29.0	4.2

Shungit powder is characterized by heat conductivity, heat capacity, electric conductivity & diamagnetic properties, microwave absorption effect. The surface of fine Shungit powder particles is of fractal nature and characterized by the presence of a thin conductive layer consisting of the micro-particles of laminar silicates carbon and crystallites. On the surface of Shungit particles there is a certain quantity of solvent extractable organic substances that may perform the role of "internal" plasticisers or processing aids (Ref. 2, 3 & 4).

Basic properties and composition of Shungit are given in Table 1 and 1.1(Ref. 2 & 3)

Effect of Shungit

Technological properties of rubber compound with Shungit were considerably improved . ML4 was reduced and MS was increased appreciably. Replacement of 20 parts by weight of commercial carbon black by Shungit does not affect technological properties and strength of rubber vulcanizates.

During mixing of rubber compounds loaded with carbon black, including combinations with pptd. silica fillers, Shungit demonstrate the properties of processing aids. Shungit accelerate the processes of incorporation, dispersion and distribution of fillers, "reduce dusting", raise the plasticity of elastomeric composites & their flow, adhesion to metal, steel and brass coated wire and improve processing of rubber compounds on mixing mill and in general improve mixing as well as the surface quality of extruded and calendered components.

Techno - commercial advantages of Shungit usage are, possibility of part or full replacement of CB & Silica by Shungit, improvement in technological properties of rubber compounds, improves processing characteristics, increases heat and fire resistance, used in rubber industry for production of tyre & technical articles, **offers substantial cost advantage**.

2. Background

Work on usage of Shungit as filler in rubber and polymers (Ref. 5), Polymer Composite (Ref. 6), different rubber products (Ref. 7 & 8) have been reported in literature.

Studies with Shungit, a non petro based performance carbon filler, in additional quantity to Carbon black in typical automotive tyre carcass, bead & apex and tread compounds have been carried out and reported here. Further evaluation work was carried out with Shungit against pptd. Silica , as part or full replacement in typical tyre tread compounds. Results of these studies are reported here.

Rubber industry uses three categories of fillers to reach the balance between the processing properties of rubber compounds, technical properties of vulcanizates and cost.: (i) reinforcing, (ii) semi-reinforcing and (iii) cost-reducing. In terms of the overall physico-chemical properties and structure-dispersion parameters Shungit can be classified as filler belonging to category ii and iii, but their function in rubber compounds extends further.

3. Experimental

Following instruments were used for the study. Electronic weighing Balance, Brabender Plasticorder Model PL2000, Laboratory size two roll mixing Mill, RPA 2000, Mooney Viscometer (MV-2000), hydraulic Curing Press, Zwick Tensile Tester (Model 1445), Air Ageing Oven, De-Mattia Flexometer, Hardness tester (Shore A), DIN Abrasion tester, Rebound Resilience tester (Dunlop Tripsometer), Dynamic Mechanical Analyzer (Metrovib Model VA-4000).

Raw Materials

All mixing ingredients used were from standard companies, Shungit supplied by Carbon Shungit International, Bead (brass coated) wire of 1mm diameter and nylon tyre cord of Denier 1680/2 were used.

Compound formulations used for the study are given in Table 2, 3, 4 & 5.

Mixing of all ingredients except curatives were done in Brabender Plasticorder to ensure the controlled mixing throughout the study. The curatives were added in the batches using mixing mill and sheeted out as per standard norms. For comparative

Table 2. Formulation: Carcass Compound

	CS-0	CS-10
Ingredient	phr	phr
NR(masticated)	100	100
Wood Rosin	2	2
Carbon Black N-660	30	30
Shungit	0	10
Aromatic Oil	4	4
Zinc Oxide	5	5
Stearic acid	2	2
A/o TDQ	1	1
A/o 6PPD	0.5	0.5
Sulphur	1.5	1.5
Insoluble Sulphur	1.9	1.9
CBS	0.9	0.9
Total	148.8	158.8

Table 3. Formulation: Bead Compound

	BS-0	BS-20
Ingredient	phr	phr
NR(Masticated)	70	70
SBR-1502	30	30
Reclaim Rubber	50	50
Carbon Black N-660	120	120
Shungit	0	20
Aromatic Oil	10	10
Resin	5	5
Zinc Oxide	4	4
Stearic Acid	2	2
A/o TDQ	1.5	1.5
Insoluble Sulfur	6.2	6.2
MBS	1.5	1.5
PVI	0.2	0.2
Total	300.4	320.4

Table 4. Formulation: Tread Compound

	TS-0	TS-20
Ingredient	phr	phr
NR	60	60
BR (High Cis)	40	40
Carbon Black N-339	55	55
Shungit	0	20
Aromatic Oil	9	9
Resin	2	2
Zinc Oxide	5	5
Stearic Acid	3	3
M.C. Wax	1	1
A/o TDQ	1	1
A/o 6PPD	3	3
Sulphur	2.3	2.3
MBS	0.8	0.8
Total	182.1	202.1

Table 5. Formulation : Silica + Shungit in Tread Compound

	I	II	III
Ingredients	phr	phr	phr
NR	60	60	60
BR	40	40	40
Carbon Black N-339	50	50	50
Shungit	-	10	15
Silica (VN3)	15	10	5
Aromatic Oil	8	9	9
Resin	2	2	2
Zinc Oxide	5	5	5
Stearic Acid	3	3	3
M.C. Wax	1	1	1
A/O TDQ	1	1	1
A/O 6PPD	2.5	2.5	2.5
Sulphur	2.3	2.3	2.3
MOR	0.8	0.8	0.8

purpose, the Shungit was mixed in one of the formulation and compared with reference one (blank i.e. without Shungit). The mixing conditions were set as follows.

Fill factor	=	0.9
Initial chamber temperature	=	30°C
Rotor speed	=	50 rpm
Mixing time	=	10 min.

Measurement of Mooney Viscosity (ML_{1+4} @100°C) and Mooney Scorch (t_5@120°C), were carried out using the Mooney Viscometer.

Rubber Process Analyzer (RPA 2000.)

The cure characteristics, e.g., scorch time (ts_2) and optimum curing time (t_{90}) including the minimum torque (ML) and the maximum torque (MH) were determined using a Rubber Process Analyzer (RPA 2000) for all compounds. With Tread rubber compound, studies for FST using RPA 2000 were carried out.

Following test conditions were used for this study

1. Frequency sweep 2 to 2000 cpm, Temperature 100°C & Strain 0.5°
2. Strain sweep 0.1° to 90°, Temperature 100°C & Frequency 6.0 cpm
3. Temperature sweep 40° to 100°C , Frequency 100 cpm & Strain 0.5°

Shear modulus, tan delta, loss modulus were recorded in the above test conditions

Moulding : Respective test specimens were moulded using the standard moulding practices and curing time used was based on rheometric data obtained from experiments.

Physico-mechanical : properties were measured with Zwick Tensile Tester machine (Model 14450) according to IS-3400 (Part-1), Hardness with Shore durometer according to ASTM D-2240, Tear strength (angle) of vulcanizates as per IS-3400(Part-17), Rebound Resilience measured as per BS-903(Part A-8), using Dunlop Tripsometer, Cut growth as per ASTM D-813 using De-Mattia Flexometer.

Ageing studies were carried out at 100°C / 72hrs and 100°C / 24hrs as per
(IS-3400 Part-4)

H-Pull Test : Adhesion between a dipped cord to rubber tested as per
IS-4910(Part-13).

Adhesion between bead wire to rubber compound was tested as per
ASTM D-1871.

Dynamic Mechanical Properties

The DMA studies of the above moulded samples were carried out using Dynamic Mechanical Analyser in tensile-compression mode at the frequency of 10 Hz, static strain 2.5%, dynamic strain 0.5% and covering temp range from 0° to 60°C. Storage modulus & tan delta were recorded for these test conditions.

4. Results & Discussions

Mixing

During the mixing of rubber compounds loaded with carbon black, Shungit demonstrate the proper-

Table 6. Mixing (Brabender Plasticorder) - From Brabender Plastogram

	Carcass		Bead		Tread	
	TS 0	TS 10	TS 0	TS 20	TS 0	TS 20
Loading Peak (Torque Nm)	116.8	115.8	53.5	42.7	90.5	49
Max. Peak (Torque Nm)	147.1	120.0	190.2	201.4	193.9	196.4
End (Torque Nm)	95.8	85.9	129.7	149.3	116.2	132.0
Loading Peak to End (Energy kNm)	317.1	290.50	354.5	306.4	378.7	343.8

ties of processing aids-they accelerate the processes of incorporation, dispersion and distribution of fillers, "reduce dusting", raise the plasticity of elastomeric composites, their flow, adhesion to metal, steel and brass coated wire, improve processing of rubber compounds on mixing mill and in general improve mixing, the surface quality of extruded and calendered components. (Ref. 2)

As stated earlier Shungit helps in the mixing of rubber compound i.e. incorporation, dispersion & distribution of reinforcing fillers in rubber. This is supported by the Loading Peak Torque, the Maximum Peak Torque and the Energy consumed during mixing-Loading Peak to End values given in the Table 4. In all cases (Carcass, Bead & Tread compound), compounds containing Shungit show lower torque for Loading Peak and energy for Loading Peak to End indicating that Shungit imparts processing aid characteristics resulting in lower energy consumption & shorter mixing cycle possibility. This would lead to better productivity at mixing stage.

Evaluation in Carcass Compound : From the results (Table 7) it can be seen that upto 10 phr of Shungit added in addition to Carbon black in the carcass compound shows :

a) Comparable Mooney viscosity values of the compounds
b) Improves the Scorch safety of the compound
c) Optimum Cure time (t_{90}) increases marginally

All physico-mechanical properties like the Modulus, the Tensile Strength, the Elongation at break & the Tear Strength are comparable and shows no negative effect of additional quantity of Shungit. The Modulus is increased marginally with Shungit addition.

On ageing at 100°C for 72 hrs again the values are comparable though the percentage retention of properties dropped sharply in both cases. Shungit

Table 7. Carcass Compound - Properties

Properties	CS-0	CS-10
Mooney Viscosity (ML_{1+4}@100°C),	24	26
Mooney Scorch (t_5@120°C), min.	17.7	20.2
Rheometric Study @ 150°C by RPA 2000		
MH (dNm)	96.87	101.64
ML (dNm)	3.017	3.086
MH-ML (dNm)	93.853	98.554
ts_2 (min)	2.37	2.58
t_{90} (min)	3.92	4.43
Physico-mechanical properties (Before Ageing)		
Specific Gravity	1.06	1.1
Hardness (Shore A)	49	51
Modulus 100% (Kg/cm²)	13	16
Modulus 300% (Kg/cm²)	47	54
Tensile Strength (Kg/cm²)	266	255
Elongation at break (%)	690	680
Tear strength (Kg/cm)	41	42
De-Mattia Flexing: Cut growth upto 12mm (cycles)	44100	37110
After air ageing for 72hrs at 100°C		
Hardness (Points)	+3	+2
Change in 100%Modulus (%)		
Change in 300%Modulus (%)		
Change in Tensile Strength (%)	-85	-87
Change in Elongation at Break (%)	-73	-79
Adhesion study		
H-Pull Test (N)	400	460

plays no role in this.

Adhesion to Cord by H Pull-test (dipped nylon tyre cord, 1680 DX2) shows marginal improvement with Shungit containing compound.

Evaluation in Bead Compound - Findings of evaluation of 20 phr Shungit in addition to Carbon black in the bead compound are given in Table 8. It shows:

a) Little increase in Mooney viscosity values
b) Improves the Scorch safety of the compound
c) Optimum Cure time (t_{90}) increases little

The Hardness increases by 5 points as also the Modulus improves whereas the Tensile strength & the Elongation at break drops a little.

On ageing at 100°C for 72 hrs, Shungit containing compound shows a marginal improvement in the retention of values for the Tensile Strength & the

Table 8. Bead Compound-Properties

Properties	BS-0	BS-20
Mooney Viscosity (ML_{1+4}@100°C)	43	52
Mooney Scorch (t_5@120°C), min.	12.3	14.9
Rheometric Study @ 150°C by RPA 2000		
MH (dNm)	147.81	166.15
ML (dNm)	10.16	11
MH-ML (dNm)	137.65	155.15
ts_2 (min)	1.23	1.54
t_{90} (min)	3.51	5.65
Physico mechanical properties (Before Ageing)		
Specific Gravity	1.25	1.29
Hardness (Shore A)	75	80
Modulus 100% (Kg/cm²)	54	65
Modulus 300% (Kg/cm²)	115	
Tensile Strength (Kg/cm²)	141	125
Elongation at break (%)	290	200
After air ageing for 72hrs at 100°C		
Hardness (Points)	+12	+10
Change in 100%Modulus (%)		
Change in 300%Modulus (%)		
Change in Tensile Strength (%)	-48	-51
Change in Elongation at Break (%)	-79	-75
Adhesion study		
Adhesion to bead wire (Kg)	65	77

Elongation at break.

Adhesion to Bead wire by Pull test shows marginal improvement with Shungit containing compound.

Evaluation in Tread Compound - Shungit was evaluated in Tread compound of a typical truck tyre at 20 phr in addition to Carbon black in the compound. Results (Table 9) show :

a) Comparable Mooney viscosity values of the compounds

b) Comparable Scorch safety of the compounds

c) Comparable Optimum Cure time (t_{90}) of the compounds

The Hardness and the Modulus increase a little whereas the Tensile Strength and the Elongation at break show little reduction with the addition of Shungit. De-Mattia flex-cut growth is marginally lower with Shungit (at 20 phr) containing compound. Earlier tests at 10 phr Shungit level have not shown such lowering effect (Ref. 3 & 4).

Table 9. Tread Compound-Properties

Properties	TS-0	TS-20
Mooney Viscosity (ML_{1+4}@100°C)	50	53
Mooney Scorch (t_5@120°C), min.	20.0	19.8
Rheometric Study @ 150°C by RPA 2000		
MH (dNm)	112.85	117.3
ML (dNm)	10.94	12
MH-ML (dNm)	101.91	105.3
ts_2 (min)	3.01	2.74
t_{90} (min)	5.62	5.42
Physico-mechanical properties (Before Ageing)		
Specific Gravity	1.12	1.19
Hardness (Shore A)	64	68
Modulus 100% (Kg/cm²)	25	31
Modulus 300% (Kg/cm²)	103	119
Tensile Strength (Kg/cm²)	239	212
Elongation at break (%)	600	510
De-Mattia Flexing: Cut growth upto 12mm (cycles)	15970	14750
After air ageing for 72hrs at 100°C		
Hardness (Points)	+10	+09
Change in 100%Modulus (%)	+88	+87
Change in 300%Modulus (%)		
Change in Tensile Strength (%)	-54	-57
Change in Elongation at Break (%)	-58	-63
Rebound Resilience, %	62	60
RPA analysis Data	Annexure	Annexure
DMA Study (Frequency 10Hz,static Strain 2.5%,dynamic Strain 0.5%)		
Tan delta at 0°C	0.331	0.308
Tan delta at 60°C	0.226	0.217
Dynamic Modulus (E') at 0°C (MPa)	9.61	10.9
Dynamic Modulus at (E') 60°C (MPa)	6.72	8.01

On ageing at 100°C for 72 hrs the values show marginal lower retention of Tensile strength and Elongation at break with Shungit containing compound.

Dynamic Mechanical Analysis (DMA) shows lower Tan Delta value with Shungit containing compound at 0°C which relates to Wet Traction. Higher Tan Delta values at 0°C will give better Wet Traction. The Tan Delta value at 60°C for compound containing Shungit also shows little lower value than the compound without Shungit. In this case lower value indicates better rolling resistance of the product.

Rubber Process Analyzer Study (RPA 2000)

RPA is a dynamic mechanical rheological tester. RPA can test the visco elastic materials at wide range of temperature, frequency & strain (single strain amplitude, SSA / Double strain amplitude, DSA). RPA provides the properties of the visco elastic material before, during and after curing.

Test samples are enclosed in a heated, pressurized, rotorless test cavity where the lower die is allowed to move and the upper die measures the response of the test sample. The RPA can vary the temperature, strain and frequency test conditions in the test cavity to simulate process and end use conditions.

Polymer / compound rheology is affected by many aspects of polymer and additives in the compound. Mooney viscosity have long been used as rheological measures. Unfortunately, the Mooney Viscometer is limited to a low shear rate test that does not describe the full range of rheological conditions typical in rubber processing.

Rheological properties at high shear rates are often more important than properties measured at low shear rates. Since rubber has both viscous and elastic properties it is necessary to look at two of the four parameters measured by the RPA at each test condition. These properties are usually separated into comparison of S' and S" values, or comparison of S* and tan δ values. (Ref. 9)

RPA analysis is done in three mode - Strain sweep, Frequency Sweep & Temperature Sweep, generally two are kept constant and the third is varied. Under Strain Sweep, lower strain show higher modulus (filler net working) and with increasing strain network breaks & G' (Shear modulus) reduces.

Δ G' = G1' (0.1% strain) - G2' (1% strain) = Energy in kPa is filler network indicator. Higher value indicates poorer dispersion or higher filler networking.

For compound TS 0 the Δ G' is 67.31 and for compound TS 20 the Δ G' is 88.73 which indicates the filler networking in compound TS 20 is more than TS 0. Also, the polymer filler interaction, indicated by the G'2 - G' inf, is lower for compound TS 0 as compared to compound TS 20.

RPA studies also show that the tan δ values are comparable for both the compounds indicating comparable heat development of the compound. There is cross over at high (40%) strain and for the compound TS 0 the tan δ values are marginally lower than the compound TS 20 indicating minor lower heat development with TS 0 compound at high strain.

In case of Frequency Sweep the tan δ values of the TS 20 compound are lower compared to TS 0 upto frequency of 20 cpm. After frequency of 20 cpm this changes indicating shear thinning behaviour of the compound. This can be related to the die swell / shape retention characteristics of the compound.

G' & G" increases and the tan δ value decreases with increasing frequency for both compound. The S" (represent resistance to deformation) is lower for TS 0 compound than TS 20 compound.

In case of Temperature Sweep, tan δ value reduces with increasing temperature.

Considering above findings usage of Shungit at 10 to 20 phr levels in Tread compound of different types of tyre, including Agricultural tyre could be recommended.

It has been reported (Ref. 3) that the use of Shungit at 5-10 phr level in the tread of Winter tyres based on carbon black in combination with silica enhances snow and ice grip characteristics as well as the processibility of compound.

Table 10. Shungit Vs pptd. Silica in Tread Compound-Properties

Properties	I	II	III
Physico-mechanical properties (Before Ageing)			
Specific Gravity	1.17	1.16	1.18
Hardness (Shore A)	60	60	61
Modulus 100% (Kg/Cm²)	18.3	18	19
Modulus 300% (Kg/Cm²)	75	74	74
Tensile Strength (Kg/Cm²)	200	195	194
Elongation at break (%)	620	590	595
Tear Strength (kg/cm)	92	85	80
Cut Growth upto 12mm (kc)	18.3	18.4	17.7
Rebound Resilience (%)	45	49	52
After air ageing for 72hrs at 70°C			
Hardness (Points)	+4	+4	+3
Change in 100%Modulus (%)	+23	+17	+16
Change in 300%Modulus (%)	+21	+15	+19
Change in Tensile Strength (%)	+3	-2	-2
Change in Elongation at Break (%)	-9	-9	-8

Studies with Silica + Shungit

Results of studies with compounds containing Silica and Shungit in different phr level are presented in Table 10. It clearly shows that Shungit can very well replace Silica filler in tread compound as all the physico-mechanical properties like Hardness, Modulus, Tensile strength & Elongation at break, Cut growth are closely comparable for the compounds containing Silica as well as the compounds where Silica is partly replaced by Shungit. Part replacement of Silica by Shungit gives rise to close value of tear strength, even little better than silica alone.

On ageing (for 72 hrs at 70°C) retention / change in values of different properties are little better with compounds where silica was replaced partly by Shungit.

Thus, Shungit can very well be recommended for usage in "Green Tyre" which is of much importance now a days. Further, Shungit usage for replacement of Silica will result in cost saving.

RPA analysis in three modes i.e. Frequency Sweep, Strain Sweep and Temperature Sweep was carried out with compounds containing Carbon Black, Silica & Shungit as given in Table 5. These results are presented in Figure. 1 & 2 comparing with the RPA analysis result of typical tread compound containing Carbon black and with & without Shungit.

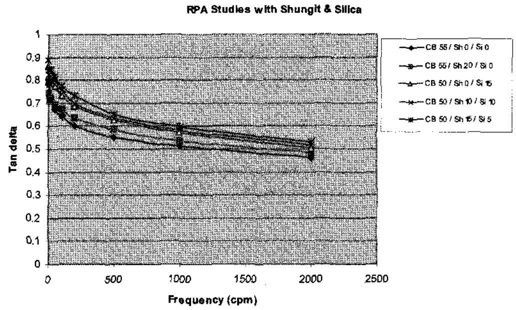

Figure 1.

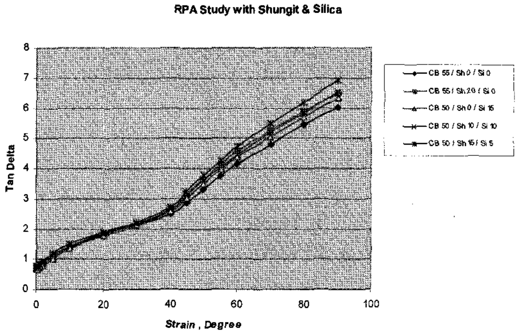

Figure 2.

It can be seen that at very low frequency, compounds containing Silica & Shungit show little higher Tan delta value (Fig. 1). However, all compounds (with CB, CB + Shungit, CB + Silica and CB + Silica + Shungit) converge to very close Tan delta values at higher frequency (> 500 cpm). Since, tyre while running creates high frequency, heat development with all compounds are expected to be similar. Normal frequency of a running bias truck tyre is around 10 Hz (600 cpm) and that of a radial is in the range of 15 to 20 Hz (1000-1200 cpm). Similarly, Tan delta values under strain is produced in Fig. 2.

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