



Effect of Crosslink Density on Cut and Chip Resistance of 100% SBR Based Tire Tread Compound

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Abstract. *The effect of Crosslink density on Cut and Chip resistance was affected on a typical 100 percent styrene-butadiene rubber (SBR)-based tire tread compound. In order to successfully develop products for tires used in off-road or poor roads and other demanding rubber applications, it is important to understand the C&C effect in rubber. Crosslink density varied by varying the sulphur to the accelerator ratio and also by changing the process aids. Basic polymer, filler and other ingredients such as activators and anti-degradants have remained unchanged. In the first setup, the sulphur was kept constant and the accelerator varied and the reverse was done in the second setup. It was made to achieve different crosslink density by changing the oil dosage and adding different resins. An attempt has been made to correlate Cut and Chip resistance to other physical properties. All these tests have been identified and optimized by the traditional tire tread compound.*

Keywords: *Cut and Chip resistance, crosslink density, off-the road, tire tread, cure system*

1. Introduction

The use of giant tires in off-the road systems (OTR), such as tractor, grader, backhoe, front end loader, etc., is routinely to be heavily cut and chipped when used in areas not accessible by traditional concrete or asphalt paths. Cutting happens when a sharp object hits a surface with sufficient force to penetrate and cut the surface [1]. The chipping of the rubber compound is generally tearing at 90° to the cutting direction, due to the effect of the traction, braking and other forces on raw or sharp surfaces. Cutting and Chipping causes the tire tread to tearing away the big chips. Cutting happens unexpectedly, without tearing; chipping is relatively sudden, but also with tear force. Neither cutting nor chipping has to do with the propagation of crack or propagation of fatigue [2].

Applications where the C&C resistance is important include mining agriculture off-the road tires, light-duty all-terrain truck tires and low road heavy-duty lorry tires [3]. A tire enters into contact with a wide variety of surfaces and textures, and the tread is subjected to intense asperities, while the tire is transmitted by a contact pad to complex vehicle forces [4]. Although C&C is complex, conventional C&C laboratory test methods use simple devices that do not alter load conditions significantly and the only information obtained from the test sample is the loss of weight [5-6]. Rubber surfaces exposed to strenuous, slipping effects bear large normal stresses that can lead to damage and eventual removal from the surface of material. Typical scenarios are tire treads on rugged terrain, agricultural or military track system service and hard granular material conveyor belts. For pneumatic applications, such tread rubber cutting and chipping (C&C) activity is a major concern when the tires are in contact with dirt, rocks, and rough ground surfaces, whether off-the road or in bad road conditions [7]. The usual difficulties in predicting C&C resistance of tire treads from the tests of rubber compounds in the laboratory are often appropriate to resort to costly research programmes involving the iterative tire building and field testing of new researchers [8].

A distinct correlation was found between the chemical crosslink structures and the mechanical properties, which, in turn, promoted the formation of the crosslink density of the rubber.

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The tensile strength slightly increased when the accelerator content was higher. In particular, the relative tensile strength and the elongation at break sharply increased by adding the secondary accelerators. In other words, the chemical crosslink characterizations were affected by the accelerator content, and these subsequently influenced the rubber's mechanical properties. The crosslink characterizations can be adjusted by changing the accelerator rather than the carbon black to improve the mechanical properties [9]. Good abrasion resistance was not co-relatable with C&C resistance. In other words, some tread compounds with good abrasion resistance had unsatisfactory C&C resistance; others with good C&C resistance had unsatisfactory abrasion resistance [10]. From these literatures limited work carried out on tire tread compounds. The aim of this work is to study the effect of crosslink density on cut and chip resistance of 100% sbr based tire tread compounds on typical tire tread compounds. In addition, the crosslink density varied by varying the sulphur to the accelerator ratio and also by changing the process aids.

2. Materials and methods

Rubber compounding with known chemicals in order to achieve the desired properties to the raw SBR rubber is formulated by adding chosen chemicals with control formulation of a typical OTR tire tread. It had been clearly found out that there may be a chance for agglomeration of filler materials inside matrix material due to improper dispersion that may result in stress concentration, which could be eluded by proper mixing [11], so due care has been made while mixing the ingredients into rubber. Master batch were prepared in a Banbery with a temperature of 160°C for 4 min by combining the rubber and other ingredients except curatives. The curing agents were then added to a two-rolling mill for 5 min in a laboratory and the final batch compounds were prepared [12].

To create crosslink at molecular level and thus to improve strength and elasticity, cross-linking agents are required. Uncured rubbers have low elasticity and strength, like a high molecular weight polymers [13]. By formulating long-chain molecules, networks are formed and the substance from a viscous fluid into an elastic solid. It's what takes place during vulcanization or cross-linking that increases strength, modulus and hysteresis. As a vulcanization agent, sulphur is commonly used in formulation. The input energy is retained by a deformed elastomer in terms of elastic potential energy [14]. It is released after crack growth and serves as the driving force for splitting. By molecular movements, the remainder of the energy is dispersed into liquid. Due to its high cross-link levels, a strong network of the molecular chain limited the chain motion, preventing the network from disappearing most of the energy that causes a fracture with low extension. Too high crosslinks are unwanted and may cause brittle fractures and may not be robust enough to survive viscous flow failure [15].

The optimal interlink density range for practical use needs therefore to be determined [16]. The rubber compound strengthening agents act as stress stoppers and must have a highly specific area. The particle therefore needs to be less than 1 micron in size. Carbon black and silica are popular fillers. The primary particle size can be obtained up to 0.1 μm , which gives a certain area of a few hundred m^2 per gram [17]. Table 1 showed the formulation of rubber compounds.

2.1 Cure Characteristics of Rubber Compounds

The cure characteristics of rubber compounds were studied on a MDR at 190°C as per ASTM D-2084-07. From the graphs, the optimum Cure time and scorch time could be determined. Rubber specimens for various physical testing including the Cut and chip test were prepared by mixing the ingredients by two stages [18].

Table 1. Formulation of the Rubber Compounds

Formulation of the Rubber Compounds															
	FORMULATION NO														
	EX1	EX2	EX3	EX4	EX5	EX6	EX7	EX8	EX9	EX10	EX11	EX12	EX13	EX14	EX15
INGREDIENTS	CONTENT IN phr ^a														
SBR-1502	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
CARBON BLACK	60	60	60	60	60	60	60	60	60	60	60	60	60	60	60
PROCESS OIL	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	3.0	11
ZINC OXIDE	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0	4.0
STEARIC ACID	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
ZINC SOAP OF UNSATURATED FATTY ACIDS	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
PHENOLIC TACKIFIER RESIN	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
PROCESS AID	-	-	-	-	-	-	-	-	-	-	-	-	-	4.0	-
GUM RESIN	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	-
MICROCRYSTALLINE WAX	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
TMQ	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
6PPD	2.2	2.2	2.2	2.2	2.2	2.2	2.2	2.2	2.2	2.2	2.2	2.2	2.2	2.2	2.2
SOLUBLE SULPHUR	1.4	1.4	1.4	1.4	1.4	1.4	0.8	1.1	1.4	1.7	2.0	1.4	1.4	1.4	1.4
TBBS	0.7	1.0	1.2	1.4	1.6	1.8	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
MBTS	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
PVI	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
HARDENER RESIN	-	-	-	-	-	-	-	-	-	-	-	-	3.0	-	-

^a phr – Parts per Hundred parts of Rubber

2.2 Determination of Laboratory Cut and Chip Resistance

The determination of cut and chip resistance was made using the B. F. Goodrich tester, for which the schematic diagram is shown in Figures 1 and 2. The frequency of the stroke of the cut and chip indenter was 1 Hz, and the rotation speed of the rubber wheel was set to be 750rpm. The corresponding speed at the surface of rubber wheel is about 1.25×10^5 mm/min. The diameter loss D_i was measured for 20 min, and the loss was divided by the number of strokes n to represent the average rate of cut and chip per stroke R_{cc} . This value was adopted as the measure of cut and chip characteristics [19-20]. The radial impact force F_i was constant for each stroke. Thus, the frictional force F_f will be different for each rubber compound, depending on its frictional characteristics (Figure 2). The surface temperature of the sample was found to rise rapidly during the cut and chip experiment; that is, it rose from room temperature to about 50° C just 2 min after the starting of the experiment. The final temperatures of black-filled natural rubber (FNR) and black-filled polybutadiene (FBR) were 55 to 65°C, respectively [21].

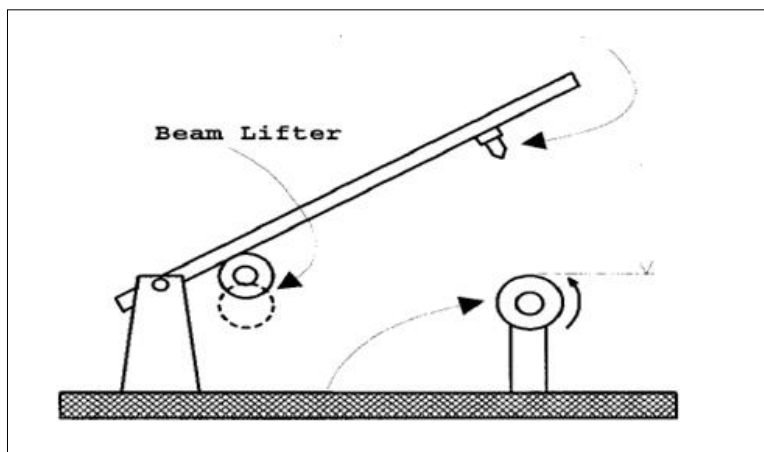
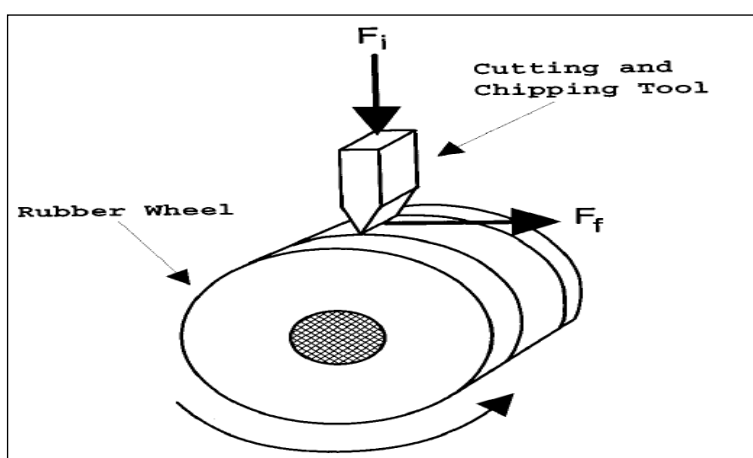


Figure 1. A Sketch of the B.F Goodrich cutting and chipping tester



Specification
Model: Tungsten Carbide Impactor
Width (w): 6.3mm
Thickness (T): 15-20 microns
Drop Height (h): 47 mm
Rubber Wheel for
Diameter: 50mm
Width: 12.5mm

Figure 2. A Sketch of the cutting and chipping part and acting forces of the B.F Goodrich cutting and chipping tester

2.3 Evaluation of Field Cut and Chip Performance

Two tread compounds based on the natural rubber (NR) 100 and NR–butadiene rubber (BR) 80/20 rubbers as in Table 2 were selected for the field tire test. The test tires of 11R22.5 in size for heavy-duty truck were then built using the selected compounds. The test tires were run on a rural road (about 20% paved), and the worn surfaces were investigated for an appropriate time interval [22].

2.4 Crosslink Density

The cross-link density was determined by immersing a small amount (known mass) of sample in 50mL of toluene for 3 days to attain equilibrium swelling. After this, the sample is taken out from the toluene and the solvent is blotted from the surface of the sample and is weighed immediately. This sample is then dried out at 70°C to constant weight. Then the chemical cross-link density is calculated (as per ASTM D 6814-02) by using the Flory–Rehner equation [23-24].

Table 2. Rubber formulations

		SBR	NR	BR
Master Batch	E-SBR 1500	98.5	-	-
	SMR20 NR	-	98.5	-
	High-Cis Nd-BR	-	-	98.5
	N330 CB	49	49	49
	Zinc oxide	1.8	1.8	1.8
	Stearic acid	1	1	1



Final Batch	Sulphur	1.9	1.9	1.9
	TBBS	2	1	2
	MTBS	-	-	0.5
	6PPD	1	1	1
	Total phr	155.2	154.2	155.7

3. MEASUREMENT OF CROSSLINK DENSITY			
3.1 Extent of Swelling:-			
VOLUME FRACTION OF THE RUBBER			
			(Weight of dry Rubber/Density of dry Rubber)
	Vr =		(Wt of dry Rubber/Density of dry Rubber)+(Wt of solvent absorbed by sample/Density of solvent)
FLORY-REHNER EQUATION FOR CROSSLINK DENSITY:-			
	x1		= Polymer-Solvent interaction parameter
	V1		= Molar volume of solvent
	Ve =		$-\frac{[\ln(1-Vr)+Vr+X1Vr^2]}{[V1(Vr^{1/2}-Vr)/2]}$

2.5. Abrasion Resistance

Abrasion test was done on the DIN abrasion tester for determining the abrasion resistance of compounds of vulcanized rubber, recommended by the Indian Standards Institution vide IS:3400 (Part 3)-1987. The mixed rubber compounds were cured by the compression molding machine at 155°C for 40 min. Two specimens from each of the 15 compounds were molded and then tested for obtaining the abrasion loss for each of the samples. The average of all the above parameters was recorded [25-26].

2.6. Experimental

A standard tester and test procedure as described in [4] is used in this study for CCC dynamic characterization. The principal scheme of BCCT is shown in Figure 1. Rotating at a set frequency between 0.25 and 2 Hz, eccentric 1 lifts and drops an arm 2 with a precision edge tungsten carbide knife 3 mounted on the end of the arm. The knife impacts a rubber disc sample 4 mounted on a rotating shaft; sample rotation speed is 200–1000 rpm. An air cylinder 5 lifts the knife arm at the completion of a preselected test time. The test specimens consist of compression molded rubber cylinders, 5.08 cm diameter, 1.27 cm wide, with a 1.27cm concentric hole at the center. Test pieces are prepared by compression molding. In contrast with standard application of BCCT, results of CCC characterization are recorded as functions of time. Thus, the wear process for each compound in terms of weight loss, W, (in g) is studied for the duration of 10 min in 30 s increments. The first specimen of each compound is tested for 30 s, each next specimen is tested 30 s longer than the previous one, and so on. The weight change is reported for each specimen after each test duration. The weight of the trimmed test piece is measured to an accuracy of 0.0001 g. A standard tester and test procedure as described in [4] is used in this study for CCC dynamic characterization. The principal scheme of BCCT is shown in Figure 1. Rotating at a set frequency between 0.25 and 2 Hz, eccentric 1 lifts and drops an arm 2 with a precision edge tungsten carbide knife 3 mounted on the end of the arm. The knife impacts a rubber disc sample 4 mounted on a rotating shaft; sample rotation speed is 200-1000 rpm. An air cylinder 5 lifts the knife arm at the completion of a preselected test time. The test specimens consist of compression molded rubber cylinders, 5.08 cm diameter, 1.27 cm wide, with a 1.27cm concentric hole at the center. Test pieces are prepared by compression molding. In contrast with standard application of BCCT, results of CCC

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Rubber compounds were prepared with two blending phases in an internal mixer (SYD-2L, Everplast, Taiwan) and the two steps were performed. In the first stage, a 50 rev/min rotor speed, a 100°C temperature mixture and a fill factor of 0.7 have been used to generate a master batch. The rubber was first mixed for 1 min with CB and mixed over 4 min until the drop temperature was 140 to 150°C. The rubber was then mixed for 1 min. The master batch then was milled and sheared at 60°C with a two roll mill. In the second stage, a rotor speed of 35 rev / min was used to prepare the final batch at the 60°C mixing chamber temperature. The first was to mix the master batch part for 1 min followed by an addition of the complete curing and mixing system for another 2 min at 100-110°C drop temperature. Then a two-roll mill at 60°C was used to mix the compound and get the final batch. The final batch was held 24 h before treatment. At 160°C with a moving die rheometer (MDR 3000 Basic, Mon-Tech, Germany), the treatment properties – including time for 90% final cure state (t_{90}) – were evaluated according to ASTM D 6204. The geometry specimens shown in Figure 2 were moulded with a compression moulding machine (LaBEcon 300, Fontijne Presses, Holland) at a thickness of 12.5 mm at 160°C for 13 min (13 min = 1 min by 1 mm) according to $t_{90} + 13$ min. 150x150x2 mm thick sheets were moulded in compression moulding machine with 90+2min for tensile and tear specimens. Specimens have also been cured for abrasion tests [27-28]. Continuous development of the equipment since the prototype was defined in 2013 [29] has resulted in the current prototype development, and a test was carried out on the rubber specimens by means of the cut and chip analyzer [30] (ICCATM), developed by Coesfeld GmbH, Germany, and distributed in Américas by Endurica, LLC, USA.



Figure 3. B.F Goodrich cutting and chipping tester

3. Results and discussions

3.1 Physical Properties

In an attempt to explain the observed results on the cut and chip resistance, some physical properties were also measured for the 100% SBR based tire tread compounds.

Table 3 and 4 shows the physical properties obtained from the previous tests. Figure 3 and 4 shows the tensile and tear strength of the individual compounds. Figure 4 shows the crosslink density measured by chemical method for all compounds. Also the Cut and Chip resistance was compared to all other properties like crosslink density, Elongation at break, 300% Modulus and relative volume loss.

As the Accelerator/Activator content was decreased, hardness, stress at 300% elongation, crosslink density and stress at break decreased considerably. For example, the ultimate tensile strength of a compound was obtained by increased crosslink density [20].

Table. 3 Physical Properties of Rubber Compounds

	FORMULATION	SP.GR	HARDNESS (SHORE-A)	T.S (kg/cm ²)	EB (%)	M300 (kg/cm ²)	TEAR STRENGTH (kg/cm)
S - CONSTANT / A - INCREASING	EX1	1.1475	68	200	614	79	66
	EX2	1.1477	71	206	583	90	69
	EX3	1.1479	71	206	552	98	70
	EX4	1.1480	72	207	525	109	71
	EX5	1.1481	72	213	524	110	74
	EX6	1.1482	68	214	499	113	74
A - CONSTANT / S - INCREASING	EX7	1.1461	64	147	722	49	60
	EX8	1.1469	71	182	625	73	67
	EX9	1.1477	71	206	583	90	69
	EX10	1.1486	74	207	491	111	72
	EX11	1.1494	74	208	423	134	72
ADDITION OF RESIN	EX12	1.1477	71	206	583	90	69
	EX13	1.1493	75	190	593	88	67
	EX14	1.1521	68	215	546	97	64
	EX15	1.1438	73	192	630	75	66

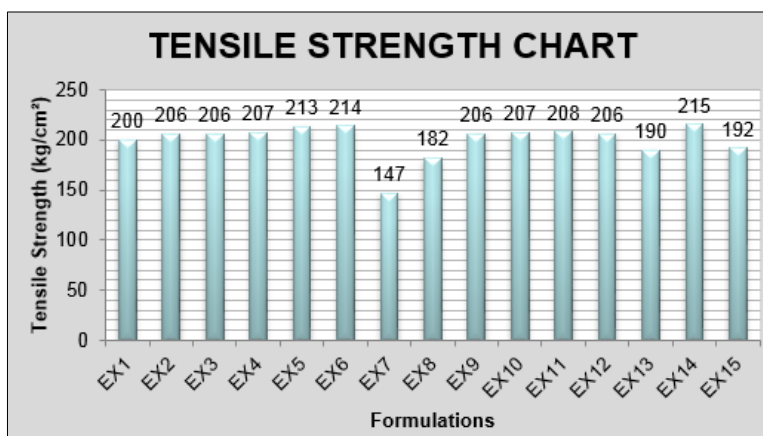


Figure 4. Tensile Strength of the compounds

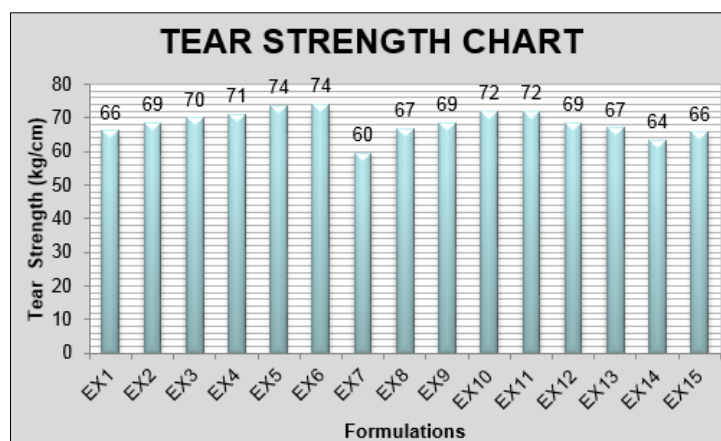


Figure 5. Tear Strength of the compound

Samples from EX1-EX6, EX7-EX11 shows gradual increase in Tensile and Tear strength with corresponding increase in the curing system, irrespective of whether it is effected by changing Sulphur or Accelerator.

Table 4. Crosslink Density and Volume Loss

	FORMULATION	CROSS LINK DENSITY (mol/cc)	C&C VOLUME LOSS (mm ³)	RELATIVE VOLUME LOSS (mm ³)	REBOUND RESILIENCE
S - CONSTANT / A - INCREASING	EX1	3.29X10 ⁻⁴	697	145	28
	EX2	4.28X10 ⁻⁴	800	125	26
	EX3	4.66X10 ⁻⁴	891	119	25
	EX4	4.96X10 ⁻⁴	920	117	25
	EX5	5.21X10 ⁻⁴	950	111	25
	EX6	5.22X10 ⁻⁴	960	109	27
A - CONSTANT / S - INCREASING	EX7	2.13X10 ⁻⁴	463	163	27
	EX8	3.29X10 ⁻⁴	664	144	25
	EX9	4.28X10 ⁻⁴	800	125	26
	EX10	5.57X10 ⁻⁴	866	122	26
	EX11	6.64X10 ⁻⁴	983	107	25
ADDITION OF RESIN	EX12	4.28X10 ⁻⁴	800	125	26
	EX13	4.19X10 ⁻⁴	813	136	25
	EX14	4.50X10 ⁻⁴	905	112	28
	EX15	3.33X10 ⁻⁴	694	138	27

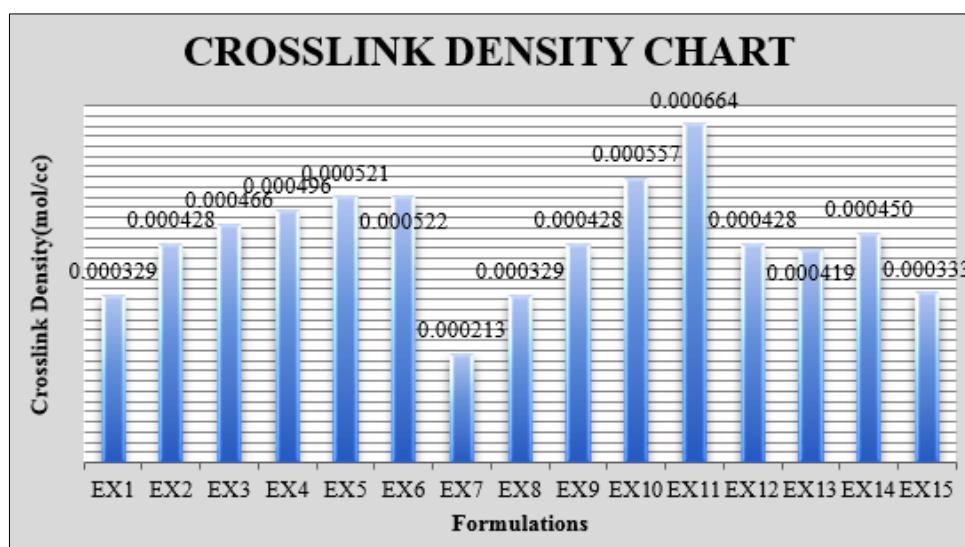


Figure 6. Crosslink Density of the compounds

Samples from EX1-EX6, EX7-EX11 shows gradual increase in crosslink density with corresponding increase in the curing system, irrespective of whether it is effected by changing Sulphur or Accelerator.

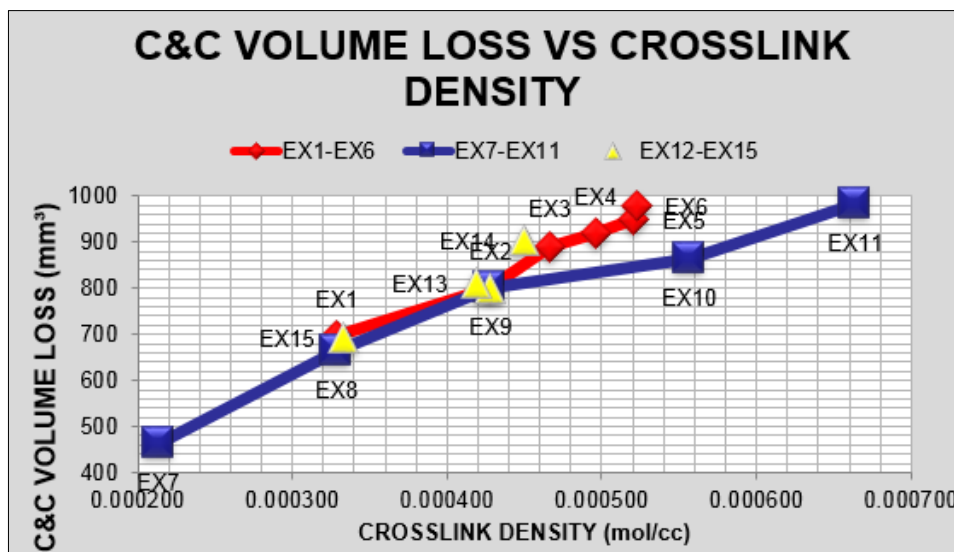


Figure 7. Comparison between C&C volume loss and Crosslink density

Samples from EX1-EX6 and EX7-EX11 shows gradual increase in C&C volume loss with corresponding increase in the crosslink density. So C&C resistance decreases with Crosslink density irrespective of whether it is effected by changing Sulphur or Accelerator. Hardener resin does not make any significant change in Crosslink density and C&C resistance. Replacement of process oil by process aid was increases the Crosslink density and reduces the C&C resistance. Replacing Gum resin by process oil shows improvement in C&C resistance with reduced Crosslink density.

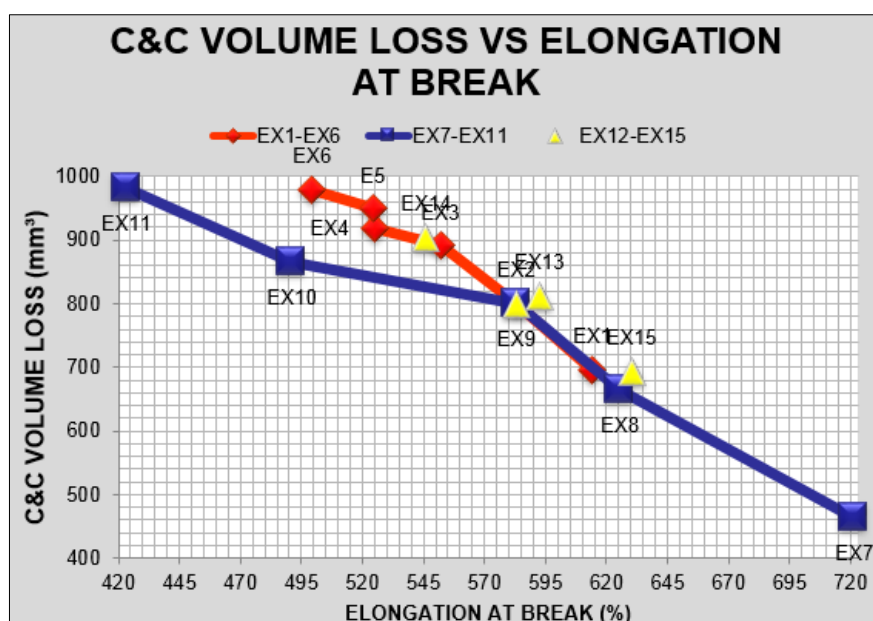


Figure 8. Comparison between C&C volume loss and EB%

For a better C&C resistance, high elongation at break achieved by reducing Sulphur is preferred as compared to reducing Accelerator. Irrespective of the input factor, C&C resistance improves gradually with increase in elongation at break.

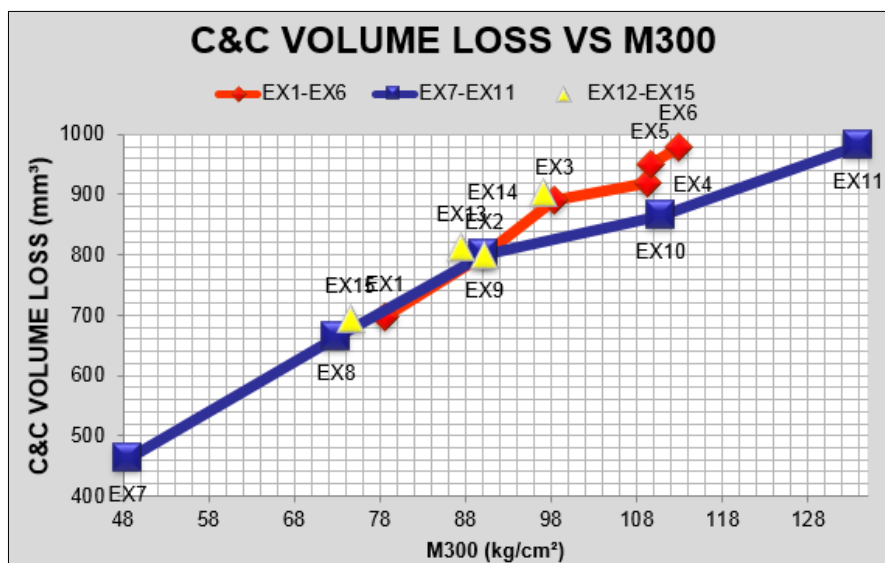


Figure 9. Comparison between C&C volume loss and Modulus

Irrespective of the input factor, Cut and Chip resistance improves gradually with decrease in Modulus. Ex14 having the highest modulus and lowest elongation at break shows the poor C&C resistance. Ex15 having the lowest modulus and highest elongation at break shows the better C&C resistance.

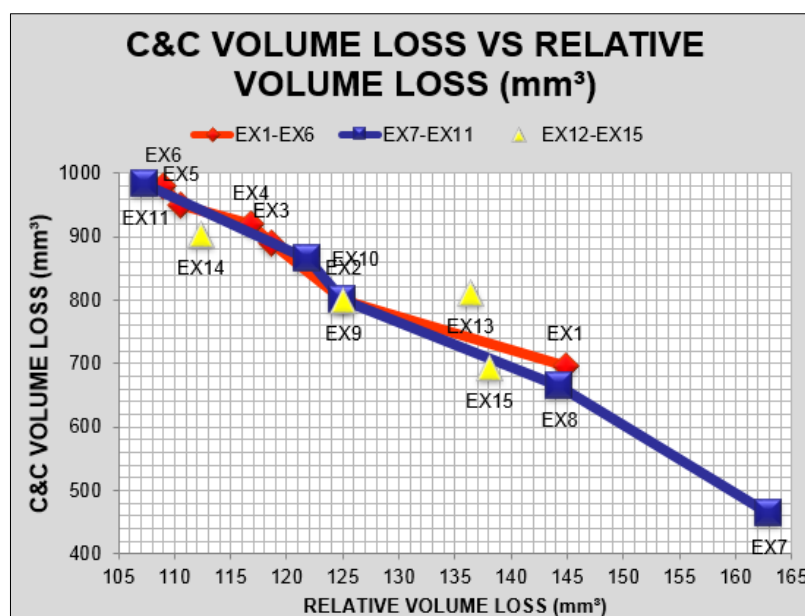


Figure 10. Comparison between C&C volume loss and relative volume loss



Like earlier work by Shamim ahmad et al, in this study also we found there is an increasing C&C resistance with decreasing abrasion resistance.

4. Conclusions

In this study the Laboratory Cut and Chip resistance of 15 compounds were investigated. The chemical Crosslink density was also calculated and compared with the Cut and Chip resistance. The Cut and Chip resistance was also compared with the other physical properties. Based on the test results were obtained in this study, the following conclusions could be reached.

An increasing trend of cut and chip resistance was found with decreasing Crosslink density. So the C&C resistance and crosslink density are in indirect proportions.

Cut and chip resistance decreases with Crosslink density irrespective of whether it is effected by changing Sulphur or Accelerator.

Hardener resin does not make any significant change in Crosslink density and C&C resistance.

Replacement of process oil by process aid was increases the Crosslink density and reduces the C&C resistance.

Replacing Gum resin by process oil shows improvement in C&C resistance with reduced Crosslink density.

For a better C&C resistance, high elongation at break achieved by reducing Sulphur is preferred as compared to reducing Accelerator.

Irrespective of the input factor, C&C resistance improves gradually with increase in elongation at break.

Irrespective of the input factor, C&C resistance improves gradually with decrease in Modulus.

Ex14 having the highest modulus and lowest elongation at break shows the poor C&C resistance.

Ex15 having the lowest modulus and highest elongation at break shows the better C&C resistance.

Also found that the better C&C resistance had unsatisfactory abrasion resistance.

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