Research Article

Carbon Black and Chopped Tyre Cord as Combined Filler System for Low Speed Solid Tyre Base Compounds made of Natural Rubber and Reclaim Rubber Blend

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Abstract

Chopped tyre cord (CTC) is a low cost filling material derived from the rejected pneumatic tyres which consist of carbon black (CB) and fibre materials other than the rubber. Due to this composition, CTC is possible to be used as reinforcement in Base compounds of the Solid tyres. Therefore, the Solid tyres Base compounds can be reinforced using a combined filler systems of pure CB and CTC to achieve acceptable properties while reducing the cost of the compounds. Therefore, the effects of combined filler system of carbon black (CB) and chopped tyre cord (CTC) was investigated on the properties of the low speed solid tyre base compounds made of Natural rubber (NR) and Reclaim rubber (RR) blend. Compounds were prepared varying CB to CTC ratios at 65:35, 55:45, 45:55 and 35:65 in parts per hundred parts of rubber (pphr) loading levels. The processing safety (Ts_2), optimum cure time (Tc_{90}) and the have been improved when the CTC content in the compounds is increased while other curing properties are acceptable. Physico-mechanical properties of the compounds have shown values within the required standard limits of solid tyre Base compounds at the 65 pphr of CTC coupled with 35 pphr of carbon black, the CB/CTC-35/65 compound compared to Control (CB/CTC-65/35). Dynamic mechanical properties have proved that the elastic properties have been increased when the strain rate or the force frequency is increased in both CB/CTC-35/65 and Control compounds. Also the CB/CTC-35/65 compound has shown better damping properties compared to Control at all the frequencies. Finally, the CB/CTC-35/65 can be recommended to use for Solid Tyre Base compounds as an ecofriendly method of managing the CTC waste produced in pneumatic tyre industry.

Keywords: *Carbon black, Chopped tyre cord, Dynamic mechanical properties, Physico-mechanical properties*

1. Introduction

Low speed solid tyres are dynamic rubber articles which are specially used for vehicles operate in hazardous conditions such as off-road construction sites, chemical industries, underground mining sites etc. Therefore, the physico-mechanical properties and dynamic properties are considered as significant properties of solid tyres. Different chemical ingredients and filling materials are usually mixed with particular rubbers such as natural rubber and synthetic rubber in order to achieve intended properties of tyres and such rubber mixes are called "rubber compounds" (Bijarimi *et al.*, 2010). The Base compounds are the rubber mixes use to prepare the most inner layer of Solid tyres which always hold the tyre with rim (Subramaniam, 2002). Therefore, the properties such as strength, hardness, grip and wear resistance should essentially be complied with the standard limits. Further, it is evident that higher creep resistance of Base layer is a significant property of solid tyres which maintains high dimension stability. In general, CB is the most common reinforcing filler in many engineering rubber articles which gives the better properties (Ciesielski, 1999). However, Everson, (1997) has investigated that the creep can be reduced using fibrous textile cords and polyester fibre in rubber compounds other than using CB alone. Further, the addition of CB into rubber compounds has been investigating from long time before as its ability to enhance the physico-mechanical properties, dynamic mechanical properties and process ability parameters.

However, instability of the price of petroleum has directly affected to the price of CB and it also could possibly be affected to the price of the tyre because CB is extensively used in tyres. Therefore, in view of enhancing the creep together with other properties the textiles and fibrous materials can be incorporated into such rubber compounds along with the CB.

Further, management of tyre waste is a major problem throughout the world. Also burning of waste tyre is one of the methods of tyre waste management and it generates hazardous compounds to the environment (Jayasekara, 2005). However, use of waste tyres or parts of them as raw materials for the manufacture of solid is possible (Subramaniam, 2002) and it could be considered as an environmental friendly method of tyre waste management. Therefore, number of studies has been done to see the possibility to use tyre wastes in solid tyres manufacturing throughout the world. As example, reclaim rubber which derived from tyres has been used in solid tyres (Sadhan *et al.*, 2001). Furthermore, rubberized fibrous cords can be used as reinforcing elements in rubber compounds which could reduce the creep, especially in base layers of solid tyres

(Mahmood *et al.*, 2016). These cords are a major waste in pneumatic tyre manufacturing industry and it could be used in solid tyres where the creep resistance is required (Łach *et al.*, 2018).

These rubberized fabric cords are available as chopped tyre cords (CTC) in the market and used in solid tyre as a filling material. However, optimization of the loading levels of CTC along with CB is a requirement for the manufacture of solid tyre Base compounds where better dimensional stability and grip are required. Moreover, CTC contains natural rubber, synthetic rubber, nylon and other fabric cords such as polyesters, carbon black, processing oils, waxes and so many chemical ingredients used in tyre industry. This composition of CTC indicates that it could have a potential to be used in solid tyre Base compounds as to improve the process ability and physico-mechanical properties while reducing the cost for the CB.

Hence a study was conducted with the objective of optimize the CTC and CB ratio to achieve optimum vulcanization characteristics, physico-mechanical properties and dynamic mechanical properties of solid tyre Base compounds as a value addition to CTC waste generated in pneumatic tyre industry.

2. Materials and Methods

2.1 Materials

Blend of ribbed smoked sheet (RSS) rubber and reclaim rubber (RR), CTC, CB (N375) and other rubber grade chemicals were supplied by the Elastomeric Engineering Co. Ltd. Horana.

Characterization of CTC

Thermo gravimetric analysis (TGA) was carried out at Rubber Research Institute of Sri Lanka, Rathmalana using Thermo Gravimetric Analyzer (TGA 4000 Perkin). CTC samples (7 g) were hold for 1 minute at 50°C for pre-heating and then the CTC samples were heated from 50°C to 995°C at the rate of 20°C min^{-1} at N_2 and O_2 atmospheres to identify the composition of CTC as weight percentages. Then the same CTC was used for the test sample preparation.

Preparation of Compounds

A standardized formulation of solid tyre Base compound was used for the preparation of test samples which shows in Table 1. The ratio of CB to CTC has been varied as mentioned in Table 1 based on the results of pilot studies.

Table 1: Formulations of solid tyre Base compounds

Compounding Ingredient	Amount (pphr)			
	CB/CTC-65/35 (Control)	CB/CTC-55/45	CB/CTC-45/55	CB/CTC-35/65
RSS/RR (60/40) blend	100	100	100	100
Processing aid	2	2	2	2
Antioxidant	3	3	3	3
Resin	5	5	5	5
CB (N375)	65	55	45	35
CTC	35	45	55	65
Processing oil	5	5	5	5
Zinc oxide	5	5	5	5
Stearic acid	2	2	2	2
Accelerator	2	2	2	2
Sulphur	4	4	4	4

(Note: some chemical names cannot be exposed due to unavoidable reasons, therefore only the type of chemical is given in the formulation)

All compounds were prepared according to a two-stage process using Tangential Internal Mixer (Banbury) having chamber volume of 1.6 L, 0.8 fill factor and at 60 rpm rotor speed. In the 1st stage Base compounds were prepared according to the mixing cycle given in Table 2 and the dumped compounds were conditioned at room temperature (25°C) for 3 hours before subjecting them for the 2nd stage Base compounding process. Sulphur, accelerators and CTC were mixed with the 1st stage Base compounds at the 2nd stage of compounding process (Table 3) using the same internal mixer.

Table 2: Mixing cycle of 1st stage Base compound

Time (Second)	Description
0th	Add RSS and Reclaim rubber
120th	Activators, resins and processing aids
330th	Add Carbon black and processing oil
400th	Ram up, sweep and ram down
420th	Dumping the 1st stage Base compound

Table 3: Mixing cycle of 2nd stage Base compound

Time (Second)	Description
0th	Add CTC
60th	Add the 1st stage Base compound
250th	Add sulphur and accelerators
300th	Dumping 2nd stage Base compound

Then the 2nd stage Base compounds were kept for 3 hours until the temperature of the compound reaches to the room temperature (25°C). Later those compounds were sheeted and further milled for 3 minutes using Two Roll Mill at 1:2 gear ratio and 30 rpm rotor speed. Finally, compound sheets having the thickness lower than 2.3 mm were obtained and stored at room temperature (25°C) for 14 hours before moulding. Then the moulding of test samples was performed at 180°C temperature and 20 MPa pressure using laboratory scale hydraulic hot press (Schubert, Germany).

Vulcanization Properties

The vulcanization properties of all compounds were determined using Dynamic Moving Die Rheometer (D-MDR 3000, USA) at 180°C according to the ASTM 2084 test method. The maximum torque (MH), minimum torque (ML), scorch time (Ts_2), optimum vulcanization time (Tc_{90}) were extracted from the vulcanization curves. Then the torque difference between MH and ML which could correlate with the cross linking density, and the cure rate index (CRI) which indicates the rate of crosslink formation were manually derived using following equations.

$$\text{Torque difference} = MH - ML \tag{1}$$

$$\text{Cure rate index (CRI)} = \frac{100}{Tc_{90} - Ts_2} \tag{2}$$

Physico-mechanical and Dynamic Mechanical Properties

Hardness, specific gravity, rebound resilience, compression set, tensile strength and tear strength were evaluated as physico-mechanical properties according to ISO 7619, ASTM D297, ISO 4662, ASTM D395, ISO 37: 2011 and ISO 34-1:2011 test methods respectively. DMA frequency sweep test was performed using TAQ 800 Dynamic Mechanical Analyzer at 120°C constant temperature keeping the amplitude at 10 µm while varying the frequency range from 0 to 10 Hz to evaluate the dynamic properties of vulcanizates of base compounds.

$$\tan(\delta) = \frac{\text{Loss modulus (E”)}}{\text{Storage modulus (E’)}} \qquad (3)$$

Tan delta (tan(δ)) or the damping property of the solid tyre base compounds was obtained using the ratio of storage modulus (E’) and loss modulus (E”).

3. Results and Discussion

Thermo Gravimetric Analysis of Chopped Tyre Cord (CTC)

Composition of CTC as a percentage of initial weight obtained from the thermo gravimetric analysis is shown in Figure 1. Three (03) main weight losses can be observed due to loss of volatile compounds such as waxes and oils, rubber and CB in CTC at three distinct temperature ranges respectively. About 5% of volatile compounds including oils and waxes, 68% of rubber and fibre materials, 25% of carbon black, and 2% of metal oxides can be quantified according to the Figure 1.

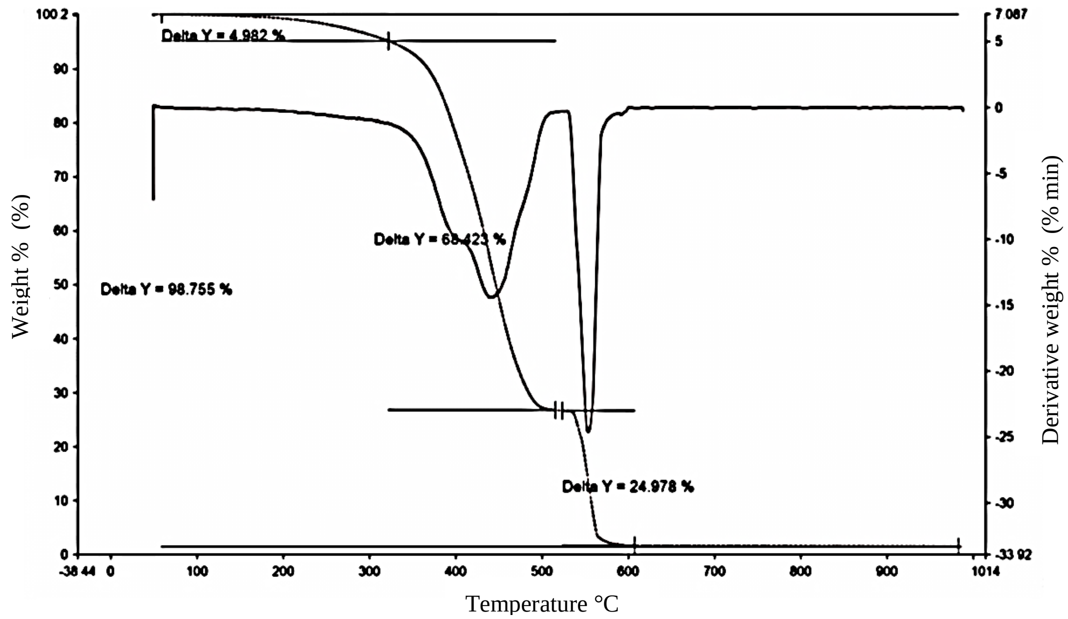

Figure 1: Thermo gravimetric analysis of CTC

The composition of CTC could have a possibility to affect to the physico-mechanical and dynamic mechanical properties of vulcanizates of newly developed compounds because of the availability of CB, fibre and other chemicals in CTC.

Vulcanization Properties

Table 4 shows the results of vulcanization properties of the prepared solid tyre Base compounds. The highest and the lowest minimum torque (ML) have been recorded in the CB/CTC-65/35 and CB/CTC-35/65 compounds respectively. Also the ML has gradually decreased when the CTC content is increased in the compounds compared to the Control. It is generally known that the lower ML values indicate the better process ability in rubber compounds with improved flowbility. Therefore, the CTC have improved the flowbility of rubber compounds according to the results of ML.

The maximum torque (MH) of compounds can be used to interpret the elastic modulus of vulcanizates (Hassan *et al.*, 2012). According to the Table 4, the MH values of compounds have decreased with the increase of CTC content of the combined filler system compared to the Control. Therefore, the decreasing trend of MH indicates the reduction of elastic modulus with the increase of CTC in the combined filler system. Further, the torque difference (MH-ML) which can be correlated to the cross linking density has shown almost same decreasing pattern indicating a lowered cross linking density with the increase of CTC compared to the Control. This may be attributed to the composition of CTC, because CTC consists of about 68% rubber and fibre. Also the oils in CTC could have a possibility to reduce the reinforcement (Hassan Al-nesrawy and Al-Maamori, 2016).

Table 4: Curing characteristics of the compounds

Compound	ML (dNm)	MH (dNm)	MH-ML (dNm)	Ts_2 (Second)	Tc_{90} (Second)	CRI (s^{-1})
CB/CTC-65/35	2.89[a]	34.52[a]	31.63[a]	29[a]	74[a]	2.22[a]
CB/CTC-55/45	2.39[b]	30.16[b]	27.77[b]	32[b]	79[b]	2.13[a]
CB/CTC-45/55	1.77[c]	27.04[c]	27.27[b]	34[c]	81[b]	2.13[a]
CB/CTC-35/65	1.41[d]	23.20[d]	21.79[c]	37[d]	83[b]	2.17[a]

(Note: Values that do not share a same letter are significantly different with respect to the particular property)

Further, these results of torque values have emphasized that the 25% of CB in CTC has not participated for the reinforcement. However, the scorch time and the optimum cure time have moderately increased when the CTC content in the combined filler system is increased. This could possible due to the reduction of heat transfer occurred because of increment of rubber content with the higher loading levels of CTC (Hassan *et al.*, 2012). The CRI has not significantly affected by the CB to CTC ratio and it indicates that the vulcanization system introduced to the compounds have not been affected by the CB to CTC (Hassan *et al.*, 2012).

Physico-mechanical Properties

Physico-mechanical properties of combined filler system incorporated solid tyre base vulcanizates have been reported in Table 5 along with the acceptable standard levels of properties of solid tyre Base compounds.

Table 5: Physico-mechanical properties of Base compounds

Compound	Hardness (Shore A)	Rebound resilience %	Specific gravity (gcm^{-3})	Tensile strength (MPa)	Elongation at brake%	Tear resistance (MPa)	Compression set %
Standard Limits	87-90	35-45	1.19-1.25	>6	>80	>50	<50
CB/CTC-65/35 (Control)	92[a]	37[b]	1.23[a]	10.44[a]	69.4[ab]	42.03[a]	54.44[a]
CB/CTC-55/45	91[ab]	38[b]	1.21[b]	10.40[a]	71.4[ab]	44.60[a]	55.46[a]
CB/CTC-45/55	89[bc]	40[a]	1.20[b]	10.26[a]	82.6[b]	47.90[a]	53.45[a]
CB/CTC-35/65	88[c]	41[a]	1.19[c]	10.46[a]	87.1[a]	54.7[a]	51.97[a]

(Note: Means that do not share a same letter are significantly different)

Hardness

When increase the amount of CTC in the combined filler system, the hardness (Shore A) of compounds has slightly decreased compared to the control sample. Only the hardness of CB/CTC-45/55 and CB/CTC-35/65 vulcanizates were within the accepted standard limit of 87-90 Shore A and other two compounds have exceeded the accepted standard limit. The reason for the slight decline of hardness may be due to slide and divergence of rubber chains each other because of the availability of processing oil in CTC (Ajam and Al- Maamori, 2016). Also high amount of CB could be considered as a cause for the increase of hardness in CB/CTC-65/35 (Control) and CB/CTC-45/55 vulcanizates due to its reinforcing ability (Bijarimi *et al.*, 2010). Also these results further explain that the CB in the CTC has not participated for reinforcing these compounds.

Rebound Resilience

Rebound resilience has increased when increasing the CTC loading ratio in compounds compared to Control. It causes to decrease the heat build-up in the solid tyre base compound that supports to improve the life span of the tyre (Evans, n.d.). This behaviour could be observed due to high rubber content of CTC and the increase of that when increase the CTC content in the combined filler system. Also the reduction of hardness and specific gravity could be attributed to this behaviour which indicates the less reinforcement and packing of rubber in the vulcanizates (Bijarimi *et al*., 2010). However, the property has indicated the values within the specification range showing that the applicability of CTC in solid tyre base compounds.

Specific Gravity

The specific gravity of such compounds is important when optimize the mass of compounds to be used for filling the tyre mould without wasting the rubber compounds. The specific gravity of rubber vulcanizates has reduced with the increase of CTC amount in the combined filler system compared to the control sample because of decreasing crosslinks between rubber and filler system and thus material is less stacked, lead to increase of formed pores within the compound and less converge of the molecules of material from each other (Ajam and Al-Maamori, 2016).

Tensile Strength and Elongation at Brake

When increasing the loading level of CTC in dual filler system of CB and CTC, the tensile strength has not shown a significant difference among each loading level. All the tensile strength values were greater than 6MPa, the specification level of tensile strength which required for solid tyre Base compounds. However, the elongation at brake can be used to study the reinforcement of rubber vulcanizates as an effective tool (Hassan Al-nesrawy and Al-Maamori, 2016). The elongation at brake of vulcanizates has increased gradually compared to control sample. This behaviour could be attributed to effect of nylon fibres and rubber content in CTC which could be blended with the rubber randomly. Generally, short length nylon fibres could increase the crosslink density of rubber (Hassan Al-nesrawy and Al-Maamori, 2016). Unfortunately, such reinforcement could not be observed in these vulcanizates. Apart from that the increment in elongation was experienced due to the effect of rubbery nature of CTC. Also this result indicates that even the same material in two separate phases such as CB in CTC (25%) and CB mixed with rubber compound has not shared each other in order to improve the reinforcement (Edirisinghe and Freakly, 1999). Also less elongation in the Control (CB/CTC-65/35) and CB/CTC-55/45 can be observed. However, this behaviour can be attributed to the reinforcement of added CB into the compounds. However, these compounds could not comply with the standard limits of the Base compounds.

The CB/CTC-35/65 and CB/CTC-45/55 samples have shown the required level of elongation (>80%) and therefore, these compound could potentially be used for solid tyre Base compound preparation.

Tear Strength

This property represents the resistant to initiate a crack and the energy required to propagate the crack. According to the results, only the CB/CTC-35/65 compound has shown the required level of tear strength compared to all the compounds. Increase of tortuous path length by CTC can be the reason for showing such increment in tear strength of CB/CTC-35/65 compound.

Compression Set

Due to the reason of decreasing the crosslink density, compression set of the vulcanizates has reduced slightly when increasing the CTC content in the CB to CTC ratio (Laine, 2016). Further, no any compound vulcanizate has shown the compression set below 50%, however, CB/CTC-35/65 sample has shown very closer value to 50% of compression set. The compression set measures the viscous nature of the vulcanizates (Laine, 2016) where high compression set indicates high damping and vice-versa. The increase of rubbery nature of compounds due to increase of CTC may reduce the compression set of vulcanizates. However, it can be concluded that the CTC is having a potential to improve the elastic nature of rubber vulcanizates.

According to the physico-mechanical properties, CB/CTC-35/65 sample can be selected as the best compound for solid tyre Base layers compared to all the other compounds. Therefore, dynamic mechanical analysis was carried out only for the CB/CTC-35/65 and Control (which practices in industry) samples during this study to find out the viscoelastic behavior against the dynamic frequency forces.

Dynamic Mechanical Properties

Dynamic property of rubber vulcanizate can be used to predict the viscoelastic nature of rubber vulcanizate against the dynamic force applied as a frequency (Meng, n.d.). Solid tyres are slowly moving dynamic rubber articles and therefore, 2-10 Hz frequency range was selected (Laine, 2016). Figure 2 and Figure 3 show a progressive increase of both storage and loss moduli from 2 to 8 Hz frequencies and a drop in storage and loss moduli of CB/CTC-35/65 after vibration frequency of 8 Hz compared to that of Control (CB/CTC-65/35). Strain induced crystallization occurs when increase the strain rate by increasing the frequency could be the reason for this behaviour. High amount of CTC in CB/CTC-35/65 compound has reduced the storage modulus compared to control sample (CB/CTC-65/35) indicating low elastic nature in such vulcanizates compared to Control.

Further, the loss modulus of both compounds has shown the same behaviour as in storage modulus. However, tan (δ) in Figure 4 can be used to explain this behavior which couples both the storage and loss moduli.

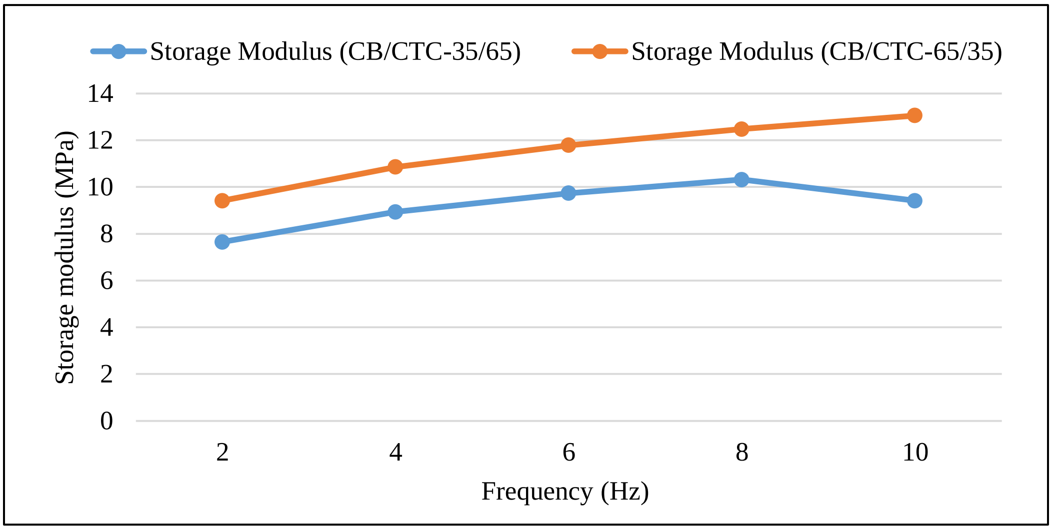

Figure 2: Storage modulus of Base compounds

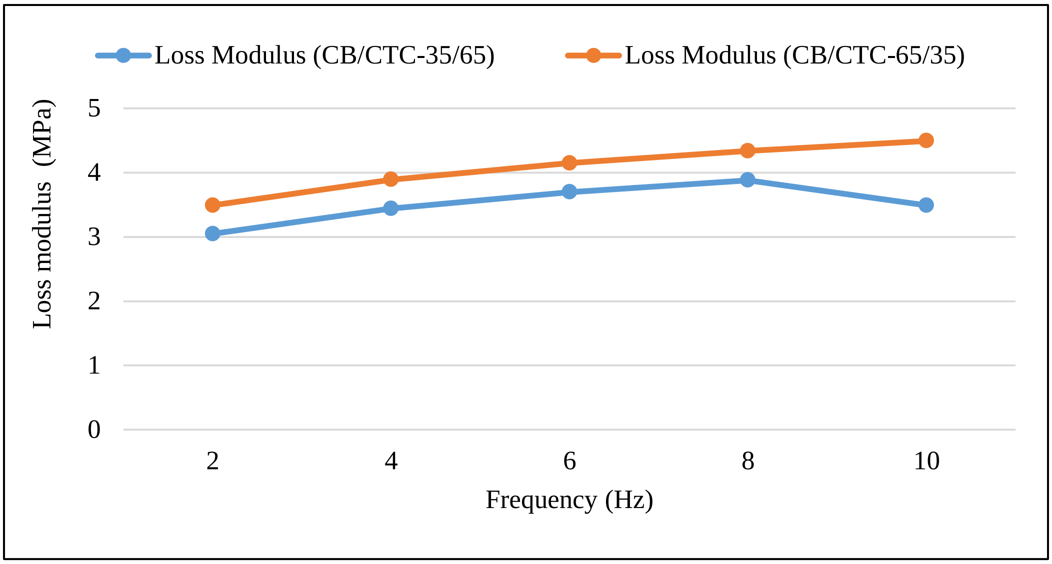

Figure 3: Loss modulus of Base compounds

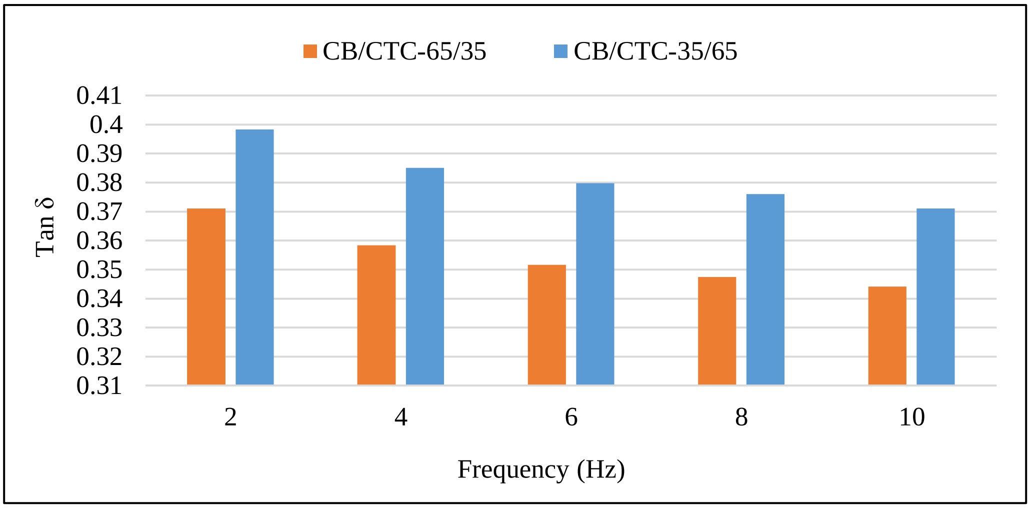

Figure 4: Tan δ of Base compounds

Tan(δ) which is the ratio between loss and storage moduli explains the damping properties of rubber vulcanizates. Higher Tan(δ) values for a given frequency at given temperature indicates high damping by means of high loss moduli and low storage moduli and vice-versa. This property can be correlated with the hysteresis and the heat-build-up of the tyre material where high heat-build-up indicates high Tan(δ) values because the absorbed energy is dissipated as heat (Edward *et al.*, 2010).

As shown in Figure 4, CB/CTC-65/35 compound has high Tan (δ) at all the frequencies which indicate higher damping than that of CB/CTC-35/65 compound (Control). CTC contains about 68% of rubber and this higher damping property of CB/CTC-65/35 compound could be attributed to the high rubber content in it compared to CB/CTC-35/65 compound. Therefore, the grip between solid tyre base and the rim could be improved at different rolling speed of tyres compared to that of control. However, tan(δ) has reduced in both cases when the frequency is increased. This behavior can be attributed to the stain induced crystallization occurs when increase the strain rate of viscoelastic rubbers (Franck, n.d.). Also the increase of storage modulus due to increase of strain rates or the frequency could reduce Tan (δ). Based on the overall dynamic mechanical properties, the CB/CTC-35/65 compound can be recommended to use without violating the specification limits of the solid tyre base layers.

4. Conclusion

In overall, four solid tyre base compounds were successfully prepared incorporating CTC. The evaluated vulcanization properties and physico-mechanical properties and dynamic mechanical properties have indicated that the CTC can be used as better filler in solid tyre base compounds replacing the carbon black content used in such tyre compounds while reducing the cost for carbon black and also as an ecofriendly method of managing CTC waste produced in pneumatic tyre industry when used such waste as a filler for solid tyre manufacturing process. Finally, CB/CTC-35/65 compound can be recommended to use in manufacture of solid tyre base compounds which is having the required properties within the acceptable standard limits.

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