



Influence of Different Fillers on Natural Rubber Composites to Assess Mechanical Performance

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ABSTRACT

Effect of different fillers on natural rubber compounds was investigated through curing, mechanical properties and morphology studies for improving mechanical performance of rubber articles. In this work, natural rubber blends with Silica as Single-filler compounds, and blends with Silica and Carbon black as Bi-filler compounds. Then, natural rubber blended with Silica, Carbon black, and Waste Tyre Rubber (WTR) particles as Tri-filler compounds were synthesized by a two - roll mill in specified operating conditions. In addition, natural rubber vulcanizate was prepared by using the same process. The different proportions were presented in Bi and Tri-filler compounds (10, 20, 25, 30, 40 phr) on CB and CB-WTR, respectively, whereas Silica was fixed at 20 phr. Curing characteristics showed that scorch time and optimum cure time significantly decreased with compared to natural rubber (NR) vulcanizate in all filler ranges. Whereas, minimum and maximum torque showed increased trend in all filler ranges. Cure rate index (CRI) also was presented and it showed significant good variation with the NR vulcanizate. Mechanical properties showed that tensile strength increased up to 30phr of CB in Bi-filler and it increased up to 10 phr with the addition of CB-WTR in Tri-filler compounds. Elongation at break decreased trend in Bi-filler and Tri-filler compounds, whereas maximum value was observed in single-filler compound. Hardness showed increased trend with addition of CB-WTR in Tri-filler, and it increased up to 25phr in Bi filler compounds. Tensile Modulus increased up to 25phr in Tri-filler and it increased trend in Bi-filler compounds. Tear strength increased up to 20phr and 25phr in Bi and Tri-fillers, respectively. Morphological study showed that better filler dispersion and good interfacial adhesion by the natural rubber matrix increased the tensile strength and opposite in tear strength. Among the rubber materials tested, Bi-filler compounds were found to be more suitable for improving the mechanical performance in the rubber applications.

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1. INTRODUCTION

The natural rubber (NR) is also elastomeric material and its pure form is poly isoprene. It is used extensively in many applications as predominant article. The reinforcement of elastomer by the incorporation of fillers has been the subject of numerous investigations. Generally, conventional fillers like Silica or Carbon

black are added into the rubber approximately 13-17 phr of the filler to achieve the desired reinforcing effect [1]. Silica (Si) is one of the reinforcing fillers widely used in rubber compounds. It improves the mechanical properties, mainly tear strength and abrasion resistance but also reduces rolling resistance in tires. Moreover, Silica leads to long processing time, high energy consumption, and environment pollution during processing. Therefore, it is quite an urgent issue for researchers to find some new filler material / hybrid fillers which can overcome the aforesaid shortcoming and partially replace Silica applications in rubber [2, 3].

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Carbon black (CB) is the most widely used reinforcing filler because it provides the excellent reinforcement strength of general purpose rubbers and also cheaper filler. The addition of reinforcing fillers increases tensile strength, tensile modulus, tear strength, and abrasion resistance of NR composite, and the dispersion state of filler determines the final properties of composites [4]. Precipitated Silica with particle size equivalent to that of CB is also used to produce highly reinforced rubber composites and incorporation of Silica and polymeric compatibilizers improves the mechanical properties while 20 phr of Silica provides good reinforcing properties [2, 3, 5]. The SiC nano particles and natural rubber compound show excellent mechanical properties in contrast to single walled carbon nano tube (SWCNT) - natural rubber compound which confirms the importance of Silica addition [6]. The transforming of scrap tyre and waste tyre rubber into powdered form by mechanical process is a more suitable method of reclaiming the waste rubber than the chemical process; then the recycled rubber powder (RRP) can be used as filler in natural rubber compound [7]. The scrap rubber particle (SRP) composite shows the poor adhesion between the rubber matrix and SRP with the large particles which indicates the effect of rubber particle size on the mechanical and rheological properties [8, 9]. Tensile strength and elongation at break decreases; while young modulus increases with reclaim rubber particle content [10]. NR-RRP compound shows that no significant changes occur where CB is replaced by RRP (up to 15%) and this compound is not recommended for tear resistance, since it leads to significant reduction in tearing forces [11, 12]. Rubber compounds have excellent tensile and tear properties, which make it ideal for many rubber applications and the potential applications of rubber composites could vary from industrial applications such as hoses, tire components, sensing devices to electrical devices [13]. Waste tyre rubber (WTR) itself has already contained CB, the addition of CB is still a good solution for obtaining the

good mechanical performance [14]. The raw WTR does not dispersed well in the rubber matrix and it contributes as weak sites upon stress-transmission, resulting in lower tensile properties [15]. Cure and mechanical behavior of WTR filled vulcanizates depends upon the kind of matrix, WTR shape/size and curatives used in rubber matrix [16]. WTR incorporated rubber blends provides enhanced toughness and also WTR containing rubber articles production accompanied with the low-cost [17]. The purpose of this research was to investigate the effect of various fillers in a NR vulcanizate to identify good filler material for improving the performance of NR composites.

2. EXPERIMENTAL

2. 1. Materials and Compound Preparation In this work , the following materials were used to make rubber compounds. Natural rubber, grade RMA 1X was used. Precipitated Silica and HAF Carbon black (N330) were used as first and second filler materials. Waste tyre rubber (WTR) was crushed into fine particles (150-250µm) by using pulverization and these particles consider as third filler material. Curing accelerators such as Sulphur, TMTD (Tetra Methyl Thiuram Disulfide), Zinc acid, Aromatic oil, Stearic acid etc., are customarily used in rubber industry. Rubber compounds were prepared in an open two-roll mill at room temperature (28°C). Initially, natural rubber was introduced to the two-roll-mill machine which pressed and transformed it into a thin and smooth forming rubber sheet round one of the rolls, rotated at 40 rpm, for the duration of (4-5 min.), 1.5mm nip gap and the sequence of ingredients was kept the same for all the compounds. The natural rubber compound ratio and ingredients were followed based on the data given in Table 1.

TABLE 1. Formulation of different rubber compounds

Name of sample	Ingredients in Phr *											Name of compounds
	NR	Si	CB	WTR	ZnO	Aromatic oil	Stearic Acid	CZ Acc.	TMTD	Sulphur	B rods Anti oxidants	
O	100	-	-	-	5	3	2	0.9	0.3	2.5	1	NR Vulcanizate (Gum)
A	100	20	-	-	5	3	2	0.9	0.3	2.5	1	Single-filler (S.F) compound
B1	100	20	10	-	5	3	2	0.9	0.3	2.5	1	Bi-filler (B.F) compounds
B2	100	20	20	-	5	3	2	0.9	0.3	2.5	1	
B3	100	20	25	-	5	3	2	0.9	0.3	2.5	1	
B4	100	20	30	-	5	3	2	0.9	0.3	2.5	1	
B5	100	20	40	-	5	3	2	0.9	0.3	2.5	1	Tri-filler(T.F) compounds
C1	100	20	10	10	5	3	2	0.9	0.3	2.5	1	
C2	100	20	20	20	5	3	2	0.9	0.3	2.5	1	
C3	100	20	25	25	5	3	2	0.9	0.3	2.5	1	
C4	100	20	30	30	5	3	2	0.9	0.3	2.5	1	
C5	100	20	40	40	5	3	2	0.9	0.3	2.5	1	

*Phr : Parts per hundred parts of rubbe

The Natural Rubber Composite (NRC) samples were made for five proportions in terms of CB / WTR loading ranges 10, 20, 25, 30, 40 in phr. After processing two-roll-mill techniques, the rubber sheet samples were put into the flat die kit and subjected to the hydraulic press with the cup at the centre of the bottom platen. The Rubber sheet was subjected to the following conditions for curing: temperature 160° C and pressure 12.95MPa in an electrically heated hydraulic press for a duration of 3 min.

2. 2. Cure Characteristics A Monsanto 100s Oscillating Disc Rheometer (ODR) was used to obtain the rheometric characteristics such as scorch time (t_{s2}), optimum cure time (t_{90}), minimum torque (M_L), maximum torque (M_H) and cure rate index (CRI) at 160 °C according to the ASTM D 2084-95.

2. 3. Testing of Mechanical Properties Tensile properties tests and tear test were performed at room temperature on a universal testing machine (INSTRON 3382) at the cross-head speeds of 550 mm/min. The dumb bell and angle shaped specimens were prepared according to ASTM D-412 and ASTM D-624 respectively and then, button shape specimens were tested for hardness based on ASTM D-2240. Hardness was tested using a Durometer (DURO TECH, model M202) shore hardness tester. Test duration of a trial was observed as 45-50sec for tensile test and 17-24sec for tear test.

2. 4. Morphological Study Scanning Electron Microscope (SEM) -- model Zeiss SUPRA 35VP FESEM was used to study the fracture surface of natural rubber gum, single, Bi, and Tri filler rubber compounds. All samples, before being scanned by the SEM, were made to undergo the procedure of sputter coating with gold to avoid poor resolution and electrostatic charging.

3. RESULTS AND DISCUSSION

3. 1. Cure Characteristics The effect of different fillers in NR matrix on curing characteristics as indicated in the following details. Figure 1 shows that minimum torque (M_L) increased significantly with increased all fillers used. It is due to cross link density of Single, Bi and Tri-fillers makes higher with NR matrix and did not allow easy viscosity flow to the matrix. Also, since these fillers contains highly aggregated and convoluted structure with NR matrix, so they increase the minimum torque. However, Bi-filler has less M_L compared to other fillers, due to CB presence which led to increase the flow. The maximum torque (M_H) is a measure of stiffness of the compounds which significantly increased in all fillers used. It is

mainly due to the dispersion of CB agglomerates which form a network themselves in the polymeric media. With increasing CB content, the network chains become shorter and the number of entanglements between two cross links decreases. The rubber molecules trapped on the CB sites, so it gives more in M_H . Tri filler contains WTR which retard the maximum torque. It is due to rubber particles trapped on the void of Si and CB sites, which decreases the M_H .

Figure 2 shows that scorch time (t_{s2}) and optimum cure time (t_{90}) of the compounds significantly reduces with all blends. Generally, shorter scorch time indicates that the cross-linking reaction started in advance. The Tri-filler compounds showing lesser scorch time compared to Single and Bi-filler compounds. It is mainly for WTR particles accelerates the reaction with rubber matrix since already it has sulphur content. The optimum cure time is the vulcanization time required to obtain the optimum mechanical properties. The Tri-filler has lesser or shorter t_{90} compared to other filler compounds which is due to more curatives in the compounds.

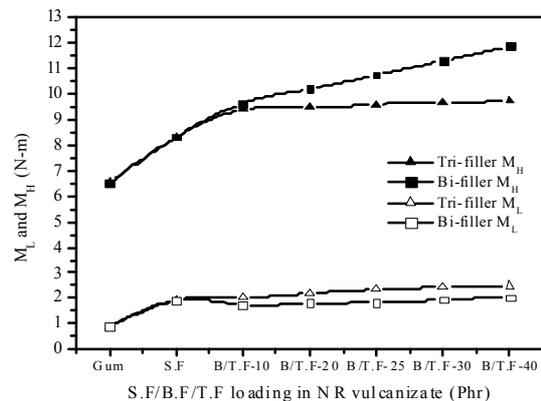


Figure 1. Effect of M_H and M_L with variation of different fillers

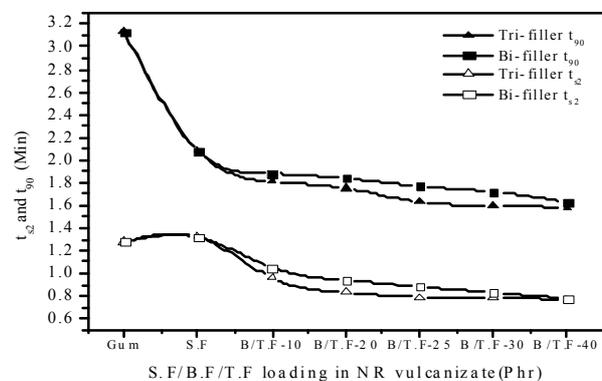


Figure 2. Effect of T_{s2} and t_{90} with variation of different fillers

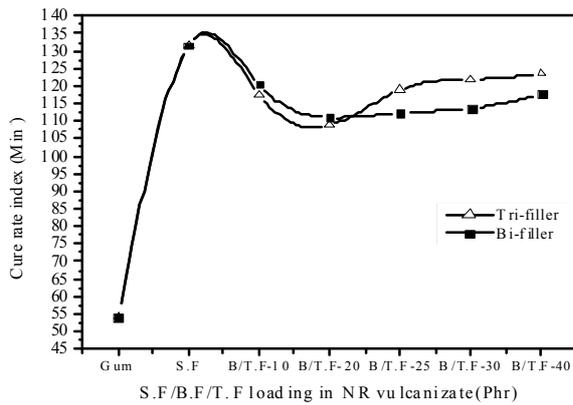


Figure 3. Effect of Cure rate index with variation of different filler

It can be concluded that Si filled rubber compound has higher processing time, whereas CB and CB-WTR filled single filler compounds has optimum and less processing time. The cure rate index (CRI) [19], which is a measure of the rate of cure reaction, is given by:

$$CRI = 100 / (t_{90} - t_{52})$$

Figure 3 shows that CRI higher in NR-Si matrix, which is mainly for the dispersion of silica into the rubber matrix, generates the active sites on the rubber matrix surface. Sulphur content trapped into the sites, and then accelerates the reaction. WTR content increased causes the CRI incremental, which is due to more curatives contains in Tri-filler compounds. Bi-filler compounds maintain the moderate range since CB presence led to decrease the reaction.

3. 2. Mechanical Properties In this research, 5 samples were undergone for testing and mean value was recorded with standard error (SE) in Table 2. The tensile strength increased to a maximum at 20 phr of Silica as constant filler into the rubber compounds as shown in Figure 4. This occurred due to fine dispersion of Silica into the rubber matrix and the resultant good interfacial adhesion between the Silica and rubber compounds. A similar observation had been reported by Sarawut Prasertsri et al. [2]. In this compound, on adding CB as second filler, the tensile strength gradually increased up to 30 phr of CB, and then decreased. It was due to the incorporation of CB into rubber matrix and filling the pores on CB surface to create strong adhesion with Si-rubber matrix and thus eventually increasing value of tensile strength. Further increase of the CB, resulted in the diminution of the interfacial adhesion between Si and CB. In this Bi-filler compound, adding the WTR particles as third filler caused the tensile strength to be slightly increased up to 10 phr and then decreased. It was due to the presence of CB in the rubber compounds. Then, any further increase of the rubber particles could

not catch the Bi-filler compounds, and thus non-uniform dispersion took place which led to the loosening or weakening of the bonding strength.

Elongation at break was studied for various proportions of all composites and the graph is plotted in Figure 5. Elongation at break maximum value was observed at 20 phr of Silica in single filler compounds. In this compounds, CB was added as second filler; it decreased with the increasing of the CB loading. It was due to its reinforcement effect of CB porous surface. Therefore, when the rubber matrix filled the pores on CB surface, it probably hindered the elongation of the rubber compounds. A similar observation had been reported by H. Ismail et al. [14] Then, WTR was added into Bi-filler compounds that resulted in the decrease of elongation at break deeply.

It was caused due to the poor dispersion of WTR in the rubber matrix and the result was poor adhesion and less cross link density with the rubber compounds. A similar observation had been reported by Xinxing Zhang et al. [15].

Tensile modulus was studied for various proportions and the graph is plotted in Figure 6. The tensile modulus for vulcanized rubber (NR gum) significantly improved with the addition of Silica. It was caused due to the uniform dispersion and good bonding with the natural rubber matrix. With these compounds, adding the CB content, the modulus instantly decreased and then gradually increases. It was due to CB which not having good adhesion with the Silica-rubber compounds. Thus, it behaves like rigid fillers which results in steady development of stiffness of the compounds.

With Single-filler compound, adding CB-WTR content, the tensile modulus increases significantly up to 25 phr and then decreasing trend. It was due to the WTR particles having been laid under the CB-Silica surfaces and the consequent increase in the agglomeration of the filler particles in the matrix which results in higher modulus. After 25phr, it decreasing trend which due to more CB content (WTR also has CB), retards the modulus, but comparatively higher with the Gum, Single and Bi-filler compounds.

Hardness was studied for various proportions and the graph is plotted in Figure 7. The hardness for vulcanized rubber (NR gum) significantly improved with the addition of Silica.

Hardness of Bi-filler compounds showed increasing trend up to 25 phr and then decreased, which was due to less coarse surface between CB – Si-NR matrix. The hardness gradually increased with incorporation of WTR in Bi-filler compounds. It was due to rubber particles having better hardness because of the higher modulus. As the WTR content in the Bi filler increases, it makes the compounds stiffer, and there would be an increase in hardness. A Similar observation had been reported by Hanafi Ismail et al. and Shuyan Li et al. [8, 18].

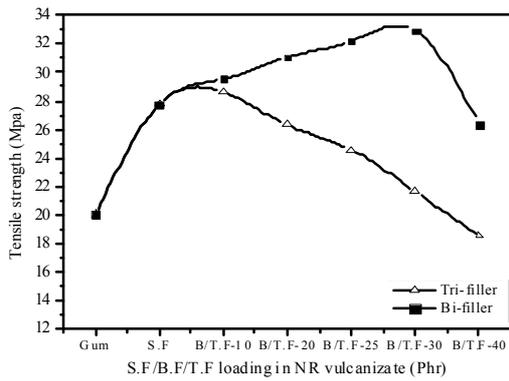


Figure 4. Effect of tensile strength with variation of different fillers

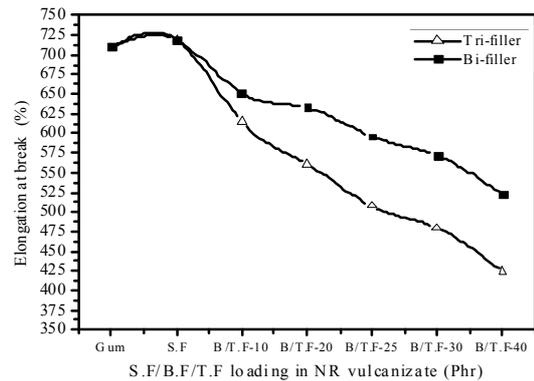


Figure 5. Effect of elongation at break with variation of different fillers

TABLE 2. Mechanical properties of natural rubber composites with different fillers

Mechanical properties mean value with standard error					
Samples	Tensile strength (MPa)	Elongation at break(%)	Tensile modulus (MPa)	Hardness (Shore A)	Tear strength (Mpa)
Methodology	ASTMD-412	ASTMD-412	ASTMD-412	ASTMD-2240	ASTMD-624
O	20.3±0.33	710±8.6	0.49±0.03	50±0.0	4.51±0.19
P	27.7±0.43	719±10.5	0.96±0.05	54±1.1	3.93±0.12
B1	29.53±0.96	651±10.6	1.17±0.04	57±0.4	5.54±0.15
B2	31.08±0.64	633±9.4	1.66±0.10	60±0.8	5.83±0.19
B3	32.2±0.75	596±9.9	2.13±0.16	62±1.2	5.1±0.14
B4	32.91±0.45	571±11.0	2.25±0.15	60±2.0	4.8±0.13
B5	26.34±1.00	522±9.0	2.35±0.12	56±1.5	4.35±0.20
C1	28.63±1.03	615±7.9	2.5±0.19	55±0.7	6.14±0.09
C2	26.33±0.82	561±11.8	2.82±0.12	59±1.3	6.51±0.20
C3	24.56±0.89	507±4.8	3.14±0.16	63±1.5	6.81±0.11
C4	21.63±0.66	479±9.4	2.89±0.14	65±1.4	5.7±0.11
C5	18.54±0.65	424±5.8	2.65±0.14	68±0.9	4.0±0.12

Figure 8 shows that tear strength increasing with the incorporation of Silica into the rubber compounds. This is due to the uniform dispersion of Silica in rubber compounds which improves the cross link density in rubber material. Incorporation of CB into this compound causes the tear strength to increase gradually up to 20 phr. This is due to retarding the aggregates and the resulting less cross link density. The same thing was observed in Tri- filler compounds, but after 25phr of CB/WTR. It was due to decrease of cross link density in WTR filled rubber matrix and the poor adhesion between the WTR and rubber compounds causing that decrease in the tear strength and a similar observation had been reported by Sang-Woo Kim et al. [16].

3. 3. Micrograph Observation

Figure 9 shows that SEM micrograph of tensile fractured surface of natural rubber gum (sample O) and 20 phr silica filled NR vulcanizates(sample A) . Sample O can be seen that no cross link chains appeared and lack of homogeneity

in structure, leads to less tensile properties. Sample A showed a appearance of many cross link chains which indicates that higher tensile strength.

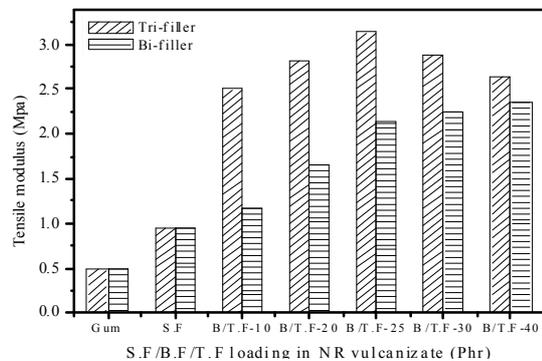


Figure 6. Effect of tensile modulus with variation of different fillers

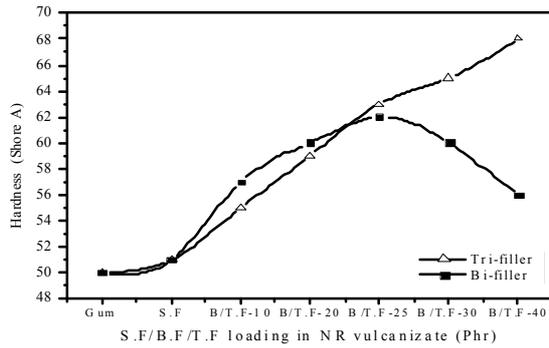


Figure 7. Effect of hardness with variation of different fillers

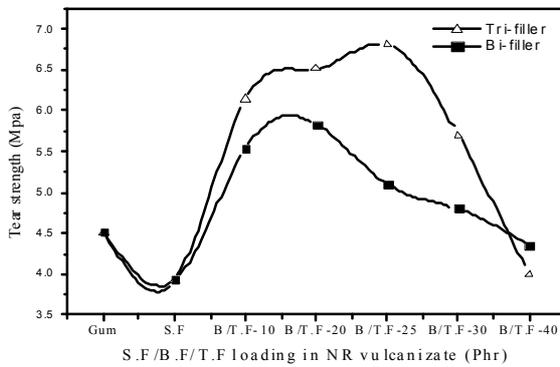


Figure 8. Effect of tear strength with variation of different fillers

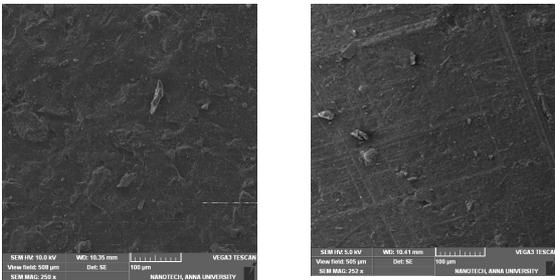


Figure 9. SEM micrographs of tensile fractured surface of natural rubber gum (sample O) and 20phr Silica filled NR vulcanizate (sample A)

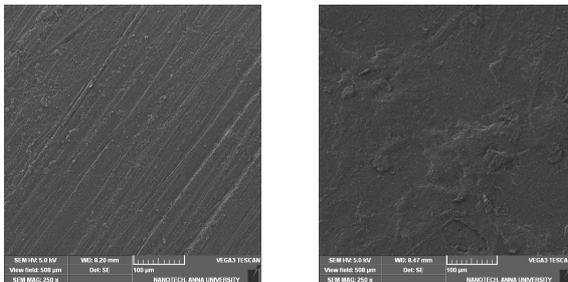


Figure 10. SEM micrographs of tensile fractured surface of Bi-filler filled natural rubber compounds with B1, B5 samples.

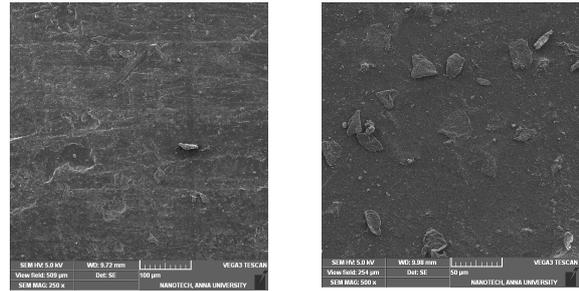


Figure 11. SEM micrographs of tensile fractured surface of Tri-filler filled natural rubber compounds with C1, C5 samples.

It is due to nano silica particles dispersed uniformly in the NR matrix and hence interfacial adhesion improved. This is evident from the fact that tensile properties have improved more in NR gum. Figure 10 shows that tensile fracture surface of samples B1 and B5. Sample B5 can be seen that more tear lines appeared along with cross link chains which indicates higher tensile strength. In B5 sample, tensile fractured surface is coarse and no cross link chains appeared. It is evident that tensile strength decreases. Figure 11 shows that tensile fractured surface of samples C1 and C5. It can be seen that there is partial filling of WTR into the Bi-filler rubber compounds and that the rubber particles have been laid under the rubber compounds and not seen on the surface. So, it can be inferred that the interfaces between rubber matrix and WTR were enough strength to resist fractures and hence fracture occurs through the bulk of rubber matrix, and not in the interfaces.

A similar observation had been reported by Sang-Woo Kim et al. [16]. C5 sample showed that, lack of adhesion between WTR and rubber matrix. So, it weakens the tensile properties. In order to obtain natural rubber compounds with satisfactory mechanical properties, good dispersion of WTR in the rubber matrix and strong interfacial adhesion are required [10]. In this work, CB and Silica fillers resulted in uniform dispersion throughout the rubber compounds, exhibiting better tensile strength, tear strength, hardness, modulus, and elongation at break.

4. CONCLUSIONS

From this work, the following conclusions have been arrived based on NR vulcanizate and Single-filler compound comparison.

1. Curing characteristics like scorch time and optimum cure time showed significantly decreased, whereas minimum and maximum torque increased trend in Bi and Tri-filler ranges.

2. Cure rate index (CRI) showed significantly decreased in Bi and Tri-filler compounds specifically compared with Single-filler compound.

3. Tensile strength increased up to 30 phr and tensile modulus showed increased trend in Bi-filler compound except for elongation at break. Whereas, tensile strength increased up to 10 phr and tensile modulus increased up to 25 phr in Tri-filler compounds except elongation at break.

Moreover, Tensile strength increased up to 64.3% for Bi-filler and 42.9% for Tri-filler when compared to NR gum.

4. Hardness increased up to 25 phr in Bi-filler compounds. Whereas, it showed increased trend in Tri-filler compounds.

5. Tear strength increased up to 25 phr in Tri-filler compound. Whereas, it increased up to 20 phr in Bi-filler compound.

6. Morphological study indicated that the better interfacial adhesion and cross link density by the natural rubber matrix increased the tensile strength and opposite in tear strength.

7. This work also indicated that among the rubber materials tested, Bi-filler compounds were found to be more suitable for rubber applications and hence, it improves the mechanical performance of rubber articles.

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Silica

Different Fillers

اثر پرکننده های مختلف در ترکیبات لاستیک طبیعی از طریق بهبود، خواص مکانیکی و مطالعات مورفولوژی برای بهبود عملکرد مکانیکی لاستیک مورد بررسی قرار گرفت. در این کار، لاستیک طبیعی با سیلیکا به عنوان ترکیبات تک پرکننده و با سیلیس و کربن سیاه به عنوان ترکیبات پرکننده دوتایی مخلوط می شود. سپس، لاستیک طبیعی که با سیلیکا، کربن سیاه و لاستیک تایر اتلافی (WTR) به عنوان ترکیبات پرکننده سه تایی مخلوط شده است، توسط آسیاب دو رول در شرایط عملیاتی ویژه ای سنتز شدند. علاوه بر این، لاستیک طبیعی ولکانیزه با استفاده از همان پروسه آماده شد. نسبت های مختلف در ترکیبات دوتایی و سه تایی پرکننده (۱۰، ۲۰، ۲۵، ۳۰، ۴۰ phr) در CB و WTR-CB به ترتیب ارائه شده است، در حالی که سیلیکا در ۲۰ ثابت نگه داشته شد. ویژگی های بهبود نشان داد که زمان سوختگی و زمان بهبود مطلوب به طور قابل توجهی نسبت به لاستیک طبیعی ولکانیزه شده در تمام محدوده پرکننده کاهش یافته است. در حالی که حداقل و حداکثر گشتاور، روند افزایشی در تمام محدوده پرکننده داشت. شاخص نرخ درمان (CRI) نیز نشان داده شد و آن تغییر معنی دار خوبی با لاستیک طبیعی ولکانیزه شده نشان داد. خواص مکانیکی نشان داد که استحکام کششی به خاطر کربن سیاه در پرکننده دوتایی تا ۳۰ phr و به خاطر اضافه کردن کربن سیاه و لاستیک تایر اتلافی به ترکیبات پرکننده سه تایی تا ۱۰ phr افزایش یافته است. زیاد شدن طول، در نقطه شکست در ترکیبات پرکننده دو تایی و سه تایی روند کاهشی داشت، در حالی که حداکثر میزان افزایش در ترکیب تک پرکننده مشاهده شد. سختی به خاطر افزودن کربن سیاه و لاستیک تایر اتلافی به ترکیبات پرکننده سه تایی روند افزایشی داشت و تا ۲۵ phr در ترکیبات پرکننده دو تایی افزایش یافت. مدول کششی تا ۲۵ phr در پرکننده سه تایی افزایش یافت و در ترکیبات پرکننده دو تایی روند افزایشی داشت. قدرت پارگی در پرکننده های دو و سه تایی به ترتیب تا ۲۰ phr و ۲۵ phr افزایش یافت. مطالعه مورفولوژیکی نشان داد که پراکندگی بهتر پرکننده و چسبندگی بین سطحی خوب توسط ماتریس لاستیک طبیعی باعث افزایش استحکام کششی و کاهش قدرت پارگی شد. در میان مواد لاستیک تست شده، این نتیجه به دست آمد که ترکیبات پرکننده دو تایی برای بهبود عملکرد مکانیکی در کاربردهای لاستیک مناسب تر هستند.

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