UNIVERSITI MALAYSIA PERLIS
International Journal of Nanoelectronics and Materials
IJNeaM
ISSN 1985-5761 | E-ISSN 2232-1535

Investigation on Properties of Carbon Black/Lignin Filled NR/BR Composites Influenced by Silane Coupling Agents

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Received 6 July 2025, Revised 14 August 2025, Accepted 8 September 2025

ABSTRACT

In this investigation, we explore the impact of silane coupling agents on the characteristics of composites comprising bis-(3-triethoxysilylpropyl) tetrasulfide (TESPT), 3-octanoyl-thiopropyl-triethoxy (NXT), and triethoxy (vinyl) (VTEO) with carbon black and lignin. The study delves into the role of these silane coupling agents, specifically focusing on their influence on the properties of the NR/BR composites filled with carbon black and lignin. TESPT, NXT, and VTEO were employed to enhance compatibility between an incompatible polar filler and non-polar rubber, forming a bridge between the filler and the rubber matrix during the mixing and vulcanization processes, thereby contributing to the improved performance of bio-filler-filled rubber compounds. The amounts of silane coupling agents added were in the range of 2-10 wt.% based on the lignin content to assess the optimum amount for interfacial bonding, rheological, and mechanical performance. A slight improvement has been observed in curing behavior, resulting in a shorter optimum cure time with the incorporation of TESPT and enhanced scorch safety with the inclusion of NXT in CB/lignin-filled NR/BR composites. Meanwhile, hardness values of the compounds with TESPT display higher values and thus higher crosslink density than NXT and VTEO compounds. The findings demonstrated the benefits of utilizing TESPT, NXT, and VTEO as effective silane coupling agents between CB/lignin fillers and rubber.

Keywords: *Carbon black, Lignin, Composites*

1. INTRODUCTION

The rubber industry traditionally relies on carbon black (CB) as a reinforcing filler due to its beneficial effects on mechanical and thermal properties. However, CB is environmentally harmful, being a byproduct of petroleum combustion and classified as a possible carcinogen. Consequently, researchers are exploring eco-friendly alternatives such as bio-fillers derived from renewable resources like lignin, a byproduct of the pulp and paper industry. Lignin's phenolic structure enhances mechanical properties and can modify chemical characteristics, improving rubber composites' performance [1-3]. A variety of surface modification techniques have been examined recently in an effort to increase the compatibility of lignin with polymer matrices [1]. The three main advantages of silane coupling agents are (i) their large-scale commercial availability, (ii) the presence of alkoxysilane groups that can react with OH-rich surfaces, and (iii) their variety of functional groups that can be tailored to fit particular polymer matrices [4]. This final attribute ensures at least a good level of compatibility between the reinforcing element and the polymer matrix, with the potential for covalent bonding. The interaction of silane coupling agents with lignocellulosic fibers, mainly cellulose and lignin, differs notably from their reaction with glass surfaces. Notably, only pre-hydrolyzed silanes have been observed to react with lignin surfaces in cellulose macromolecules. While chemical bonding is the most recognized mechanism by which organosilane coupling agents operate, alternative theories have been suggested, such as the interpenetrating network theory, which proposes that the polymer matrix diffuses into the silane interphase, creating an entangled network [5].

This study aimed to examine the impact of various silane coupling agents on the properties of NR/BR-based composites reinforced with a hybrid filler of CB and lignin. In this work, an NR/BR matrix containing 10 phr of lignin and 40 phr of CB as a hybrid filler, along with different types of silane coupling agents, was prepared using the dry-mix method. The effects of these silane coupling agents on the curing behavior, tensile properties, hardness, and compression set of the NR/BR/CB/lignin composites were then analyzed.

2. EXPERIMENTAL SECTION

2.1. Materials

Standard Malaysia Rubber Grade SMR10 was provided by the Rubber Research Institute of Malaysia (RRIM), while polybutadiene rubber (BR9000) was sourced from Beijing Yanshan Petrochemical Company, Ltd., a subsidiary of China Petroleum and Chemical Corporation. Kraft lignin (KL), used as a filler in the dual-filler system, was derived from kenaf biomass through an in-house isolation process. KL has an approximate particle size of 250 µm, a molecular weight of 3526 g/mol, and a dispersity (Đ) of 2.64. Silane coupling agents, including bis[3-(triethoxysilyl)propyl]tetrasulfide (TESPT), 3-octanoylthio-1-propyltriethoxysilane (NXT), and vinyltriethoxysilane (VTEO), were obtained from Sigma Aldrich, United States. All other chemicals used in this study were of standard commercial grade. Carbon black N220 was supplied by Cabot Corporation, while Bayer (M) Ltd provided additional reagents, including stearic acid (SA), treated distillate aromatic extracted (TDAE) oil, paraffin wax, N-phenyl-p-phenylenediamine (6PPD), zinc oxide (ZnO), N-tert-butyl-2-benzothiazylsulphonamide (TBBS), tetramethylthiuram disulfide (TMTD),), 2,2,4-trimethyl-1,2-dihydroquinoline (TMQ), and sulfur.

2.2. Preparation of Hybrid CB/Lignin-Filled NR/BR Compounds with the Presence of Silane Coupling Agent

In this work, various types of silanes were employed to enhance the interfacial compatibility between the dual filler CB/lignin surface and the rubber matrix, selected based on the quantity of mercapto groups in their structures. The in-situ technique was utilized to incorporate silane with dual CB/lignin into the rubber compound using a two-roll mill, with a mixing temperature of approximately 60°C. The rubber formulation is presented in Table 1, with loading varying in the range of 0.2–1.0 phr (2–10 wt.% based on lignin content).

2.3. Characterization of Hybrid CB/Lignin-Filled NR/BR Composites

2.3.1 Cure Characteristics

The rubber compound's curing properties were assessed using a rotorless rheometer (MDR 2000, Moving Die Rheometer, Alpha Technologies, OH, USA). A sample of the compound was placed in the oscillating chamber. The rubber compound's curing properties were assessed using a rotorless rheometer (MDR 2000, Moving Die Rheometer, Alpha Technologies, OH, USA). which operated at 0.5° arc with a frequency of 1.7 Hz at 150 °C, following ASTM D5289 standards. From the rheometer chart, key parameters such as minimum torque (ML), maximum torque (MH), and delta torque (MH – ML) were analyzed. Additionally, scorch time (tS2) was determined as the time required for the onset of crosslink formation, typically defined as the point where the torque increases by one unit from the minimum. The optimum cure time (tc90) represents the time needed to achieve 90% of the total torque change from minimum to maximum. The cure rate index (CRI) was calculated using Equation 1.

$$\text{Cure Rate Index (CRI)}=100/(t_{C90}-t_{s2}) \quad (1)$$

Table 1 Formulations of carbon black/Kraft lignin-filled NR/BR with different types of silane coupling agents and varied loading

Ingredients	Amount (phr)				
	NR/BR/ Silane 2	**NR/BR/ Silane 4**	**NR/BR/ Silane 6**	**NR/BR/ Silane 8**	**NR/BR/ Silane 10**
NR (SMR10)	50	50	50	50	50
BR (BR9000)	50	50	50	50	50
ZnO	5	5	5	5	5
SA	2	2	2	2	2
Carbon black (N220)	40	40	40	40	40
Kraft lignin	10	10	10	10	10
Silane[1]	0.2	0.4	0.6	0.8	1.0
TDAE	5	5	5	5	5
6PPD	2	2	2	2	2
TMQ	1	1	1	1	1
Paraffin Wax	2.5	2.5	2.5	2.5	2.5
TBBS	1.2	1.2	1.2	1.2	1.2
TMTD	0.35	0.35	0.35	0.35	0.35
Sulfur	1.5	1.5	1.5	1.5	1.5

[1] *Three types of silanes: (a) TESPT, (b) VTEO, and (c) NXT*
Tensile properties

Tensile testing of the composite samples was conducted using an H10KS tensometer (Hounsfield Test Equipment Co., Ltd., Croydon, UK) with a Type C dumbbell-shaped die, in accordance with the ASTM D412 standard. The specimens were subjected to a pulling force at a crosshead speed of 500 mm/min. The primary purpose of the tensile test was to assess the fracture strength and elongation at break of the material, enabling the evaluation and analysis of the mechanical properties of different composites and polymer blends.

2.3.2 Crosslink Density

NR/BR samples with dimensions of 30 × 5 × 2 mm^3 were prepared to measure the crosslink density of the rubber system. The samples were immersed in toluene inside a sealed bottle for 72 hours at room temperature (23 °C). After soaking, the samples were removed, immediately wiped, and weighed to obtain the swollen sample weight (WS). They were then dried at 60 °C for 30 minutes and weighed again to determine the dry sample weight (Wd). Using these weight measurements, the crosslink density (VC) was calculated based on the Flory–Rehner Equation 2:

$$V_c = \frac{-[\ln(1-Q_p)+Q_p+\chi {Q_p}^2}{V\times({Q_p}^{\frac{1}{3}}-(\frac{Q_p}{2})} \tag{2}$$

where Qp is the volume fraction of rubber in the swollen gel, χ is the Flory–Huggins interaction parameter between toluene and rubber, χ = 0.367, V is the molecular volume of toluene, and Qp is the volume fraction of swelling rubber, which was calculated by Equation (3):

$$Q_p = \frac{1}{[1+\left(\frac{w_s-w_d}{w_d}\right)\times\frac{\rho_p}{\rho_s}} \tag{3}$$

where ρp and ρs represent the density of the polymer and solvent, respectively.

2.3.3 Hardness

A Shore A durometer was used to measure the permanent indentation depth on the surface of the NR/BR composite, commonly referred to as hardness. The test was conducted at room temperature following the ASTM D2240 standard. Rubber samples were prepared with a thickness of 6.0 mm. Five measurements were taken at different locations on the same sample, and the average of these values was recorded.

2.3.4 Compression Set

The compression set test was conducted to evaluate the ability of the NR/BR composite to recover its original thickness after prolonged compressive stress under specific conditions of temperature, deflection time, and pressure. The test was performed at 70 °C for 22 hours, following the ASTM D395 standard. The percentage of compression set (C%) was calculated using Equation (4):

$$C\% = \frac{t_0-t_1}{t_1-t_s}\times 100 \tag{4}$$

where t_0, t_1, and t_S stand for the original sample's thickness, the sample's thickness following compression, and the spacer's thickness, respectively

3. RESULTS AND DISCUSSION

3.1. Cure Characteristics

The rheometric data are presented in Table 2. Although the silanization reaction between organosilane and the hydroxyl groups on the lignin surface significantly improves sulfur curing behavior, lignin substitution negatively affects curing characteristics, leading to a lower curing rate and longer curing time. However, no notable change was detected in delta torque. Since torque difference is directly related to the degree of crosslinking, it suggests that chemical crosslinks may have formed between the lignin surface and rubber chains during mixing and curing. TESPT delays the curing process due to the steric hindrance caused by its bulky end group. In contrast, NXT exhibits less steric hindrance compared to TESPT [6-7]. All silane types demonstrate an increasing trend in t90 as silane content rises. TESPT enhances the vulcanization reaction due to its sulfur donor effect, leading to shorter optimum cure times than blocked NXT silane. On the other hand, compounds containing NXT display the longest cure times because its mercapto group is blocked by a long aliphatic-containing carboxylic group, reducing silane-NR reactivity during processing. As a result, NR/BR coupling with silane occurs only during vulcanization.

Essentially, the NXT compound is more stable at high temperatures than TESPT and VTEO, providing better or longer scorch safety [7]. Furthermore, the blocking of mercaptosilane by an octanoyl group reduces premature release of sulfur, causing some premature vulcanization of the compound [8]. Moreover, the lower value of tS2 for TESPT might be due to the higher sulfur rank of TESPT compared to NXT and VTEO, which also leads to a higher reactivity of TESPT. This higher reactivity allows TESPT to react with rubber easily during the vulcanization reaction and reduces t_{S2} [2]. TESPT may "donate" some of its sulfur to the compound, implicitly increasing the amount of free sulfur and consequently providing extra crosslinking. Hence, the high cure rate index of TESPT was observed, indicating a faster crosslinking reaction compared to NXT and VTEO.

The compounds containing TESPT and NXT exhibit nearly identical minimum cure torque (M_L). However, after vulcanization, the maximum cure torque (M_H) and torque difference (M_H-M_L) of the NXT compound are lower than those of the TESPT compound. Increasing the NXT silane loading significantly reduces M_L, extends the induction period before vulcanization, and lowers M_H due to its plasticizing effect combined with silanization. The M_H value serves as an indicator of the rubber compound's crosslink density, where higher MH values correspond to greater crosslink densities. Therefore, the results suggest that TESPT leads to higher crosslink densities compared to NXT and VTEO. The lowest MH value is observed for VTEO, attributed to the absence of sulfur in its silane structure [8].

Compounds containing VTEO exhibit lower minimum torque (M_L) compared to those with TESPT and NXT. Increasing the VTEO concentration significantly reduces the maximum torque (M_H) and torque difference (M_H-M_L), likely due to the plasticizing effect of the large absolute amount of VTEO, which decreases the stiffness of the final vulcanizate. Additionally, the small VTEO molecules readily react with the hydroxyl groups of lignin but may not effectively shield the lignin surface. A higher VTEO concentration negatively impacts the curing behavior of the composites. The absence of sulfur in VTEO/lignin composites prevents coupling reactions via sulfur atoms, resulting in lower network density and, consequently, lower MH and MH-ML values compared to TESPT and NXT compounds. The torque difference (M_H-M_L) is closely related to crosslink density, as it represents both chemical bonding and physical interactions in the material. A greater torque difference indicates a higher degree of crosslinking in the rubber compound. Among the three silanes, TESPT shows the highest torque difference due to its bifunctional nature and higher sulfur content, which allows it to form crosslinks at multiple sites on both the lignin surface and rubber molecules. In contrast, VTEO is a monofunctional silane, capable of reacting with only a single site, leading to the lowest torque difference and fewer crosslinks in the vulcanizate [4].

Table 2 Curing characteristics of hybrid carbon black/Kraft lignin-filled NR/BR composites with different types of silanes

Sample	Optimum cure time, t_{90} (min)	Scorch time, t_{s2} (min)	Cure rate index (CRI)	Minimum torque, M_L (dN.m)	Maximum torque, M_H (dN.m)	Torque difference, M_H-M_L (dN.m)
NR/BR/CB50 (without lignin)	4.57	1.76	35.59	1.45	13.12	11.67
NR/BR/KL10	6.11	2.72	29.50	1.27	10.14	8.87
NR/BR/KL-TESPT 2	4.79	1.89	34.48	0.93	9.25	8.32
NR/BR/KL-TESPT 4	4.99	1.99	33.33	1.00	9.35	8.35
NR/BR/KL-TESPT 6	4.82	2.22	32.15	1.03	9.48	8.45
NR/BR/KL-TESPT 8	5.49	2.36	31.95	1.19	9.96	8.77
NR/BR/KL-TESPT 10	6.15	2.63	28.41	1.24	9.81	8.57
NR/BR/KL-NXT 2	5.94	2.86	32.47	1.10	8.73	7.61
NR/BR/KL-NXT 4	5.99	2.88	32.15	1.12	8.99	7.89
NR/BR/KL-NXT 6	6.33	2.98	29.85	1.16	9.05	7.89
NR/BR/KL-NXT 8	6.47	2.93	28.25	1.17	9.48	8.31
NR/BR/KL-NXT 10	6.53	3.05	28.74	1.15	8.90	7.75
NR/BR/KL-VTEO 2	5.58	2.31	30.58	1.28	8.83	7.55
NR/BR/KL-VTEO 4	5.58	2.62	33.78	1.15	8.79	7.64
NR/BR/KL-VTEO 6	5.81	2.77	32.89	1.13	8.66	7.53
NR/BR/KL-VTEO 8	6.03	3.01	33.11	1.00	8.58	7.58
NR/BR/KL-VTEO 10	6.25	2.95	30.30	0.93	8.26	7.33

3.2. Crosslink Density

Crosslink density is a key parameter used to assess the degree of filler-rubber interaction in vulcanized compounds. Figure 1 illustrates the effects of different silane types and concentrations on the crosslink density of lignin-filled compounds containing silane. Among the silanes studied, the NR/BR compound filled with lignin and TESPT exhibited the highest crosslink density, followed by those with NXT and VTEO. Initially, increasing silane content (0-6 wt.%) enhances crosslink density, suggesting better rubber-lignin compatibility and improved network formation. However, beyond 6 wt.%, crosslink density declines, likely due to excessive silane interfering with vulcanization or causing steric hindrance. The strong filler-rubber interactions observed in compounds containing sulfur-based silanes, such as TESPT and NXT, are not solely due to an efficient silanization reaction. They are also attributed to the enhanced adsorption of thiols onto the lignin surface, which facilitates interactions through hydrogen bonding and/or chemical bonding between the ethoxy groups of the silane and the hydroxyl groups of lignin [5, 6]. The exceptionally high crosslink density in the TESPT compound is primarily due to its tetrasulfide structure, which donates free sulfur into the system, promoting additional crosslink formation during mixing and curing. Furthermore, the high ethoxy group content in TESPT extensively reacts with the hydroxyl groups of lignin, enhancing filler-rubber interactions. In contrast, the compound with VTEO exhibits the lowest crosslink density, confirming its lower efficiency due to its minimal shielding effect when used with lignin in NR/BR compounds. Its capacity to lessen the quantity of accelerator trapped on the lignin surface and actively engage in the sulfur vulcanization reaction because of the sulfur in its molecular structure is responsible for the overall rise in crosslink density when a silane coupling agent is present. Its capacity to lessen the quantity of accelerator trapped on the lignin surface and actively engage in the sulfur vulcanization reaction because of the sulfur in its molecular structure is responsible for the overall rise in crosslink density when a silane coupling agent is present.

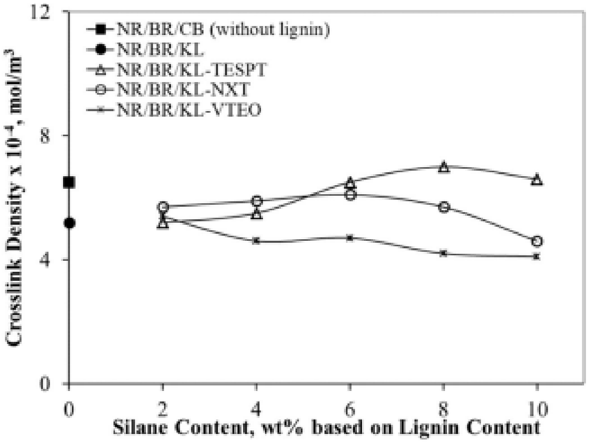

Figure 1. Effect of different silane systems on the crosslink density of CB/lignin-filled NR/BR composites.

3.3. Hardness

Figure 2 illustrates that incorporating silane into rubber compounds increases hardness. Among the tested compounds, those containing TESPT exhibit higher hardness values than those with NXT and VTEO silanes. This trend is consistent with the MH values and the modulus at 300% strain, as previously reported in Table 1 and Figure 1. These results indicate that TESPT contributes to a greater crosslink density compared to NXT and VTEO compounds [10]. The silanization process strengthens the bonds between lignin and rubber chains, and during vulcanization, strong covalent bonds form between the lignin surface and rubber via the silane bridge. This suggests an optimal balance between two reinforcement mechanisms: the presence of strong bonds and enhanced slippage of surface-attached rubber chains, which dissipate more energy through deformation. However, at low concentrations (up to 4 wt.% based on lignin content), the addition of NXT and VTEO silanes yields tensile strength comparable to that of TESPT. A further increase in silane content slightly improves the tensile strength of TESPT- and NXT-filled composites, whereas for VTEO-filled composites, it causes a slight decline.

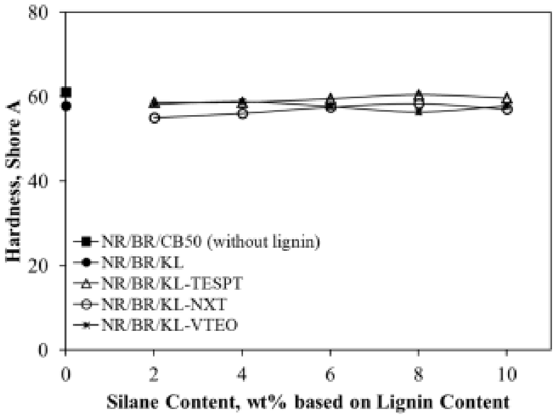

Figure 2. Effect of different silane systems on the hardness of CB/lignin-filled NR/BR composites.

3.4. Tensile Properties

The tensile properties of the lignin-filled NR/BR composites having different types of silane coupling agents are shown in Figure 3 and Figure 4. As can be seen, the values of M100 and M300 of NXT and VTEO compounds are significantly lower as compared with the TESPT compound. It is noteworthy that the composites with high elongation at break have low M100 and M300 modulus values. From the graph observed, the tensile modulus at 100% strain is in the range of 1.5-2.0 MPa. The composites with TESPT silane coupling agent showed significant improvement of the 100% modulus with increasing TESPT content, as demonstrated in Figure 4a. Whilst the lignin-filled NR/BR vulcanizates with NXT and VTEO show only a small change in tensile modulus with increasing silane contents. Furthermore, the stress-strain behavior is up to 300% strain independent of the chemically crosslinked content.

The elongation at break (EAB) results further support the sulfur contribution effect. As shown in Figure 3b, composites containing TESPT exhibit lower elongation at break compared to those with NXT and VTEO. This indicates that TESPT enhances the degree of cure more effectively than NXT and VTEO due to its sulfur-donating capability [9]. This is further validated by the swelling test results shown in Figure 1. The maximum deformation extent also aligns with MH, which serves as an indicator of crosslink density in the material. Additionally, tensile strength and modulus at 300% elongation exhibit a correlation with crosslink density, as illustrated in Figures 1, 3, and 4.

The ratio of tensile moduli at 300% to 100% elongation is commonly used to determine the reinforcement index. As shown in Figure 4, the reinforcement index of lignin-filled NR/BR composites with TESPT is the highest compared to those without silane and those containing NXT or VTEO. This corresponds to the higher crosslink density (Figure 1) and MH (Table 1), attributed to TESPT's higher sulfur content and sulfur-donating ability. As a result, increased chemical bonding between TESPT and rubber leads to a higher modulus. In contrast, the lower modulus of composites with VTEO indicates the absence of an effective coupling reaction.

3.5. Compression Set

Compression set testing is a standard method for evaluating the effect of silane content (wt.% based on lignin content), as shown in Figure 5.

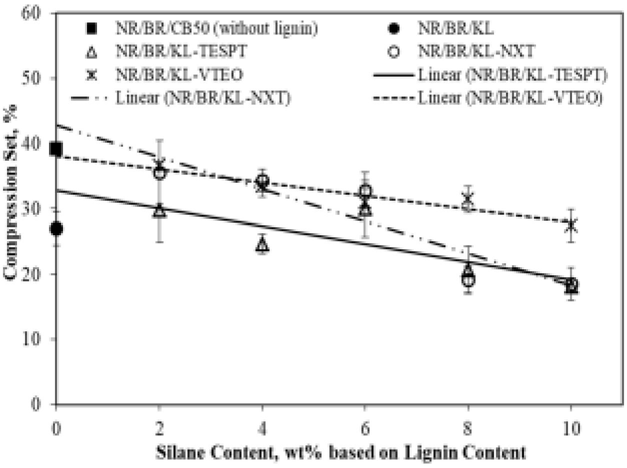

Figure 5. Effect of different silane systems on the compression set of CB/lignin-filled NR/BR composites.

The presence of a silane coupling agent significantly reduces the compression set as silane concentration increases. This reduction is attributed to a combination of factors, including improved filler dispersion and a higher degree of crosslinking upon the addition of silane. Additionally, as silane content increases, the higher triol content enhances the number of chemical crosslinks, thereby restricting polymer chain mobility. Consequently, the rise in crosslink density reduces the number of deformable physical crosslinks. With more chemical crosslinks, elastic recovery becomes stronger, and viscoelastic recovery occurs more rapidly. Since compression set and viscoelastic properties are directly linked to the number of physical network points, the increase in chemical crosslinks leads to improved performance [8]. Since TESPT can promote filler disagglomeration better than NXT and VTEO, it leads to good distribution of fillers. Thus, TESPT usually offers rubber composites with slightly lower compression set compared to the other two types of silane coupling agents. Besides, TESPT may also contribute extra sulfur to the rubber matrix. Thus, the number of polysulfidic bonds in the system with TESPT is expected to be higher compared to the system with the other two silane coupling agents. According to Sae-oui et al. [9], the sulfur contribution effect affected the number of polysulphidic bonds in the rubber system. These longer sulfidic bonds result in a small difference in compression set at elevated temperature as the concentration increases. It is mainly due to the less thermal stability of the shorter sulfidic linkages [10]. As can be observed for compounds with VTEO that are not involved in additional polysulfidic and/or disulfidic linkages, the compression set was improved by increasing the concentration, and the value is comparable with compounds with TESPT in higher concentrations.

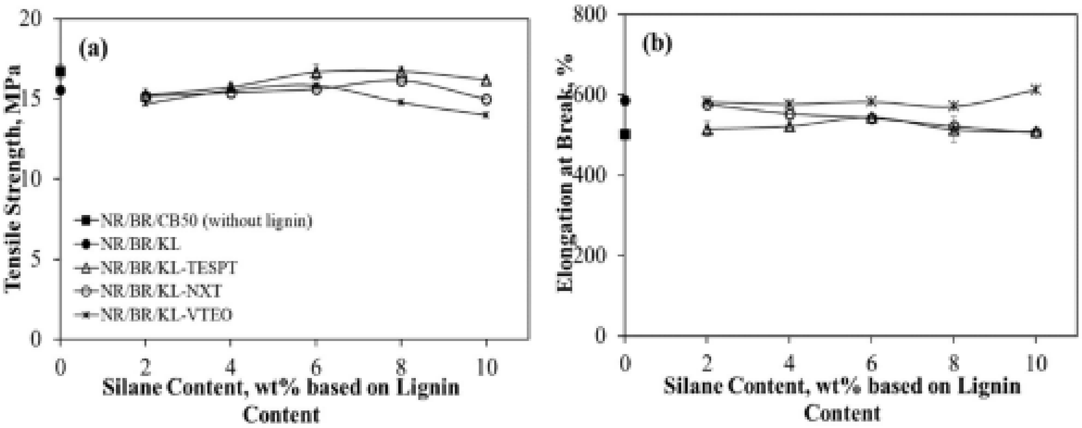

Figure 1. Effect of different silane systems on the tensile strength (a) and elongation at break (b) of CB/lignin-filled NR/BR composites.

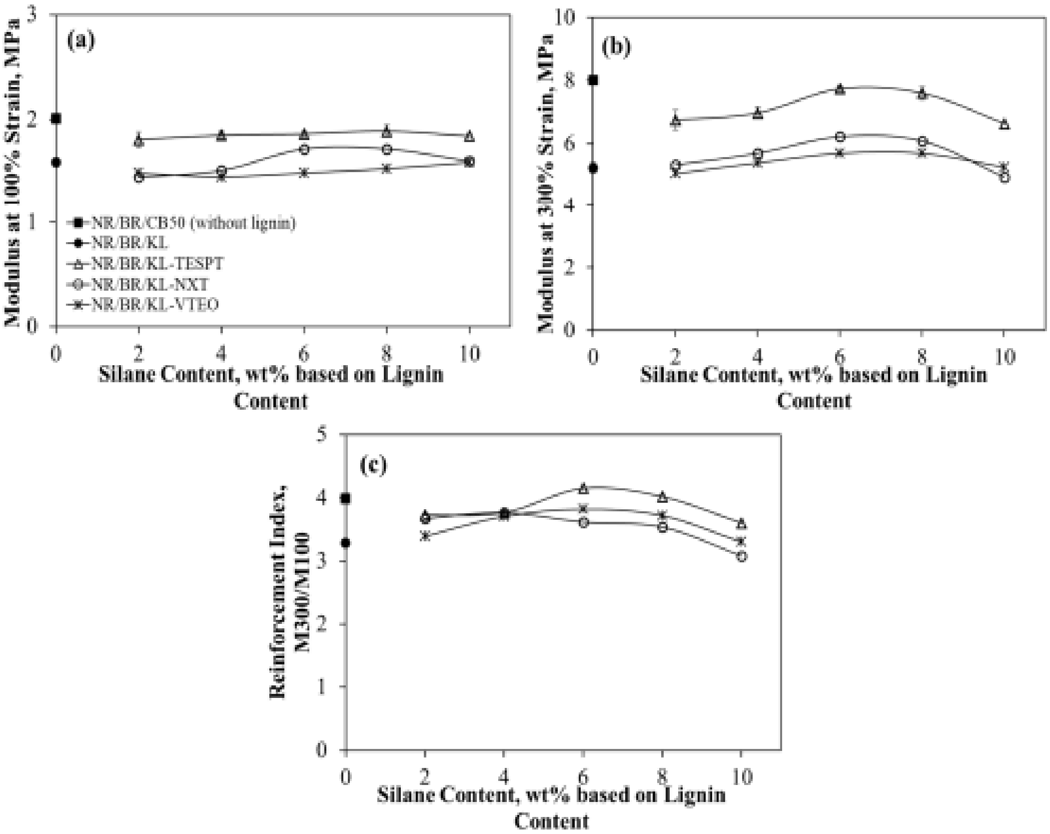

Figure 4. Effect of different silane systems on the tensile modulus at 100% strain (a), 300% strain (b), and reinforcement index (c) of CB/lignin-filled NR/BR composites.

4. CONCLUSION

In this study, lignin-filled NR/BR compounds treated with three different silane coupling agents (TESPT, NXT, and VTEO) exhibited varying effects on lignin reinforcement within the rubber matrix. Among them, VTEO demonstrated the weakest overall properties due to its low shielding efficiency and the presence of a vinyl group, which struggles to form a stable bridge with NR/BR rubber chains in the presence of sulfur. However, due to its less bulky structure and lower viscosity, VTEO more effectively reduces filler-filler interactions than TESPT and NXT, enhancing processability. Meanwhile, NXT improves composite properties, including cure behavior, crosslink density, and mechanical performance, compared to composites without silane. However, its properties remain slightly inferior to those of TESPT, primarily due to its lower crosslink density, as TESPT possesses an inherent "sulfur-donating" effect. It can be concluded that TESPT offers superior reinforcement compared to NXT and VTEO, primarily due to its dominant sulfur contribution effect in this system.

ACKNOWLEDGMENTS

The authors acknowledge the support from the School of Materials and Mineral Resources Engineering (SMMRE), Universiti Sains Malaysia (USM), Prince of Songkla University, Pattani Campus (PSU), Fundamental Research Grant Scheme (203.PBAHAN.6071350), USM Bridging Grants (304.PKIMIA.6316041) and the Postgraduate Research Attachment (PGRA) Fund, Institute of Postgraduate Studies, USM.

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