

Modified Natural Rubber Based on the Sulphur Curing System as Rubber Compound Formulation on Basic Design of Seismic Bearing

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Abstract

Indonesia is highly susceptible to earthquakes, with the southern and western coasts of Java and Sumatra being the most vulnerable regions. Due to this vulnerability, it is necessary to establish a culture of disaster mitigation in the most fertile and heavily populated islands to reduce the number of fatalities and economic losses caused by earthquakes. One of the promising real-world seismic base isolation methods is using a rubber seismic bearing constructed of rubber and metal layers. This study aimed to examine the typical behavior of natural rubber compounds subjected to various sulfur-curing processes as basic designs in the manufacture of rubber seismic bearings. The experiment was carried out by arranging the sulfur curing system into three categories, namely an efficient which applied N-cyclohexyl-2-benzothiazole sulfonamide (CBS)/Sulfur (S) ratio as 1.4/0.15 and 1.4/0.25, semi-efficient with CBS/S ratio of 1.4/1.4 and 1.4/1.7, and conventional with CBS/S ratio of 1.4/3.0 and 1.4/3.5. The results showed that the ideal modification condition for NR macromolecule chain found in seismic-bearing rubber compound was a semi-efficient sulfur curing system with a CBS/S ratio of 1.4/1.4. It suggested that a semi-efficient sulfur curing system was appropriate for developing rubber compound formulation for seismic rubber bearings, mainly for low-damping types.

Keywords: Earthquakes, damping, mechanical properties, natural rubber, seismic isolator

1. INTRODUCTION

In the Pacific Ring of Fire, Indonesia is highly susceptible to natural disasters such as earthquakes, tsunamis, and volcano eruptions ¹. Specifically, areas along the Western Coast of Sumatra and the Southern Coast of Java are vulnerable to strong-magnitude earthquakes ^{2,3}. History records that a massive earthquake triggered another natural disaster, resulting in extensive human casualties and severe damage to infrastructure and settlement ^{4,5}. It necessitates mitigating structural damage during earthquakes, particularly in both areas due to their high fertility and population densities. Previous investigations have shown that collapse structure

contributes to significant human casualties and economic losses ⁶.

Installation of seismic base isolation is a practical solution to mitigate the high magnitude of seismic ground motion by providing lateral flexibility and energy dissipation by inserting the isolation device between the foundation and building structure ^{7,8}. Seismic base isolators absorb or reflect part of seismic energy input into the structure during earthquake events ⁹. These isolators are commonly classified into lead rubber bearing (LRB), low-damping rubber bearing (LDRB), and high-damping rubber bearing (HDRB) ¹⁰. According to EN 15129, dynamic properties analysis of seismic bearing consists of hysteresis and shear modulus parameters.

LDRB requires hysteresis values below 6%, while HDRB is characterized by a seismic isolator that possesses hysteresis above 6% ¹¹. Both parameters play an important role in determining the design of rubber seismic bearings primarily based on the location of seismic bearing installation ¹².

Hwang et al. ¹³ summarized that the hysteresis behavior of elastomeric seismic bearing significantly depends on several factors, namely compounding, vulcanization, excitation frequency, ambient temperature, experienced shear strain level, and axial load. Furthermore, Saedniya and Talaeitaba ¹⁴ expressed that the most critical step in modeling an elastomeric isolator is defining the materials, particularly the selection of rubber. This selection determines rubber composite layers' compounding and vulcanization process within a laminate elastomeric seismic bearing. The rubber used for seismic isolators must possess good damping properties, high elasticity, strength, and flexibility in horizon direction to absorb seismic waves and resist damage ^{15,16}. Natural rubber (NR), polychloroprene (CR), and isobutylene isoprene rubber (IIR) have been used as primarily based materials for structural bearing for several decades ¹⁷.

NR is a biopolymer composed of repeating monomer units of isoprene to form a 1.4 cis configuration of polyisoprene macromolecular chain ¹⁸. To improve the characteristics of rubber composite, the NR biomolecule is modified through a crosslink curing reaction. Tamasi and Kollar ¹⁹ stated that NR-based composite, even with a similar rubber compound formula, shows different curing behaviors and properties depending on the system used. Furthermore, Zhao et al. ²⁰ claimed that sulfur, accelerator, and activator play a significant role in the sulfur-curing reaction of NR. The arrangement of these three components results in variation in network structure, including density and type of crosslinks,

leading to a change of physical and mechanical properties of rubber vulcanizate.

The study aimed to experimentally evaluate unfilled NR vulcanizate's physical and mechanical properties by modifying the NR macromolecule chain at various curing systems. The unfilled vulcanizate refers to vulcanized rubber containing no filler materials except additives necessary for vulcanization. The curing system specifies the crosslink network types and density, which are known to influence the property of the unfilled NR vulcanizates significantly. The result is expected to produce an appropriate curing system, which can be further used to develop the design of a compound formula for a seismic rubber bearing pad for a seismic base isolator.

2. RESEARCH METHODS

Materials and Methods

NR, specifically Standard Indonesian Rubber (SIR) 20 grade, was provided by the Indonesian Rubber Research Institute, Bogor, West Java, Indonesia. Furthermore, all technical grade rubber chemicals consisted of zinc oxide (ZnO, Rubber Activator, Lanxess, Germany), stearic acid (Aflux 52, Rhein Chemie, Germany), N-cyclohexyl-2-benzothiazole sulfonamide (CBS, Kemai Chemical Co Ltd, China), and sulfur (Midas SP-325, Miwon Chemical Co Ltd, Korea) were purchased from PT Multi Citra Chemindo Nusa, Jakarta, Indonesia. The formula of the unfilled NR compound was designed as presented in **Table 1**. Rubber compound formula was arranged based on various curing systems, including an efficient system that applied CBS/sulfur ratio at 1.4/0.15 and 1.4/0.25, an efficient system for CBS/sulfur ratio of 1.4/1.4 and 1.4/1.7, as well as conventional curing system for CBS/sulfur ratio of 1.4/3.0 and 1.4/3.5, respectively. Rubber and chemical additive compositions were determined per hundred rubber (phr) units.

Table 1. NR compound formulation

Materials	Composition (phr)					
	Efficient		Semi Efficient		Conventional	
NR – SIR 20	100	100	100	100	100	100
ZnO	5	5	5	5	5	5
Stearic acid	2	2	2	2	2	2
CBS	1.4	1.4	1.4	1.4	1.4	1.4
Sulfur	0.15	0.25	1.4	1.7	3.0	3.5

NR compounding was carried out according to ASTM D 3182 using a laboratory-scale two-rolled open mill (Berstorf, Germany). The procedure commenced by masticating SIR 20 into the softened mass to facilitate the mixing of solid-phase rubber chemicals. Rubber chemicals were added into a softened mass of NR through a specific order, namely activator (ZnO and stearic acid), accelerator (CBS),

and vulcanizing agent (sulfur). The mixture of SIR 20 was blended and re-milled, followed by adding rubber chemicals into a homogenized unfilled NR compound. Subsequently, the unfilled NR compound obtained matured for at least 24 hours before further processing. The curing reaction of NR was conducted at a solid-state reaction at 150 °C using a hydraulic press machine (Kobe Machinery Co Ltd, Japan). For every

condition, the time of the curing reaction was in accordance with the optimum curing time ($T_c 90$) resulting from the curing characteristic test.

As a curing characteristics sample, 50 g of NR compound was weighted. The test was conducted by using Moving Die Rheometer (MDR) Alpha 2000 (Alpha Technology, USA) at 150 °C for 30 minutes. The remaining rubber compound was pressed using a hydraulic press machine (Kobe Machinery Co Ltd, Japan) at 150 °C to be used for evaluating physical and mechanical properties. The test parameters were hardness (ASTM D 2240²¹, Shore A Durometer, Karl Frank GmbH, Germany), tensile strength and elongation at breaks (ASTM D 412²², UTM Llyod 2000R), tear strength (ASTM D 624²³, UTM Instron), rebound resilience (ASTM D 1054²⁴, Tripsometer), and compression set (ASTM D 395²⁵, E-Set Tester). The compression set test was conditioned at room temperature for 72 hours.

Crosslink density was determined using the Flohry-Rehner equation, using a swelling method, which was performed following the guidelines in ISO 1817. The unfilled NR vulcanizate test sample was cut to 20 x 20 x 2 mm. Each test piece was weighed using an electrical balance before being immersed in toluene for 72 hours at room temperature in dark conditions. The swollen sample was removed from the solvent; residual toluene was removed from the sample surface before being weighed. Subsequently, the samples were dried at room temperature for 24 hours, and their weights were measured to obtain the final weight. Crosslink density was determined through the following eq (1) and eq (2).

$$v = - \frac{\ln(1 - v_{ro}) + v_{ro} + \chi_1 v_{ro}^2}{2 V_1 \left(v_{ro}^{\frac{1}{3}} - \frac{v_{ro}}{2} \right)} \quad (1)$$

$$v_{ro} = \frac{\frac{(Ds - Ff Aw)}{\rho r}}{\frac{(Ds - Ff Aw)}{\rho r} + \frac{As}{\rho s}} = \frac{\frac{(Ds)}{\rho r}}{\frac{(Ds)}{\rho r} + \frac{As}{\rho s}} \quad (2)$$

v : crosslink density (mol/cm³), $v_{ro} v_{ro}$: volume fraction of swollen rubber, $\chi_1 \chi_1$: coefficient interaction of NR and toluene as 0.393, V_1 : molar volume of toluene as 106.3 cm³/mol; D_s : final weight of rubber, F_f : volume fraction of filler as 0 (unfilled), A_w : initial weight, A_s : toluene weight absorbed in

rubber, ρ_r : density of rubber as 0.9203 g/cm³, ρ_s : density of toluene as 0.867 g/cm³.

A dynamic shear modulus test was conducted using UTM (MTS, USA). Subsequently, the unfilled NR compound test sample was molded and formed into a double shear test piece (quadruple) at dimensions 70 x 20 x 5 mm. The shear modulus test was carried out at room temperature, 0.2 Hz constant frequency, and 100% strain amplitude. Shear modulus and damping values were determined during the third deformation cycle.

3. RESULTS AND DISCUSSION

The curing characteristic test for the unfilled NR compound at various curing systems was summarized in **Table 2**. The result indicated parameters such as minimum torque (ML), which correlated to the processability of the unfilled NR compound. Maximum torque (MH) expressed the stiffness of vulcanized rubber, and torque difference (MH-ML) reflected crosslink density and scorch time (ts_1), which showed the period before rubber vulcanization started. In contrast, optimum curing time (tc_{90}) represented the time required by the rubber compound to reach 90% of the state of cure²⁶.

As illustrated in **Table 2**, an increase in sulfur content is attributed to the elevation of torque modulus (MH, ML, and MH-ML), followed by shortening ts_1 , and tc_{90} . Higher sulfur-containing rubber molecules created more three-dimensional sulfur bridges of NR macromolecular crosslink in the form of monosulfide, disulfide, and polysulfide networks. This linkage caused increasing rigidity of the unfilled NR vulcanizate, resulting in rotor movement during curing characteristic analysis to produce higher force, as reflected by torque value. Meanwhile, higher incorporation of sulfur into NR molecules facilitated the curing reaction, as indicated by a reduction in ts_1 and tc_{90} .

Unfilled NR compounds designed at various curing systems produced different vulcanization behaviors, as confirmed by the photograph illustrated in **Figure 1**. During the early phase of the photograph, an efficient curing reaction comprising 0.15 and 0.25 phr of sulfur showed a declivous cure curve. Meanwhile, a conventional curing reaction with a higher sulfur loading had the sharpest cure curve. A semi-efficient curing reaction was also observed, indicating that increased sulfur content facilitated the curing reaction.

Table 2. Curing characteristic of unfilled NR compound

Parameters	Efficient		Semi Efficient		Conventional	
	1.4/0.15	1.4/0.25	1.4/1.4	1.4/1.7	1.4/3.0	1.4/3.5
MH, dNm	1.53	2.30	6.48	7.30	10.17	9.60
ML, dNm	0.24	0.46	0.48	0.25	0.45	0.43
MH – ML, dNm	1.29	1.84	6.00	7.05	9.72	9.17
ts1, min:sec	15.54	11.36	5.37	4.56	3.54	3.41
tc90, min:sec	19.59	17.09	8.45	7.33	7.19	8.13

The mechanism of curing reaction in NR compound depends on the crosslink formation and chain entanglements. A greater cure curve indicates a higher crosslink density of unfilled NR compound, leading to the production of more stiff and rigid rubber²⁰. This mechanism is particularly evident during the curing and post-cure phases. Many covalent disulfide and polysulfide (S-S) bonds among unfilled NR macromolecular chains from conventional curing reactions resulted in weak interaction. It caused a decrease in heat resistance, as indicated by the reversal pattern of the cure curve over time. In contrast, an efficient curing reaction showed a marching tendency, as it was dominated by mono-sulfidic (C-S) bonds among the macromolecular chain. C-S bonds showed better thermal stability than S-S bonds; thereby, the cure curve of the semi-efficient curing reaction presented a plateau curve, which was predicted to contain equal C-S and S-S bonds²⁷.

As shown in **Figure 2**, the crosslink density value of each unfilled NR compound from the Flohry-Rehner Equation was comparable to MH-ML obtained by curing characteristic analysis. The conventional

curing system showed a significant improvement in crosslink density value with the addition of 3 phr of sulfur. However, a slight reduction of crosslink density was observed at unfilled NR vulcanizate cured by incorporating 3.5 phr of sulfur. This reduction was due to the breakdown of the disulfide and polysulfide bridge between the polyisoprene macromolecular chains during the post-cured condition²⁸.

The hardness of the unfilled NR vulcanizate described in **Figure 3** showed that the hardness level aligned with the sulfur content used in the curing reaction. Higher sulfur dosage tended to produce harder unfilled NR vulcanizate, generating more crosslink networks among polyisoprene macromolecular chains bridged by sulfidic bonds. Increasing crosslink density restricted the mobility of inter crosslink chains, leading to a significant rise in hardness for the unfilled NR vulcanizate. However, a slight reduction in hardness was shown by unfilled NR vulcanizate with the addition of 3.5 phr of sulfur. This reduction was correlated with decreased crosslink density of the vulcanizate²⁹.

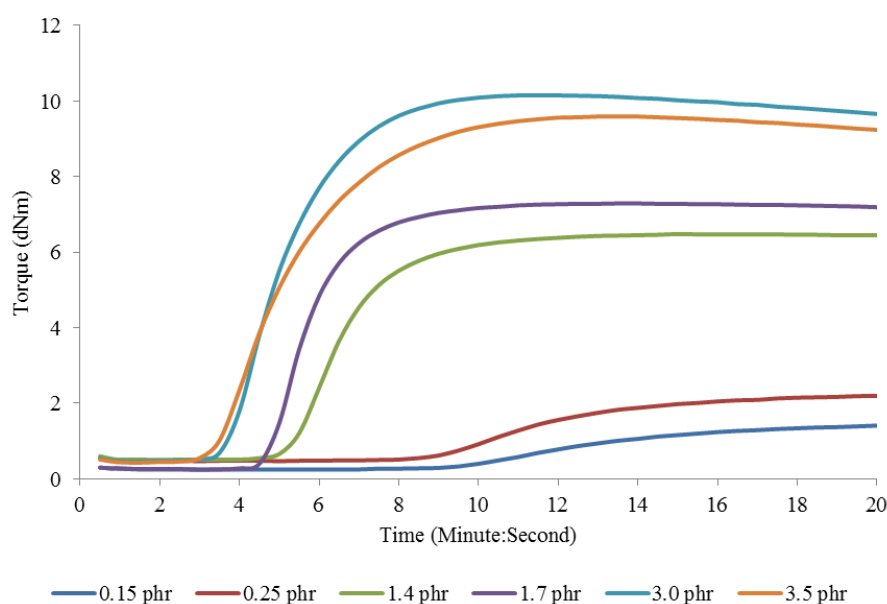


Figure 1. Rheograph of the unfilled NR vulcanizate

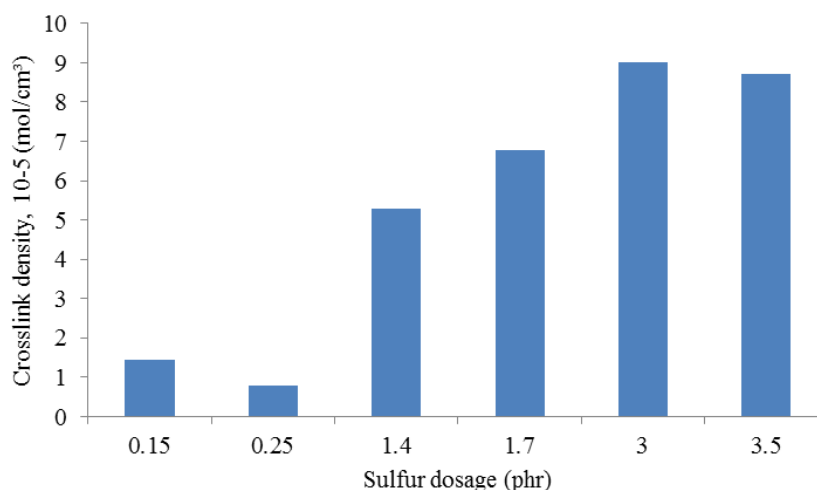


Figure 2. Crosslink density of the unfilled NR vulcanizate

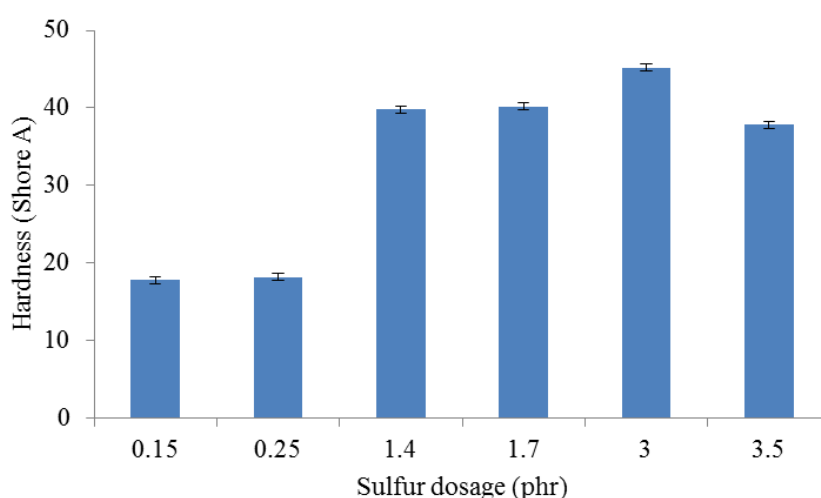


Figure 3. Hardness of the unfilled NR vulcanizate

Tensile properties, which represented strength and elongation at break parameters of the unfilled NR vulcanizate, were shown in **Figures 4 and 5**, respectively. A semi-efficient curing reaction presented the highest tensile strength, adding 1.7 phr of sulfur. Furthermore, **Figure 5** exhibits the highest elongation at breaks, resulting in an efficient curing system with the addition of 0.15 phr of sulfur equal to 0.25 phr sulfur. In this condition, tensile strength reached its maximum value, followed by a decrease due to the rise in sulfur content. Meanwhile, unfilled NR vulcanizate crosslinked by a sulfidic bridge formed strain-induced crystallization during stretching. More crosslink linkage created increased rubber crystallinity, resulting in higher tensile strength. Zhao et al.²⁰ stated that an excessively high crosslink density value significantly reduced the distance between two crosslink points was excessively, thereby decreasing the mobility of rubber molecular chains, as indicated by a conventional curing reaction. The rise of rubber rigidity also caused

a reduction in elongation at breaks, indicating that a higher degree of crosslink density introduced a lower elasticity into the polymer, thereby decreasing the elongation at breaks³⁰.

The result of tear strength measurement, as presented in **Figure 6**, showed a similar pattern to tensile strength. Bhowmick et al.³¹ stated that the threshold tear strength was found to be dependent on filler loading, the nature of the filler, and the small strain modulus. Since this experiment was conducted without filler addition, the tear strength properties were strongly dependent only on a small strain modulus. A semi-efficient curing reaction showed superior tear strength properties to an efficient or conventional curing reaction due to more crosslink linkage formation among the polyisoprene macromolecular chains. It indicated that increasing crosslink density also accounted for the reduction of the strain modulus, contributing to the high tear strength.

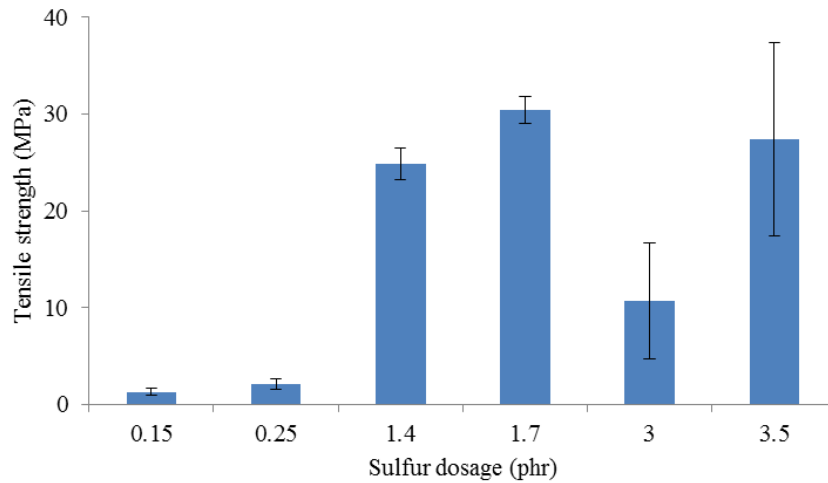


Figure 4. Tensile strength of the unfilled NR vulcanizate

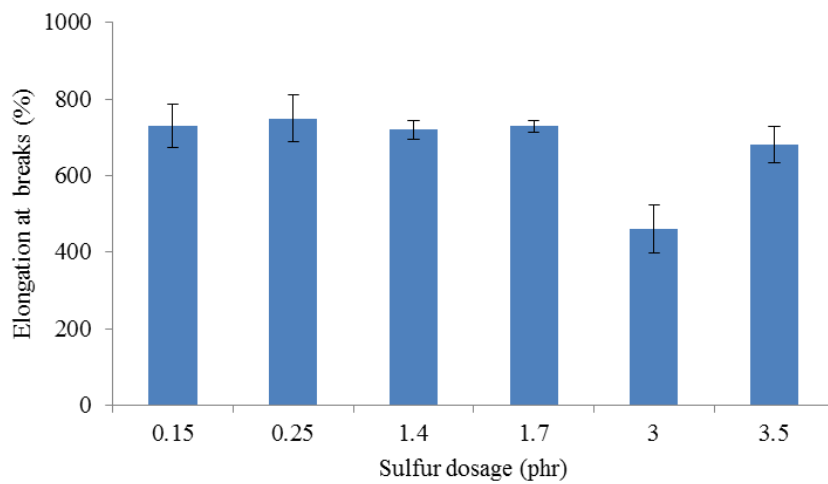


Figure 5. Elongation at breaks of the unfilled NR vulcanizate

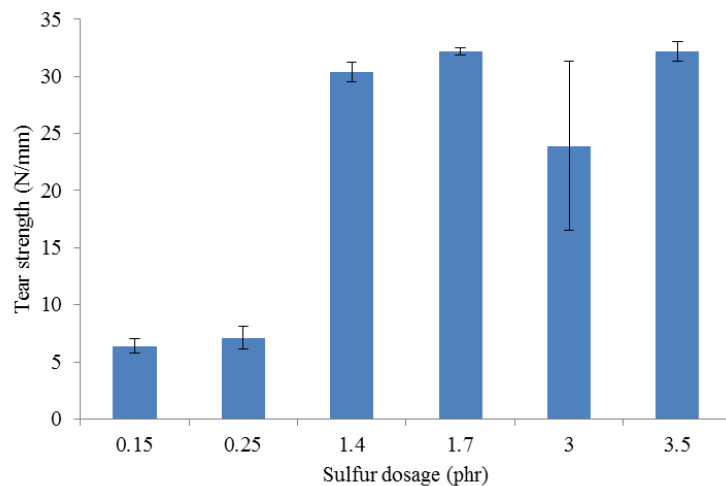


Figure 6. The tear strength of the unfilled NR vulcanizate

The high crosslink density due to increased sulfur loading of unfilled NR vulcanizates was responsible for their greater ability to bear subjected compression load, as evidenced by the results of the compression set presented in **Figure 7**. The results showed that the compression set of the unfilled NR

vulcanizate in semi-efficient and conventional curing systems became comparable when the sulfur content was up to 3.5 phr. In this range, both unfilled NR vulcanizate presented greater elasticity. Movahed et al.³² stated that the compound with the least compression set had the highest level of elasticity and

the least viscous properties. Generally, a lower compression set percentage is desirable, indicating a greater ability to return to its original shape after being compressed to a specific deformation for a certain period.

Rebound resilience plays a significant role in affecting seismic rubber-bearing characteristics. Generally, seismic rubber bearing requires good damping properties, which commonly correlates with better rebound resilience value. This rebound resilience parameter indicates rubber material's ability

to absorb mechanical energy during impact. A lower value indicates higher damping properties, measuring the ratio of the energy returned to the energy applied for deformation by an indentation due to a single impact³³. Meanwhile, unfilled NR vulcanizate produced by semi-efficient and conventional curing reactions pronounced a higher percentage of rebound resilience due to its greater elasticity, indicating an increased bounced or lower damping ability. The result of the rebound resilience measurement is shown in **Figure 8**.

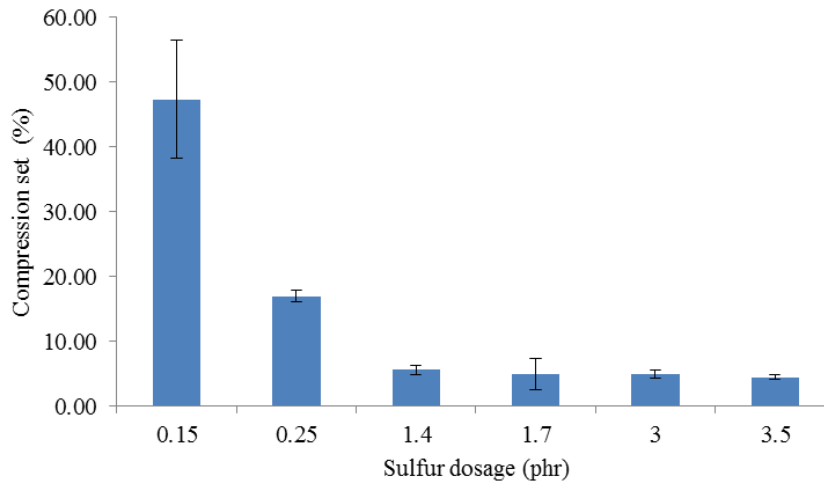


Figure 7. Compression set of the unfilled NR vulcanizate

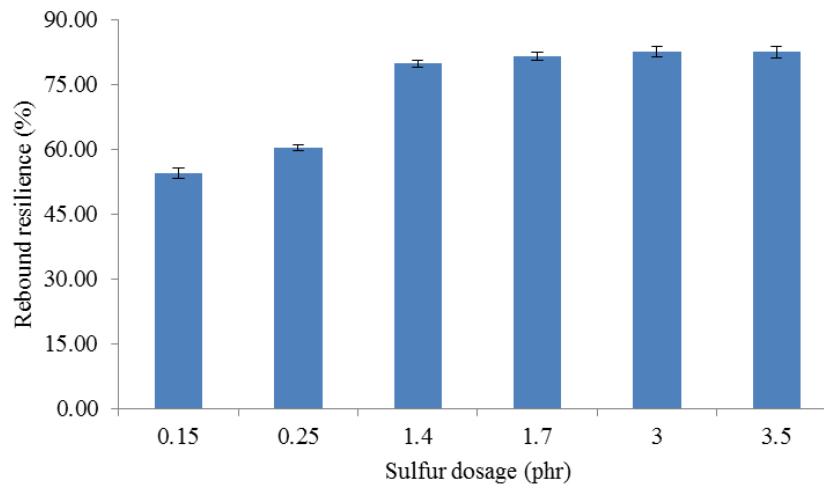


Figure 8. Rebound resilience of the unfilled NR vulcanizate

The elliptical curve shown in **Figure 9** represented the hysteresis of shear force versus lateral displacement of unfilled NR vulcanizate cured using various curing systems. The area of this elliptical hysteresis represents energy absorbed by the damping effect of rubber³⁴. As plotted in Figure 9, the area of elliptical hysteresis showed a very narrow area. It indicated that the unfilled NR vulcanizate had less horizontal stiffness, resulting in minimal energy dissipation during the structural vibration. The damping value of this type of unfilled NR vulcanizate

ranged from 0.6% to 2.3%, which was appropriate for the LDRB type. Meanwhile, the highest damping properties were obtained from an efficient curing system with 0.25 phr sulfur addition formulation.

In designing seismic rubber bearings, damping properties play an essential role in the evaluation of seismic device performance³⁵. Referring to the result of the study, the curing system highly affects the mechanical and damping properties of the seismic rubber bearing. Therefore, determining the sulfur dosage or the curing system by arranging the ratio of

sulfur and accelerator in the formulation of rubber compound highly depends on the type of rubber seismic bearing to be designed. Due to the low energy dissipation of the unfilled NR vulcanizate, it is essential to conduct the reformulation of the rubber compound. The addition of reinforcing filler and

hardener petroleum-based resin was proposed by Ismail et al.³⁶ and Islam et al.⁷ to increase the damping properties of NR. Another effort included the addition of dampers such as viscous or yielding dampers, as Shahabi et al.³⁷ recommended.

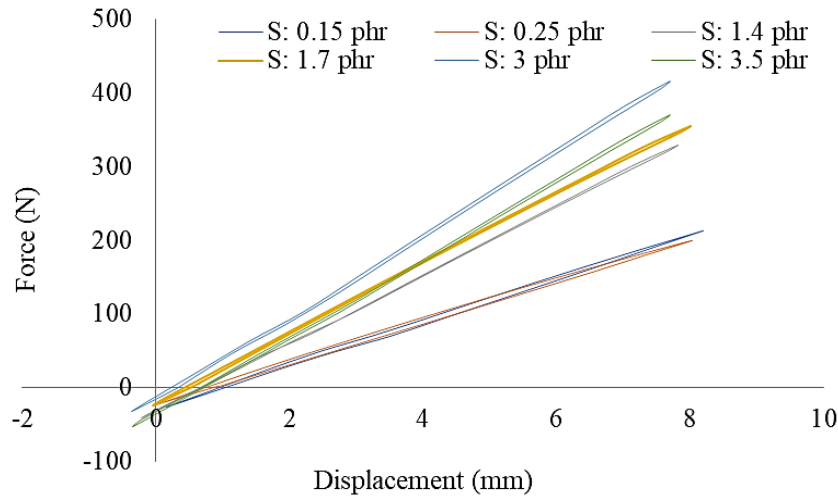


Figure 9. Hysteresis curve of unfilled NR vulcanizate

4. CONCLUSIONS

In conclusion, this study showed that the properties of unfilled NR vulcanizate were influenced by the curing system applied in modifying NR macromolecules. Differences in sulfur content within the unfilled NR compound formulation led to the variation in crosslink density. The density increased linearly with the total sulfur content added to the NR compound formulation. A higher crosslink density level impacted the rise of hardness, tensile strength, tear strength, and rebound resilience. Meanwhile, elongation at breaks and compression sets was reduced, leading to a distinct correlation between the rebound resilience. The lower rebound resilience indicated improved dynamic properties of the unfilled NR vulcanizate. Based on the results, the dynamic properties of unfilled NR vulcanizate ranged from 0.6% to 2.3%. It indicated that a semi-efficient curing system resulted in better-unfilled NR properties compared to a conventional and efficient curing system, particularly with a sulfur content of 1.4 phr. Therefore, a semi-efficient curing system was considered appropriate for developing rubber compound formulation for seismic rubber bearings, particularly the LDRB type.

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