## PHYSICAL PROPERTIES OF SBR COMPOUNDS FOR THE EFFECT OF DIPHENYL GUANIDINE AND GRAIN DIRECTION

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ABSTRACT: This study investigates the processability and physical properties of styrene butadiene rubber (SBR) compounds, particularly focusing on the effect of additive and grain direction of the two-roll process. SBR composites were prepared at varying diphenyl guanidine (DPG) levels of 0.5 and 1.5 phr, and the compounds were cut according to the machine direction (MD) and transverse direction (TD) prior to vulcanization. A lower DPG amount (0.5 phr) in SBR compounds significantly improved cure rate index and torque difference compared to a higher DPG amount (1.5 phr). The physical properties revealed that SBR with lower DPG content outperformed SBR with higher DPG, particularly in the TD orientation. The findings were further supported by swell measurement, toluene uptake and SEM analyses to assess the crosslinking and morphology. The study enhances understanding of the relationship between chemical treatment and grain orientation on the physical performance of rubber composites. It offers valuable insights for improving manufacturing processes and product quality in the rubber industry.

KEYWORDS: Styrene Butadiene Rubber; Cure Characteristics; Swell Measurement; Grain Direction

## 1.0 INTRODUCTION

Styrene-butadiene rubber (SBR) is a synthetic rubber derived from the polymerization of styrene and butadiene monomers. According to Choi et al. [1],the arrangement of 1,2-units, cis-1,4-units, and trans-1,4-units in SBR significantly influences its material properties. The alternating long chain of styrene and butadiene monomers in SBR balances rigidity and elasticity, which is crucial for its final properties. The concentration of styrene, typically between 10% and 25%, and the amount of butadiene are key determinants of SBR's properties. SBR's versatility is evident in its wide range of applications that includes tire [2], footwear [3], manufacturing [4], and construction [5] industries. Its applications extend to mechanical goods, shoe bottoms, outsoles, waterproof materials, industrial belts, sporting goods, wire and cable insulation, coatings, foam products, etc. SBR exhibits excellent heat, aging and wear resistances, and its elasticity allows it to withstand significant stretching without permanent deformation [6].

The unique mechanical characteristics of SBR compounds result from their different chemical compositions and molecular structures. According to Yaacob et al. [7], SBR maintains its characteristics over a wide temperature range due to their excellent aging stability, contributing to its durability. Most studies on SBR composites addressed the effect of formulation, such as types of fillers (silica, carbon black, graphene oxide etc.) [8,2], types of functionalization [9] and resins [10]. Yamada et al. [9] investigated the effect of different functional groups in SBR. The study found that an amine functional group at the SBR polymer chain used in silica-filled compounds can accelerate the silanization reaction. Meanwhile, Wang et al. [11] examined several levels of crystallinity, including trans-1,4polvisoprene (TPI) and trans-1,4-poly(butadiene-isoprene) copolymer rubber (TBIR), alongside SBR. Their study on formulation reported a wide range of composition and structural alterations that directly affect the mechanical properties of the resultant SBR compounds.

Still, the processing variables also dictate the properties of the SBR composites. Along with the effect of fillers, Kim et al. [8] also investigated the effect of curing temperatures between 150, 160 and 170 °C. The cure analysis indicated that the temperature difference ( $\Delta$ T) showed a decreasing trend with an increase in the cure temperature

due to the reduction in the total crosslink density. The production of rubber products involves several stages, transforming raw rubber materials into finished goods. These stages include preparing raw materials, mixing, compounding, and finally, curing and finishing [12]. During the preparation stage, elastomers, fillers, chemicals, and specialty additives are mixed and compounded in a mill under shear in a specific sequence. Then, compound material is then shaped using a two-roll mill machine, resulting in sheets that offer flexibility, elasticity, and abrasion resistance [13]. The two-roll mill process is a common method for mass-producing rubber goods. Despite the versatility, the compounds quality highly depends on the compounders' skills. Therefore, it challenges the final properties of the rubber products that may relate to the grain orientation.

Vulcanization or curing is a process to bond polymer chains chemically. Kruželák et al. [14] mentioned that sulfur and peroxides are commonly used agents, which form crosslinks between polymer chains. The crosslinks enhancing the rubber's mechanical strength, elasticity, and heat resistance. In the vulcanization of SBR, sulfur is often used. This process improves the mechanical qualities, elasticity, and wear resistance of SBR. Li et al. [15] stated diphenyl guanidine (DPG) is a co-accelerator used alongside sulfur. It increases crosslinking speed and reduces the vulcanization time required for the rubber compound. DPG regulates the vulcanization process to ensure an optimal rate, enhancing the rubber's modulus, tensile strength, and other mechanical properties. This study addresses the knowledge gap in SBR composite studies on the effect of DPG treatment (0.5 and 1.5 phr) and two-roll mills grain orientations (MD and TD) on the cure characteristics and physical properties. The study compares the effect on curing time, torque values, cure rate index, hardness, and density. The results were supported by swell percentages, toluene uptake, and morphological analysis using scanning electron microscopy.

#### 2.0 METHODOLOGY

#### 2.1 Raw Materials and Preparation

The styrene butadiene rubber (SBR) compounds were provided by Rubber Leisure Products Sdn Bhd. Two compounds were incorporated with 0.5 phr (low DPG) and 1.5 phr (high DPG). Table 1 summarizes the details of the samples. The two-roll mill compounding process was performed by personnel in the Rubber Leisure Product (M) Sdn. Bhd. (RLPSB). The mixed rubber compound was fed into the two-roll mill

machine using a specific formula: SBR rubber, curing agent, activator, accelerator and other ingredients. After undergoing a two-roll mill process becoming sheet (Figure 1), the compounds received from RLPSB were fabricated and simultaneously vulcanized using a hot press machine. During this step, the desired weighed compound was filled into a mold with a 3 mm thickness. Prior to vulcanization, the sheet was cut from the bulk according to the samples for further testing and analysis. Two different grain directions, Machine Direction (MD) and Transverse Direction (TD) were used for testing and analysis. Each rubber composite sample was subjected to five times trials.

Table 1. The details of obly samples				
SBR (high DPG)	SBR (low DPG)			
0.5	1.5			
MD	MD			
TD	TD			
	SBR (high DPG) 0.5 MD TD			

Table 1. The details of SBR samples



Figure 1: Styrene butadiene rubber compound (0.5 phr DPG)

## 2.2 Testing and Analyses

The curing characteristics of the SBR composites were obtained by using a U-CAN DYNATEX INC moving die rheometer (UR-2010), which was used to determine torque, scorch time (Ts2), cure time (T90) and cure rate index (CRI) according to ASTM D5289. There were 4 grams of the respective compound samples tested at the vulcanization temperature of 160°C. The cure time was 4 minutes, and the pressure was 5 kg/cm<sup>2</sup>. The density of composites was measured per ASTM D792 using an electronic densimeter MD-300S (Alfa Mirage, Japan).

The average value of at least three measurements was taken for each sample. The samples' hardness (Shore A) was measured by Durometer Hardness Meter Bareiss HPE II according to the ASTM D2240. After repeated measurements for one sample, the median hardness was recorded.

The swell measurement was carried out according to ASTM D471. The cured specimens with dimensions 50mm x 25mm x 2mm were weighed using an electric balance, followed by immersion of toluene for 24 hours at room temperature (25 °C) in a dark environment. After the conditioning period, the swollen specimens were taken out and weighed again to determine the swelling percentage using Eq. (1). Then, the specimens were dried in an oven at 60 °C until a constant weight was reached and the toluene uptake was determined using Eq. (2). The symbols are W<sub>0</sub> = initial mass before immersion (g), W<sub>s</sub> = mass after the swelling (g), W<sub>ds</sub> = mass after oven-dried until constant weight (g), and W<sub>t</sub> = molecular weight of toluene (92.14 g/mol).

Swelling percentage (%)=
$$\frac{W_s - W_o}{W_o}$$
 (1)

Toluene uptake= 
$$\frac{W_{s}-W_{ds}}{W_{o}\times \left(\frac{100}{W_{t}}\right)}$$
(2)

Scanning electron microscopy (SEM) model Hitachi SU8000 was used to identify the morphology of the silicone rubber composites. The morphological inspection on their fracture surface was conducted at 100X and 1000X magnifications at 10kV accelerating voltage.

#### 3.0 RESULTS & DISCUSSIONS

3.1 Cure Characteristics

The cure characteristics of the SBR compounds are presented in Table 2. The table presents rheometer test data for SBR composites with high and low DPG content. It compares six parameters: Scorch Time (Ts2), Cure Time (T90), Minimum Torque (ML), Maximum Torque (MH), Torque Difference (MH - ML), and Cure Rate Index (CRI). It can be observed that SBR with low DPG has a shorter Ts2 and T90 compared to high DPG. It suggested that the curing experienced by SBR at low DPG was faster than that at high DPG, hence increasing the CRI value.

Meanwhile,  $M_L$  and  $M_H$  reflect the viscosity and rigidity of the compounds under shearing [16-17], respectively. The  $M_H$  is higher for

SBR with low DPG, while the M<sub>H</sub> is slightly higher for high DPG. In addition, the M<sub>H</sub> - M<sub>L</sub> is also higher for low DPG. The increased M<sub>H</sub> - M<sub>L</sub> value mostly accepted to be the indication of enhanced crosslink density. This data suggests that the amount of DPG content can significantly influence the cure characteristics of SBR composites. A lower DPG content enhances the curing process, possibly improving physical and mechanical properties [17].

No.	Parameter	SBR (Low DPG)	SBR (High DPG)
1	Scorch time, Ts2 (min, sec)	1.18	1.51
2	Cure time, T90 (min,sec)	2.09	3.02
3	Miniumum torque, ML (dNm)	33.70	31.26
4	Maximum torque, Мн (dNm)	7.84	7.50
5	Torque difference, MH - ML (dNm)	25.86	23.76
6	Cure rate index, CRI (min-1)	109.89	66.23

Table 2: Rheometer Test Data for SBR

#### 3.2 Density

Figure 2 shows the density of SBR composites varies by DPG content and grain orientation. Overall, all samples exhibited densities higher than 1.0 g/cm<sup>3</sup>. The density of styrene-butadiene rubber (SBR) treated with different amounts of DPG showed consistently higher density in the transverse direction (TD) compared to the machine direction (MD) across all samples. This clearly indicates that the arrangement of molecules or components inside the rubber composites significantly influences density. TD-produced samples have a higher compaction level than the MD samples, indirectly reflecting crosslink levels.



#### Figure 2: Density vs DPG content and grain direction

Specifically, SBR with higher DPG concentration exhibited the highest total density, measuring 1.214 g/cm<sup>3</sup> in TD orientation. It indicates a more compact configuration of molecules or components along the transverse direction. In contrast, SBR with lower DPG concentrations showed slightly reduced crosslink densities [18] and agreed with the swell measurement findings. The high DPG concentrations at 1.5 phr contributed to increased crosslink levels, as evidenced by swell measurement tests. The observed density variation between SBR materials implies that the presence of DPG in SBR notably impacts the overall material density.

#### 3.3 Hardness

Figure 3 shows clear patterns in the hardness and elastic modulus of SBR samples with varying amounts of diphenyl guanidine (DPG) and different orientations. SBR samples, regardless of DPG concentration, display slightly reduced hardness in TD compared to MD.



Figure 3: Hardness vs DPG content and grain direction

Among the tested materials, SBR with high DPG concentration demonstrates the highest hardness at 64.48 Shore A, supporting the hypothesis that increased DPG levels lead to greater hardness in SBR. The increment of hardness at high DPG suggests increased SBR composite chain rigidity [19]. Yet, the observed trend in hardness is inconsistent with the M<sub>H</sub>. This highlights the complex relationship between material composition, crosslinking, and mechanical behavior under external stress.

#### 3.4 Swell Measurement

#### 3.4.1 Swelling Percentage

Figure 4 depicts the swell percentage of SBR samples in toluene. All samples were observed to expand more than 100% of their original volume. According to Choy et al.[20], this condition showing the susceptibility of the rubber composites under organic solvent. The swelling is measured by how much the samples expand. As explained by Surya et al. [21], it reflects the openness of the rubber structure, with more open structures experiencing higher swelling due to toluene particles penetrating the rubber.

The graph in Figure 4 clearly shows that the grain orientation highly influences swelling percentage. It reveals that samples fabricated from the compound in the machine direction (MD) exhibit greater swelling than those in the transverse direction (TD). This observation suggests that MD-oriented samples may have a more porous structure, allowing increased toluene ingress. Conversely, TD-oriented samples seemingly have a more enclosed structure, reducing toluene absorption. The SBR compounds fabricated by TD orientation might facilitate the formation of crosslinks in the composites, reflected from the toluene uptake. This emphasizes particle orientation's importance in shaping rubber specimens' structural properties and permeability.



Figure 4: Percentage of swelling % vs DPG content and grain direction

Examining the impact of diphenyl guanidine (DPG) concentration on swelling percentage, the data indicates that samples with higher DPG content show reduced swelling compared to those with lower DPG content. The interpretation suggests that high DPG content may lead to a more confined structure, limiting toluene penetration and decreasing swelling [22]. Conversely, samples with low DPG content and

potentially more open structures exhibit higher toluene absorption. The observed correlation offers valuable insights into how chemical composition influences the permeability and reaction of rubber materials to external solvents like toluene. The swelling percentage indicates the incorporation of toluene into the molecular structure of rubber samples and acts as a surrogate for crosslink density.

#### 3.4.2 Toluene Uptake

Figure 5 shows the toluene uptake for the effect of DPG and grain orientation. The data reveals that samples oriented in the transverse direction (TD) achieve greater crosslink density, leading to decreased toluene absorption, while machine direction (MD)-oriented samples exhibit lower crosslink density, allowing enhanced toluene penetration.



Figure 5: Toluene uptake vs type of materials

Examining the relationship between crosslink density and diphenyl guanidine (DPG) concentration, the data suggests that DPG content had a trivial effect on the toluene uptake, except when combined with MD grain orientation. At this combination, the higher DPG concentration (1.5 phr) results in greater crosslink density than the lower DPG concentration (0.5 phr), consistent with the understanding that DPG functions as a crosslinking agent [23]. However, it introduces a crucial subtlety, emphasizing that while high DPG content contributes to dense crosslinking, it may not always be advantageous for all rubber formulations. The potential compromise is excessive crosslink density may generate rigid and inflexible products with reduced elongation at break. This observation underscores the importance of achieving effective crosslink density for balancing desirable mechanical properties and structural integrity, emphasizing the need for a nuanced approach in rubber formulation.

## 3.5 Scanning Electron Microscopy (SEM)

Figure 6 depicts the SEM images of the SBR composites corresponding to the effect of DPG and grain orientation. SEM analysis distinguishes rubber matrix and fillers, revealing a mix of ductile and brittle failure modes with cavities formed by filler detachment under tensile stress and level of filler dispersions [24].



Figure 6: SEM images of the SBR samples for the effect of grain direction (TD and MD) and DPG content (a, c) low DPG and (b, d) high DPG at 100X magnifications; (a1, b1, c1, d1) at 1000X magnifications

MD samples exhibit larger, uneven fillers and cavities, leading to weaker bonding and lower mechanical properties. Meanwhile TD samples show smaller, uniformly dispersed fillers and cavities, indicating stronger interaction and improved cure characteristics and physical properties [25]. Using a higher DPG concentration in rubber led to larger fillers, making the material rigid and brittle, reducing its strength. A lower DPG concentration (0.5 phr) was suggested as optimal to maintain a balance between crosslinking density and filler dispersion. In all samples, the transverse direction consistently had higher tensile strength than the machine direction, likely because the material's alignment in the transverse direction allows for better stress distribution, emphasizing the importance of material composition and

structural orientation for designing rubber products with desired mechanical performance.

#### **4.0 CONCLUSIONS**

The research compared the processability and physical properties of SBR compounds, focusing on two-roll mills grain direction and diphenyl guanidine (DPG) concentrations. SBR compounds exhibited better curing and physical properties in the transverse direction (TD) compared to the machine direction (MD). The study highlighted that a reduced DPG amount (0.5 phr) in SBR compounds had improved the curing rate and torque difference. The research identified an optimal SBR-based chemical compound with low DPG concentration at transverse orientation with specific properties (CRI: 109.89 min<sup>-1</sup>, M<sub>H</sub>-ML: 25.86 dNm, density: 1.205 g/cm<sup>3</sup>, hardness: 58.1 Shore A, toluene uptake 1.39 % g/mol). These findings highlight the importance of grain orientation and DPG concentration in influencing mechanical offering practical insights for improving characteristics, the compounding process in two-roll mills. The results contribute valuable historical data for the rubber sector, aiding in developing compounds tailored for specific uses.

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## AUTHOR CONTRIBUTIONS

The contribution to the paper is as follows: S.N.R. Fauzi: Methodology, Data Curation, Writing- Original Draft Preparation; N. Mohamad: Conceptualization, Methodology, Writing-Reviewing and Editing; H.E. Ab Maulod: Data Curation; A.R. Jeefferie: Writing-Reviewing; S.T. Chuan: Methodology, Data Curation; Q. Ahsan: Reviewing.

## **CONFLICTS OF INTEREST**

The manuscript has not been published elsewhere and is not under

consideration by other journals. All authors have approved the review, agree with its submission and declare no conflict of interest on the manuscript.

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