Natural rubber composites for solid tyre used for forklift tensile properties and morphological characteristics

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Abstract: The engineering of natural rubber and synthetic rubber into vulcanized solid tyres for forklifts has been carried out to obtain the optimum ratio of formula and process temperature according to the standards of solid tyre commercial forklifts. Composite formulated materials consist of natural rubber, synthetic rubber, carbon black, silica, calcium carbonate, and coal fly ash, while the temperature, time, and other process materials are fixed variables. The results of testing the mechanical properties of forklift solid tyres for formula C are as follows: specific gravity 1.195 g/cm³, hardness 78 Shore A, tensile strength 17.3 Mpa, tear strength 55.2 kN/m, abrasion resistance 160.8 mm³, modulus 300% is 6.6%, and ozone resistance at 50 pphm, 20% strain, 24 h, 40 °C is no cracks.

1. Introduction

The rubber industry began to develop after Goodyear's discovery the use of sulfur to natural rubber. Addition of sulfur in natural rubber matrices is carried out to improve the vulcanization process which forms cross linking by producing better mechanical properties.

Since the discovery of mixing natural rubber compounds with sulfur, many rubber-finished goods were created through the vulcanization process by adding sulfur elements. Likewise with the manufacture of various types of tires, including solid tires for forklifts. Furthermore, to improve the performance of sulfur forming cross linking reactions at the time of vulcanization various types of accelerators have been developed. The accelerator used in this study was zinc diethyl dithiocarbamate (ZDEC) and tetraethyl thiuram disulfide (ZDEC). This type of accelerator is widely used for natural rubber, and synthetics such as IR, SR, SBR, and EPDM.

The related research to the manufacture of solid tires has generally been developed from natural rubber composites, synthetic rubber, fillers and process materials which are one unit when vulcanization is carried out. Composite is a mixture of various types of complementary materials to get products that meet standards.

Solid tires generally consist of several layers with thickness and several different compounds that form a composite structure. Solid forklift tires have been developed from various materials including polyurethane, but these tires are used only in buildings. The advantage of polyurethane solid tires can carry loads with a greater capacity than rubber tires, while the disadvantages of polyurethane solid tires are not resistant to open spaces.

The material used in the manufacture of vulcanized rubber has various purses according to its mechanical properties. The ratio of material, vulcanization process, time and temperature of vulcanization greatly determines the quality of the solid tire forklift produced. Vulcanization process
to improve some superior properties of natural rubber such as tensile strength, elasticity and other mechanical properties [7,8]. The vulcanization time determines the mechanical properties of the products produced. Where the vulcanization time is too long, the rubber will degrade to form a disulfide ring which causes a decrease in the mechanical properties of rubber vulcanization [9].

Natural rubber as a polymer is a renewable material widely used for various industries, especially for tires, seals, gaskets, in the fields of automotive, aerospace, food and pharmaceutical industries and much more. The automotive industry is the largest consumer of rubber products [10]. Synthetic rubber is in accordance with its mechanical properties, is resistant to oil, heat, hydrocarbon solvents, flame retardant, impermeability to gases, volatile substances, resistant to water and steam.

Natural rubber composites, synthetic rubber and carbon black are the main materials used for solid forklift tires in this study. This is due to the fact that natural rubber is a natural elastomer which has superior properties, especially elasticity and its ability to return to its original state when receiving a load and when stretched. Weaknesses of natural rubber are not resistant to oil and heat, susceptible to attack by atmospheric oxygen, and organic solvents [11-14].

To improve the mechanical properties of natural rubber as the main material for solid tire forklifts in this research, composites with synthetic rubber from styrene butadiene rubber, polybutadiene, chloroprene rubber and ethylene propylene diene monomer rubber were carried out. Addition of carbon black to rubber composites to increase modulus, tensile strength, and abrasion [15-20]. The material 6 PPD added to composites as an anti-oxidant to increase the resistance of solid tires to ozone attack and increase service life [21]. In order for all materials to be more easily distributed into natural rubber and synthetic rubber composites during vulcanization castor oil is used as a plasticizer [22], and sulfur as a curative agent.

An important factor in formulating materials to make solid tires is mainly against physical loads and pressures that affect vibration [23]. The effect of vibration on solid tires is related to the ability of natural rubber composites and synthetic rubber to reduce vibration. Tires are composites consisting of various materials and various components that affect vibration [24].

This study aims to study the mechanical properties of natural-synthetic rubber composites, with carbon black as main fillers, also kaolin and calcium carbonate as supporting materials. In addition, this research is to obtain the optimum conditions for making forklift solid tires from several formulas that have been made.

2. Materials and Methods

2.1. Material
The material used for this study consisted of rubber tools of the type SIR-20, styrene butadiene rubber 1505 (SBR-1505), polybutadiene (BR-9000), chloroprene rubber-232 (CR-232), ethylene propylene diene monomer rubber (JR- EPDM 2), zinc oxide cast No 1314-13-2, stearic acid, carbon black N375, kaolin from Belitung island, calcium carbonate, N-Cyclohexyl-Benzothiazole-2-Sulfenamide (CBS) as accelerator, zinc diethyl dithiocarbamate (ZDEC 14324-55-1) as accelerator, tetraethyl thiuram disulfide (TETD 97-77-8), PCTP as a peptizer, silane coupling agent (Si69), 6PPD-4020 as an anti-oxidant, Jatropha oil (MJP) as plasticizer, and sulfur as urative agent.

This study uses a tool consisting of an open mill rubber, analytical balance capacity of 3.200 grams of the brand Mettler Toledo ME3002, platen vulcanizing press, infrared thermometer brand KRISOW-KW08-280, and molding for printing specimens.

2.2. Methods

2.2.1. Rubber Compounding. According to the formula in Table 1, natural rubber masticated by using the open mill until plastic condition, then while continuing to be ground synthetic rubber is added until all the mixture becomes homogeneous. While continuing to be milled, an activator is added, the co-activator until homogeneous and then add the fillers, plasticizers, and anti-degrading accelerator.
While still rolling until all ingredients homogeneous ingredients are added to sulfur as a curative agent.

The mastication process, vulcanization of natural rubber, synthetic rubber in the open mill and the addition of other process materials for each formula (Table 1) were carried out at a temperature of 45°C ± 5°C for 25 minutes. Vulcanized printing of solid tire from composite was carried out at a temperature of 145°C ± 5°C for 20 minutes.

| Material     | Formula a |
|--------------|-----------|
| NR           | 50 55 60 65 |
| SBR          | 20 20 20 20 |
| BR           | 10 15 10 5  |
| CR           | 10 5 5 5   |
| EPDM         | 10 5 5 5   |
| Zinc Oxide   | 4.85 4.85 4.85 4.85 |
| Stearic Acid | 2.15 2.15 2.15 2.15 |
| Carbon Black | 70 68 66 64 |
| Kaolin       | 20 18 16 14 |
| Calcium Carbonate | 10 8 6 4 |
| Fly ash      | 15 12 10 8  |
| Si69         | 3.75 3.75 3.75 3.75 |
| MJP          | 10 10 10 10 |
| CBS          | 1.45 1.45 1.45 1.45 |
| ZDEC         | 0.55 0.55 0.55 0.55 |
| TETD         | 0.25 0.25 0.25 0.25 |
| 6PPD         | 1.75 1.75 1.75 1.75 |
| PCTP         | 0.85 0.85 0.85 0.85 |
| Sulfur       | 2.85 2.85 2.85 2.85 |

Table 1. Formulation of Rubber Compounding

a unit formula in phr

2.2.2. Physical-Mechanical Properties Testing. Testing the mechanical properties of vulcanized solid tire specimens for forklifts included specific gravity with D.297-15 methods, hardness methods D.2240-15, tensile strength methods D.412-16, modulus D.412.16, elongation at break with methods D.412-16, tear strength D.624-00 (Ra2015), Abrasion resistance (DIN) methods D.5963-04 (Ra2015), compression set, 25% defl, 70°C, 22h methods D.395-16e1, and ozone resistance 50 pphm, 20% strain, 24 h, 40°C with methods D.1149-16.

3. Results and Discussion
The results of testing the mechanical properties of vulcanized solid tires for forklifts from natural rubber composites, synthetic rubber and other materials as shown in Table 2 below.

| Parameter               | Test Results |
|-------------------------|--------------|
| Specific gravity, g/cm³ | 1.295 1.257 1.195 1.162 |
| Hardness, Shore A       | 83 80 78 78  |
3.1. Specific Gravity

Specific gravity is a parameter related to the level of hardness, elasticity and mechanical properties of vulcanized. Material ratio, uniformity of particle size and distribution of materials throughout the composite section can be illustrated by the value of specific gravity. If the measurement of specific gravity is done randomly in each part of the material, it is found that the results of the test are not uniform, possibly due to the uneven distribution of the material at the vulcanization. This has an effect on the results of measurement of other mechanical properties. The ratio of ingredients added to form vulcanized composite composites forklift tires such as natural rubber, synthetic rubber, carbon black and other materials (Table 1) can determine the difference in vulcanized specific gravity values. The material ratio is directly related to the weight of the material used by the per cubic centimeter.

Natural rubber as the main composite material has specific gravity values between 0.9 g/cm$^3$ - 0.97 g/cm$^3$ [25,26], while the specific gravity value of natural rubber reported 1.12 g/cm$^3$ [26]. Besides synthetic rubber used such as SBR with specific gravity values between 0.94 g/cm$^3$ - 0.95 g/cm$^3$ [25,27], BR specific gravity values 0.90 g/cm$^3$ (JSR Corporation), CR value of specific gravity 1.42 g/cm$^3$ (Minnesota Rubber and Plastics), EPDM with specific gravity values 1.35 g/cm$^3$ (Minnesota Rubber & Plastics), and specific graffiti carbon black 1.8 g/cm$^3$ [25].

The data of specific gravity values from some of the materials mentioned above shows the influence of material ratios, the value of specific gravity materials, and the distribution of materials into rubber composite matrices which greatly influence the value of specific gravity of vulcanized solid tires produced. Test results of specific gravity of solid tire composites with different material ratios (Table 1) which cause differences in the value of specific gravity.

Based on the measurement data for formula A the specific graphite value is higher (1,295 g/cm$^3$) than the other formulas. When viewed from the ratio of ingredients for formula A, carbon black as an active filler, kaolin, calcium carbonate, and fly ash as filler enhancers the volume ratio is higher than formula B, C, and formula D. The ratio of mathematical fillers is multiplied with the value of specific gravity materials; it affects the specific gravity values of vulcanized specimens of solid tires. In addition, carbon black added to natural rubber and synthetic rubber composites with different ratios has an effect on differences in the values of specific gravity [28]. The use of carbon black to the polymer matrix can increase the value of specific gravity and hardness with a certain density level [29].

3.2. Hardness

The hardness test results illustrate the level of Vulcanization hardness of formula A; B; C and formula D in Table 1 that form it. The ratio of material and type of material used determines the difference in the value of vulcanized solid tires, where the material used has a different hardness value. As well as differences in the value of specific gravity, natural rubber as raw material the hardness value is between 40 Shore A - 65 Shore A [26]. The value of natural rubber and synthetic rubber hardness according to the ratio added, contributes significantly to the difference in the value of solid tire Vulcanized hardness.

Data from the test results show that the hardness value for formula A is higher (83 Shore A) (Table 2) compared to the other hardness values of the formula. According to the ratio of ingredients in formula A, the ratio of the filler material is higher than the ratio of formulas B, C, and D (Table 1). Hardness value is directly related to the level of material density, where the level of material density
can be described from the results of specific gravity tests. The material density value is related to the distribution of materials distributed into the matrix of composite natural rubber and synthetic rubber. The distribution of material into the natural rubber composite matrix is related to the activity of castor oil as a plasticizer and Si69 as a coupling agent to soften natural rubber and synthetic rubber composite molecules. Softening the polymer matrix is done so that all materials including reinforcing material (carbon black) and volume enhancer fillers (kaolin, calcium carbonate, and fly ash) can be inserted into the polymer matrix composite.

The increase in hardness value is caused by the performance of zinc oxide as an activator, stearic acid as a co-activator, CBS and ZDEC as an accelerator, and sulfur as a curative agent that forms a three-dimensional network to strengthen the matrix of composite natural rubber and synthetic rubber. At the time of vulcanization, a carbon chain crosslink is formed which is between the matrix bonds of natural rubber and synthetic rubber composites. The crosslinking that is formed causes the bond between the vulcanized structures to become more sturdy. Sulfur as a curative agent forms a crosslink agent or cross bond. While the accelerator material is added to condition cross-linking requirements [20].

3.3. Tensile Strength
Tensile strength is a mechanical property of rubber products that can be used to see whether the products produced meet technical specifications. The material properties, material ratio, vulcanization process and vulcanization time determine the tensile strength value. The filler ratio added has an effect on tensile strength and vulcanization time [30,31]. The ratio of materials used such as natural rubber, synthetic rubber, fillers, activators, co-activators contributes to tensile strength. Long chain of natural rubber composed of isoprene monomers, when stretching will return to its original state. Natural rubber and synthetic rubber naturally have tensile strength values. It has been reported that tensile strength of natural rubber 3000 min Psi and 20 Mpa [26]. In addition, tensile strength SBR 300 kg/cm² [27], CR tensile strength > 100 g/cm³ (Minnesota Rubber and Plastics), EPDM tensile strength 6 MPa (Minnesota Rubber & Plastics), tensile strength vulcanized solid tires will be stronger and stronger.

Test results data shows that for formula A the tensile strength value of 14.2 Mpa is smaller than the tensile strength formula, B, C, and D. According to the formula A, the ratio of filler ratio is greater than the other fillers, while the ratio of natural rubber for formula A is smaller than the ratio of natural rubber for formulas B, C, and D (Table 1). The ratio of filler added and the homogeneity of the material during vulcanization takes place affecting the tensile strength value. Excess fillers such as carbon black can reduce tensile strength values [15].

During the vulcanization process a crosslink precursor reaction occurs that interacts by performing a reaction that binds unsaturated carbon chains from natural rubber and synthetic rubber molecules. The next process is formed polysulfide cross bond. The polysulfide cross bond increases the tensile strength. The number of crosslink bonds formed is directly proportional to the availability of sulfur, accelerator and interaction between rubber particles during vulcanization. Interactions between particles, especially fillers, can increase the strength of vulcanized that is formed [32].

3.4. Tear Strength
The results of the tear strength test data from the four samples had a significant difference (Table 2). The difference in tear strength values is directly related to the material ratio (Table 1), where each ingredient contributes to the value of tear strength. The tear strength test results for all treatments were the lowest at 55.2 kN/m (Formula B), and the highest tear strength value was 57.8 kN/m (Formula D). The difference in tear strength is caused by differences in the ratio of natural rubber, synthetic rubber and filler composites (Table 1). Tear strength is directly related to other mechanical properties such as specific gravity, tensile strength, hardness, and abrasion.

Tear strength is influenced by the bond strength between molecules, cross bonds and the level of molecular density. The results of the study of tear strength as shown in Table 2 follow the ratio of natural rubber, synthetic rubber, and filler ratios (Table 1). The material that forms vulcanized acid
strengthens the binding capacity of vulcanized molecules derived mainly from carbon black as reinforcing fillers. Carbon black as a reinforcing filler can improve the mechanical properties of vulcanisate. The strength of bond between molecules significantly influences the mechanical properties of vulcanized [32].

3.5. Abrasion Resistance
Abrasion resistance testing to determine the loss of vulcanized surface to friction with other objects. Test results in abrasion resistance between 164.3 mm$^3$ to 171.5 mm$^3$ (Table 2). The value of abrasion resistance is influenced by the strength of bonds between molecules, where the bond strength between molecules is inseparable from the formation of three-dimensional tissue that strengthens the vulcanized structure. Zinc oxide and stearic acid together with sulfur and accelerators form bonds that strengthen the three-dimensional network structure [33]. The strength of the three-dimensional network structure formed can strengthen abrasion resistance.

Bonding strength between molecules, occurring due to cross reaction of sulfur with natural rubber material, synthetic rubber during vulcanization and printing. In addition, resistance is also affected by the ratio of natural rubber and synthetic rubber. Synthetic rubber added to natural rubber matrices to improve scrape resistance when in contact or friction occurs with other objects. Fillers that are distributed into the polymer matrix affect abrasion resistance [34].

Carbon black as reinforcement filler material, kaolin, calcium carbonate, and fly ash as volume enhancer fillers with the presence of castor oil as a softener and Si69 as a coupling agent facilitates the insertion of all materials into the matrix of natural rubber and synthetic rubber. The interaction between vulcanized-forming materials is characterized by the homogeneous distribution of all materials throughout the natural rubber and synthetic rubber composite molecules. Materials that are evenly distributed into natural rubber and synthetic rubber composite molecules. Materials that are evenly distributed into natural rubber and synthetic rubber composite molecules can increase the abrasion resistance. Carbon black, calcium carbonate, and silica as fillers can increase resistance abrasion, hardness, and modulus [35].

Abrasion resistance is the ability of all composite forming materials to maintain polymer chain formation against friction resistance. The ability of vulcanized molecules to maintain surface morphology loss will be better. This shows that cross link bonds that form three-dimensional networks are able to survive when there is friction with other objects. Vulcanized rubber will form cross-bonds that complement to each other, which in turn can maintain its surface against friction forces [20].

3.6. Modulus
The results of the modulus 300% test on the vulcanized of solid tires based on four formulas (Table 1) showed that modulus from formula C was 300% higher (6.6%) the formula A, B (Table 2), and Formula D. Modulus values describe the power needed to stretch vulcanization at maximum.

Modulus is related to the bond strength between molecules formed when the sample is stretched in the opposite direction. The bond strength between molecules is influenced by the ratio of materials (Table 1) added to vulcanization. Where at the time of mastication and vulcanization three-dimensional tissue formed that strengthens the composite structure.

The more elastic mechanical properties of natural rubber compared to synthetic rubber have an effect on the vulcanized modulus produced. The added material has a significant effect on the modulus value of 300%, while the volume enhancer (kaolin, calcium carbonate) affects the hardness value. Fillers such as carbon black in rubber compounds aim to improve modulus and other eating properties better [36,37].

The 300% modulus of the four formulas for the ratio of added ingredients (Table 1) has an effect on the modulus of 300%, where the power to stretch vulcanized does not require high energy. It has been reported that fillers from carbon black for rubber compounds can increase the mechanical properties of vulcanized rubber [38].

3.7. Ozone Resistance
Ozone resistance testing is carried out to determine the resistance of vulcanized resistance to ozone attack. Ozone attacks occur when vulcanization is in an open space, where the surface of the vulcanized is fractured. Natural rubber composites, synthetic rubber using anti-ozone (Table 1) are carried out to improve the structure of composite molecules to resist ozone attack. Natural rubber composites, synthetic rubber and other compound forming materials at certain ratios can improve the mechanical properties of vulcanized acid produced [39]. Solid tire vulcanized whose emerge is in an open space without being protected by anti-degrade are susceptible to free radical attack, UV light, oxygen and ozone attack. Anti-ozone in rubber compounds is generally added in relatively small amounts between 1-2 Phr [40].

The ozone attack against vulcanization can be seen visually where there is a fracture on the permanent surface. The results of testing of four samples that were given ozone exposure of 50 pphm, 20% of strains for 24 hours at 40°C for all samples did not experience physical damage such as cracks on the surface. Ozone attacks occur mainly against carbon double bonds in rubber molecules that cause oxidation reactions with chemical elements contained in the air.

4. Conclusion
The development of natural rubber into solid tires for forklifts is carried out with natural rubber composites, synthetic rubber with carbon black, kaolin, calcium carbonate, and fly ash fillers. Natural rubber is used from SIR-20 type, styrene butadiene rubber 1505 synthetic rubber, polybutadiene-9000), chloroprene rubber-232, ethylene propylene diene monomer rubber-JR-2. Compound production is carried out with mastication and vulcanization followed by molding process. The test results show that natural rubber composites with synthetic rubber can produce vulcanized of solid tires for forklifts.

Acknowledgment
Thank you to the Head of the Palembang Institute for Industrial Research and Standardization for providing the research facilities, and Director of PT. Shima Prima Utama who has provided assistance in conducting this research activities.

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