The Effect of Natural Rubber Composite using Monomer Diene Ethylene Propylene on Mechanical Properties in Tubes Collar

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Abstract. Tube collar is a part of quad cane type patient sticks. Its function is to adjust the height of the stick when used. The materials widely used to make tube collars are plastic. This study aims to study the mechanical properties of tube collars made from natural rubber composite materials with ethylene propylene diene monomers. Natural rubber and ethylene propylene diene monomers vary in ratios (70: 30; 80: 20; 90: 10; and 100: 0) phr. The activator, coactivator, softener, filler, accelerator, antioxidant and vulcanizing agent are conditioned. The experiments were carried out through the process of mastication, compounding and vulcanization. Vulcanization is carried out at 135 °C for 8 minutes. Curing characteristic test results with a rheometer at a temperature of 150 °C showed $\Delta S$ maximum of 9.70 kg/cm, maturation time ($t_{90}$) 4.59 min, and scorch time ($t_{92}$) 0.02 min. The results of testing the mechanical properties of vulcanized rubber showed that the difference in the ratio of natural rubber with ethylene propylene diene monomer affects the mechanical properties of vulcanized rubber.

Keywords: ethylene propylene diene monomer, mechanical properties, natural rubber, tube collar, vulcanizates rubber

1. Introduction

The use of natural rubber for various purposes has been widely used. The use of natural rubber for various industrial raw materials began to develop since the discovery of the vulcanization process using sulfur by Charles Goodyear in 1839 from the United States [1-2]. Vulcanization is the change of a polymer from an elastomeric state to a thermoplastic [3]. The development of natural rubber into various types of products cannot be separated from the vulcanization process. Vulcanization is the process of forming cross bonds that form a three-dimensional network of the rubber vulcanized matrix [4-5]. The cross-link formed during the vulcanization process is a cross-link of sulphur-elastomers consisting of C-S-C (monosulfide), CS2-C (disulfide), and C-Sx-C (polysulfide) [6]. The cross-link speed that is formed is influenced by the accelerator used [7]. The cross-linking formed in the rubber vulcanizate matrix affects the mechanical properties of the vulcanizate.

The use of natural rubber as raw material for products that have high elasticity cannot be replaced with synthetic rubber [8]. Natural rubber as raw material for various types of products such as tires, seat covers, medical gloves, stethoscope hoses, drug bottle stoppers, balloons, has its own advantages [9]. The advantages of natural rubber are elastic, high tensile strength, flexibility, and not easily cracked. Besides that, natural rubber has the advantage, when it is compressed it will return to its original state, while synthetic rubber cannot return to its original state like natural rubber [10].

To improve certain products, natural rubber and synthetic rubber need to be made composites. Composite materials can complement each other according to their strengths and weaknesses [11]. Research on the use of natural rubber for various types of medical devices has been carried out widely.
The development of natural rubber into tube collars in this study was carried out by adding synthetic rubber. The addition of synthetic rubber to natural rubber aims to improve the mechanical properties of the resulting product [11]. The synthetic rubber used in this study was ethylene propylene diene monomer/monomer ethylene propylene diene (EPDM).

The development of tube collars from natural rubber composites with EPDM with several considerations based on the results of research that has been conducted by previous researchers. Natural rubber composites with EPDM can increase the hardness of rubber vulcanizate [13]. EPDM is a type of synthetic rubber with the advantages of having low specific gravity, oxidation resistance, resistance to chemicals and weathering [14-19]. EPDM has low-temperature resistance [20]. EPDM has ozone resistance and is resistant to open spaces [21-22]. In addition, EPDM has higher ageing resistance than EPDM which uses carbon black fillers [23].

EPDM is a polymer of saturated material with unsaturated side chains which is widely used for electrical insulation and automotive equipment [24]. According to [25], the mixture of NR and EPDM from the SEM test results increased the physical properties of the two-stage mixture. NR/EPDM composites with silica are suitable for application in the internal insulation of rocket motors [26]. EPDM is more stable when compared to conventional elastomers [27]. Concerning to the results of several studies that have been done above, the development of natural rubber with EPDM into tube collars can improve the mechanical properties of the resulting tube collars.

Tube collar is part of the quad cane type patient stick made of plastic with a level of hardness of 100 shore A or 45 shore D. The plastic tube collar is not flexible and difficult to be installed. It needs to be soaked into hot water first that it is easy to install and does not experience cracks or breaks when attached to the patient’s wand. This is because the tube collar is made of inelastic plastic with a high degree of hardness. The development of tube collars made of natural rubber composites with a modifiable level of elasticity and hardness can cover the weaknesses of plastic tube collars.

The development of tube collars in this study, apart from NR and EPDM composite materials, was also added with multi fillers consisting of carbon black (CB), kaolin, and CaCO₃. Addition of filler to strengthen the tube collar structure and improve its mechanical properties. The vulcanized rubber without the addition of fillers and additives cannot meet the mechanical and thermal properties [28]. The cure characteristics of the rubber compound and the mechanical properties of the vulcanizate were studied in this study to obtain information from each formula. It is hoped that the tube collar resulting from this study can be further developed to be applied to the quad cane type patient stick.

2. Materials and Methods

2.1. Materials

The material used consists of natural rubber (NR) SIR-20 from one of the crumb rubber factories in Palembang City; ethylene propylene diene monomer (EPDM) AT-903 Hardness: 50 – 55 Shore A; zinc oxide (ZnO) HS: 28170010, purity 99%, bulk density 5.606 g/cm³; stearic acid Cas No: 57-11-4, Mf: C₁₇H₃₅O₂, purity C₁₇H₃₅O₂ = 38-45%; carbon black (CB) code N-330, mf SiO₂, case no: 1333-86-4 Purity 99%; kaolin 325 mesh, SiO₂ content 49%-54%. Al₂O₃ content 36%-48%; calcium carbonate (CaCO₃) purity 99%, particle size 2 & μm; m-20 pm-50 & μm; paraffin wax melting point min 52-66 °C; paraffinic oil-60; N-Isopropyl-N’-phenyl-p-phenylenediamine (IPPD), formula molecular: C₁₈H₁₈N₂, molecular weight: 226.31 density at 20 °C 1.180-1.190 g/cm³; N-(1,3-dimethyl-but)-N-Phenyl-P-Phenylenediamine (TMQ) Mf: C₁₈H₁₈N₂ freezing point ≥ 44 °C; additive rubber DPG rubber vulcanizate Mf: C₁₇H₁₃N₃, CAS: 102-06-7 diphenyl guanidine purity 99%; CBS-80 specific gravity 1.20 g/cm³; tetramethyl thiram disulfide (TMTD) molecular formula C₆H₁₂N₂S₄, molecular weight 240.41, specific gravity approx.1.26 g/cm³, moisture (from active TMTD) < 1.0%; and Sulfur (S), content 80.0 ± 1.0%, mf C₁₇H₂₂FN₃O₆.
2.2. Methods

2.2.1. Rubber Compounding

Natural rubber (NR) and ethylene propylene diene monomer (EPDM) from each treatment according to the formulation given in Table 1. NR was first masticated using an open roll mill Type SK-230 (5 minutes). After NR became plastic then EPDM was added (2 minutes). The Zn as an activator and stearic acid as a co-activator were added simultaneously until everything was homogeneous in the NR and EPDM composites (2 minutes). Then the filler (carbon black, kaolin and CaCO₃), paraffin wax, and paraffin oil added sequentially (4 minutes). The antioxidants in the form of IPPD and TMQ added (2 minutes) until homogeneous. The DPG, CBS and TMTD as accelerators added into the mixed (2 minutes). Finally, when all ingredients are homogeneously mixed in the NR matrix and EPDM rubber, then sulfur as a vulcanizing material was added (3 minutes). The total time for making the rubber compound is 20 minutes.

The compound was vulcanized at 135 °C for 8 minutes.

Table 1. The ratio of tube collar vulcanizing material

| Materials     | TC-001* | TC-002* | TC-003* | TC-004* |
|---------------|---------|---------|---------|---------|
| NR            | 70      | 80      | 90      | 100     |
| EPDM          | 30      | 20      | 10      | 0       |
| ZnO           | 5.5     | 5.5     | 5.5     | 5.5     |
| Stearic acid  | 2.5     | 2.5     | 2.5     | 2.5     |
| CB            | 87      | 87      | 87      | 87      |
| Kaolin        | 18      | 18      | 18      | 18      |
| CaCO₃         | 52      | 52      | 52      | 52      |
| Paraffin wax  | 1.25    | 1.25    | 1.25    | 1.25    |
| Paraffinic oil| 1.5     | 1.5     | 1.5     | 1.5     |
| IPPD          | 1.2     | 1.2     | 1.2     | 1.2     |
| TMQ           | 1.3     | 1.3     | 1.3     | 1.3     |
| DPG           | 1.2     | 1.2     | 1.2     | 1.2     |
| CBS           | 1       | 1       | 1       | 1       |
| TMTD          | 1.2     | 1.2     | 1.2     | 1.2     |
| Sulfur        | 7.25    | 7.25    | 7.25    | 7.25    |

*phr = part per hundred of rubber

2.2.2. Test method

The compound was tested for ozone resistance test was carried out according to ASTM D.1149-16. The samples were stretched at 20% strain and kept at 38 °C for 24 hrs before experiencing ozone at concentration 25 pphm (D.1149-16), specific gravity (ASTM D.297-15), hardness testing before and after ageing (ASTM D.2240-15), tensile strength before and after aging (ASTM D.412-16), and compression set, 25% defl, 70 °C, 22 h (ASTM D.395-16e1).

3. Results and Discussion

3.1. Cure Characteristics Composites Compounds of NR with EPDM

The maturation characteristics of the compounds from NR composites with EPDM are shown in Table 2. The test results of the cure characteristics of composite NR and EPDM compounds showed that there was a difference curing characteristic for each treatment. The differences can be caused by the bonds formed in the composite matrix of each treatment which follow the ratio of materials. The curing characteristics of the compound are described by the scorch time (tₛ₂), cure time (tₒ), maximum torque (Sₘₐₓ), delta torque (ΔS) (Sₘₐₓ - Sₘᵟᵢₙ) [29].
Table 2. Cure characteristics of composite NR and EPDM compounds

| Curing Characteristic | Formula  |
|-----------------------|----------|
|                       | TC-01    | TC-02    | TC-03    | TC-04    |
| S* Min, kg-cm         | 59.17    | 135.56   | 68.13    | 68.61    |
| S* Maximum, kg-cm     | 63.60    | 145.26   | 70.86    | 70.92    |
| Δs (Kg/cm)            | 4.43     | 9.70     | 2.73     | 2.31     |
| Opt cure time (t_{90}) min; sec | 4.04 | 4.59 | 3.50 | 3.44 |
| Scorch time (t_{s2}), min; sec | 0.40 | 0.02 | 0.06 | 0.06 |

Note: = Test Method PPK, Using MDR 2000 (Not accredited)

From the data in Table 2, it can be seen that the highest delta torque value (Δs 9.70 Kg/cm) is obtained from the TC-02 formula. According to [30-31], the interaction of the fillers material in the composite matrix can increase the stronger torque. The delta torques value (Δs) describes the number of cross-links formed during vulcanization. This can be interpreted that the ratio of NR/EPDM (80/20) is the optimum ratio for the formation of cross-links in the composite matrix.

The speed and number of cross-links formed are influenced by additives such as ZnO as an activator and stearic acid as a co-activator. ZnO and stearic acid in the vulcanization process accelerate the performance of accelerators (DPG, CBS and TMTD) to form cross-link by sulfur in the rubber composite matrix (NR with EPDM). ZnO is an inorganic pigment with accelerators to accelerate vulcanization [32]. The cross-link formed is also influenced by the ratio of materials and the homogeneity of the materials in the composite matrix.

The number of cross-links formed affects the mechanical properties of rubber vulcanizates such as hardness, tensile strength, elongation at break, tear strength, and compression set. The tube collars that are currently made of plastic have tough mechanical properties (100 Shore A or 45 Shore D) but are easy to break or crack.

This is probably due to the process of making tube collars from plastic materials does not use materials that can form cross-link. The formation of cross-links on tube collars made of plastic material because it has not gone through the vulcanization process. Judging from the compound maturation time of the cure time (t_{90}) in this study, for the TC-04 formula, the cure time (t_{90}) is the shortest (3.44 min; sec) than the other formulas, while for the scorch time (t_{s2}) the fastest (0.02 minutes; seconds) of the TC-02 formula.

The number and type of accelerators determine the optimum time (t_{90}) [33]. The scorch time describes the ripening time of the compound, the shorter the scorch time (t_{s2}), the better the vulcanizate results. Scorch time is influenced by the number and interaction of accelerators (DPG, CBS and TMTD) with NR and EPDM composites. Where DPG is a moderate accelerator, CBS accelerator is fast-delayed, and TMTD accelerator is very fast. The combination of accelerators that complement each other, affects the rate of formation of cross-links.

3.2. Physical Properties

3.2.1. Ozone resistance

Polymer materials are generally susceptible to oxidative degradation, especially in direct contact with surrounding air. The rate of oxidation in polymers is influenced by the type of material that forms it, the way it is made and its placement. Rubber vulcanizates that undergo oxidation affect mechanical properties such as flexibility, specific gravity, hardness, tensile strength, tear strength, abrasion resistance, and compression set. The service life of rubber vulcanizates is determined by their resistance to oxidation reactions resulting from ozone attack from the air.

The occurrence of oxidation reactions is due to the formation of peroxyl free radicals from friction, especially between rubber molecules, and also by friction with other objects. To increase ozone resistance and inhibit the rate of oxidation reactions in NR vulcanizates and synthetic rubbers, antioxidants need to be added [34].
The addition of antioxidants to the polymer matrix is generally carried out in the compounding process to make it more homogeneous. The adding antioxidants aim to increase the cross-link stability of the polymer from ozone attack [35-36]. The materials used to increase resistance to ozone attack in tube collars in this study consisted of NR composites with EPDM, as well as antioxidants (IPPD and TMQ). TMQ as an antioxidant in NR more resistance from NR with BHT [37]. Paraffin wax and paraffinic oil as softeners, apart from softening the composite material, they also function to protect rubber vulcanizates from ozone attack. Besides being resistant to ozone, EPDM is also resistant to heat and light [38]. EPDM has a high resistance to ozone and heat ageing [39]. EPDM as a material that is compositive with NR has resistance to ozone attack. Test on the ozone resistance for tube collars with NR and EPDM composites using IPPD and TMQ antioxidants results revealed that there were no parts that had cracks for all treatments.

3.2.2. Specific gravity

The measurement of specific gravity is carried out to see the density level of the materials used in composing the tube collar composite matrix. The ratio of materials added affects the specific gravity value of the rubber vulcanizate for the tube collar. The results of the specific gravity test of the four formulas are shown in Figure 1.

![Figure 1. Effect of NR and EPDM ratios on specific gravity](image)

The density level of the material is influenced by the interaction of activator, accelerator, and filler during vulcanization. The interaction of materials during vulcanization causes the formation of cross-links in the composite material. Besides that, the bonding of the material and the level of material density affect the hardness and tensile strength.

The results of the specific gravity test showed that the specific gravity value of the TC-02 formula is higher than the other formulas. This illustrates that the density level of the forming material (Table 1) in the TC-02 formula is denser per unit cubic centimetre than the other formulas. When connected with the results of the compound curing characteristic test results for delta torsion ($\Delta S$) in Table 2 the value is highest (9.70 Kg/cm) than the delta torsion formula of the other formulas. This shows that the cross-linking formed during vulcanization for the TC-02 formula is more than other formulas. The increase in the delta torques value in the TC-02 formula is directly proportional to the increase in the value of specific gravity.

Natural rubber with a ratio of 80 phr and EPDM with a ratio of 20 phr gives the optimum specific gravity value. Changes in the ratio of NR and EPDM will affect changes in the value of specific gravity. As an illustration, NR has a specific gravity of 1.12 g/cm$^3$, and EPDM with a specific gravity value of 1.35 g/cm$^3$ [40]. When the NR and EPDM ratios change, at the same time the specific gravity value...
changes (Table 1). Likewise, the value of compound curing characteristic (Table 2) also changes following changes in the ratio of NR and EPDM.

3.2.3. Hardness

Hardness describes the level of material hardness per unit cubic centimetre. The hardness value of a material is influenced by the mechanical properties and the specific gravity value of each material that forms it. The design of the hardness of a material is related to its use. Composite materials (NR and EPDM) in this study were made for the needs of tube collars as a substitute for tube collars made of plastic. The test results for tube collars made of plastic are as presented in the introduction, the value is 100 shore A or 45 shore D. The test results for the tube collar of the NR and EPDM composites of the four formulas in this study varied in value (Figure 2).

![Figure 2. Effect of NR and EPDM ratio on the hardness](image)

Hardness test data from four samples of tube collar vulcanizate before ageing and after ageing, for TC-02 the value is higher (95 Shore A) than the other formulas. Theoretically, the hardness value is directly proportional to the specific gravity value, where if the specific gravity value increases, the hardness value will also increase. Changes in delta torque ($\Delta_S$) also affect changes in hardness values. The higher the delta torques value, the more cross-links are formed.

The cross-links formed have an effect on the hardness value. The data in Table 2 and Figure 1 showed that for TC-02 an increase in the delta torque was followed by an increase in the value of specific gravity (Figure 1), and followed by an increase in the hardness value before ageing and after ageing (Figure 2). The hardness value of vulcanizates after ageing and before ageing is related to the stabilization of the hydrocarbon chains during compounding and vulcanization [41]. The tube collar hardness value TC-02 (Figure 2) is close to the tube collar value for the quad cane type patient stick from commonly used plastic material with a hardness value of 100 shore A.

3.2.4. Tensile strength

The tensile strength describes the strong bonds between the molecules that form the vulcanizate when they are pulled in opposite directions. The difference in tensile strength values before ageing and after ageing is influenced by the polysulphide and mono sulfide cross-links formed during vulcanization. The number of cross-links that occur during vulcanization causes an increase in tensile strength. In addition, the interaction between rubber, additives, and fillers makes the vulcanizate difficult to stretch [42]. The results of the tensile strength test of the four samples in this study showed that the TC-03 value was higher than the other formulas (Figure 3).

The tensile strength for the TC-01 before ageing and after ageing formulas was the same (10.01 MPa), while for TC-02, TC-03, and TC-04 the after ageing value was higher than that before ageing.
This result is inversely proportional to the statement, the cross-linking decreased after receiving thermal heat after ageing [43]. Some research points out that the tensile strength value after ageing is not always lower than the tensile strength value before ageing.

![Figure 3. Effect of NR and EPDM ratios on tensile strength](image1)

The antioxidants added to the NR and EPDM composites can improve the oxidant resistance during ageing. In addition, EPDM is a type of rubber with high polarity on NR and can increase the mechanical properties after ageing. NR and EPDM composites, associated with their higher polarity are resistant to ageing [44]. The value of the tensile strength at the tube collar greatly determines the resistance to loads when attached to the patient stick. The lower the cross-linking density, the lower the tear strength of a material [6]. The tear strength at the tube collar affect on its strength to withstand the load.

3.2.5. Compression set
Test on compression set is carried out to see the flexibility of the tube collar when receiving a load to hold or return to its original state. The tube collars made from rubber composite materials (NR and EPDM) have different properties with the tube collars made from plastic materials that are available today. Rubber tube collars will return to their original state when under pressure, while plastic tube collars when under load have the potential to suffer physical damage because they are not flexible like natural rubber.

Physical damage can also occur due to stress and chemical oxidative thermal processes [45]. The compression set test results of the rubber vulcanize for the tube collars of 4 samples showed the highest value (69.26%) at TC-02 (Figure 4).

![Figure 4. Effect of NR and EPDM ratio on the compression set](image2)
The test results of the compression set sample showed that TC-01 (60.18%) has a lower value than TC-02 but higher than TC-03 (49.27%) and TC-04 (50.59%). It can be seen that the difference in the ratio of material (NR and EPDM) determines the compression set value, and other mechanical properties (Figure 1, 2 and 3) as well as cure characteristics of the compound (Table 2).

The curing characteristics of the compound (Table 2) relate to the mechanical properties of the rubber vulcanizate as shown in Figures 1 to 4. As described above, when there is a change in the value of minimum torque ($S_{\text{min}}$), maximum torque ($S_{\text{max}}$), delta torques ($\Delta S$, scorch time ($t_{s2}$), opt cure time ($t_{90}$), there is also a change in the rate of cross-link formation which then affects to changes in the compression set value.

4. Conclusion
Natural rubber monomer and ethylene propylene diene together with activators, coactivators, fillers, softeners, accelerators, antioxidants, and sulfur through the compounding and vulcanization process can be made into tube collars. Making tube collars is done through a process of mastication, compounding and vulcanization. In the vulcanization process, a three-dimensional network structure is formed to strengthen the mechanical properties of the resulting tube collar. TC-02 test results for curing characteristics with a rheometer at a temperature of 150 °C $\Delta S$ maximum 9.70 kg/cm, cure time ($t_{90}$) 4.59 minutes, and scorch time ($t_{s2}$) 0.02 minutes. The results of the ozone resistance test for the four ozone resistant formulas showed no cracks, the hardness value of 95 Shore A was close to the tube collar made of plastic, namely 100 shore A. Hardness is a key parameter for bearing loads in use. The resulting tube collars can be used for quad cane type patient stick tube collars.

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