Evaluation of azodicarbonamide (ADC) on density, expansion ratio and closed cell properties of natural rubber foam

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Abstract. Natural rubber foams were made with a one-step foaming process, the effect of density, expansion ratio, and closed-cell properties via microscope were investigated. This research aims to investigate novel half vulcanization and full vulcanization process with three various blowing agents by compression moulding to fabricate natural rubber and carbon black foams. Closed cells of natural rubber used as shoe soles were formed by azodicarbonamide (ADC) and organic blowing agents. The experimental design used three blowing agent of Azodicarbonamide (ADC) A, B C, and control. The data analysis for images was performed in ImageJ software to get more quantitative results. Experimental data were created to perform a full (AF; BF; and CF) and half vulcanization (AH; BH; and CH) process. The different structure model based on digital microscope images was shown. The AH, BH, and CH with half vulcanisation process revealed a higher expansion ratio and cell number value. The microscope showed that rubber foam with the highest expansion ratio and cell number has a more homogenous structure with more cell connection of A and CH, causing a lower density value of 0.6480. In addition, AH and CH with ADC showed higher values than BH with an organic blowing agent, respectively.

Keywords: Cell number, organic blowing agent, rubber foam

1. Introduction
Natural rubber foam is interesting developed material having a micro-cellular structure. It has some advantages by using natural rubber foam material, such as easy weight, porous material and not expensive which is attractive and ergonomics for users [1], for example, solid tyres [2], shoe and protectors [3].

For preparation and characterization of plastic, the foam has been performed [4, 5, 6]. Other studies revealed that processing parameters (foaming temperature, time, and pressure) and formulation parameters (rubber, filler, blowing agent, and curing system) could influence rubber foam morphology and qualities [7, 8].

Several researchers reported that increasing the cure temperature hastened the cure response while decreasing cure time and maximum torque [9, 10]. High temperature could increase the volume and pressure of the gas inside the foam. The influence of cure temperature on foam morphology revealed that increasing foaming temperature could produce many cells with a wide variety of cell sizes and decreased cell thickness and foam density [11, 10, 9, 12]. Furthermore, increasing cure temperature could decrease foam’s mechanical properties, including modulus, elongation at break, tensile strength, and hardness.
The effect of foaming pressure on natural rubber foam reported by Kim [13] that increasing foaming pressure could also increase foam density and mechanical properties, including its tear strength, hardness, modulus, and stiffness. In addition, Ariff et al. (2007) used three classes of natural rubber also to investigate the effect of rubber grade on natural rubber foam qualities. [10]. The highest cure rate and cure density was achieved from epoxidized natural rubber (ENR) rather than Standard Malaysian Rubber (SMR) and Standard Malaysian Rubber Light (SMR-L). ENR showed the smallest cell size and large cell wall thickness compared to other foam materials. The increasing foaming agent could decrease the cross link density, foam density, and ultimate torque [11]. This was also reported for poly (ethylene propylene diene) (EPDM) and natural rubber foams [12, 14].

Two-step compression moulding was used to prepare natural rubber foam [9]. During the procuring process, the compound was partially cured. The pressure was then released instantly, and the curing process was completed simultaneously as the foaming process. Effect of foaming condition and carbon black content on physical, mechanical, and curing behaviour foam were recorded. As a result of the increased carbon black content, the total density of the foam rose. Therefore, mechanical features of natural rubber foam, such as tear strength, hardness, and stiffness, increased in value. It could be because of the higher foam density and carbon black content.

Our research proposes novel half vulcanization and full vulcanization process with three blowing agents by compression moulding to fabricate natural rubber and carbon black foams. These processes could result in density, expansion ratio, and closed-cell properties of natural rubber foam. This also contributed that there are differences for both treatments with a different blowing agent, respectively. Therefore, this is also the first time that natural rubber foam properties have been investigated.

2. Material and methods
Natural rubber (pale crepe) from Indonesia Rubber Research Institute (IRRI) Sembawa was used as a rubber foam material. Other compounding ingredients include zink oxide, stearic acid, ionol, paraffin wax, sulfenamide (CBS), thiuram (TMTD), sulfur, carbon black.

The rubber was compounded in the Research Centre for Rubber Technology laboratory on August 2021 at room temperature using 2 roll milling according to formulation Table 1. First, natural rubber was masticated for 1 minute, and then all materials were mixed, except for the curing agent, which was added slowly when milling continued for about 3 minutes. The experimental design used three blowing agent of Azodicarbonamide (ADC) A, B, C, and control. Then, to obtain more quantitative results, data analysis for images was performed in the ImageJ program.

For curing behaviour of compounds was observed by using a rheometer at a temperature of 160°C according to ASTM D2084. The samples were divided into 2 parts to see inside compounds to study closed-cell properties of natural rubber foam. The closed-cell structure was examined by using Digital Microscope Images.

| Component            | Amount |
|----------------------|--------|
| Natural rubber       | 100    |
| Zink oxide           | 5      |
| Stearic acid         | 2      |
| Ionol                | 2      |
| Paraffin wax         | 3.5    |
| Sulfenamide (CBS)    | 2      |
| Thiuram (TMTD)       | 0.2    |
| Sulfur               | 0.5    |

*Notes : all the components are given in per hundred rubber (phr).
3. Results and discussions

3.1. Closed-cell properties

A digital microscope was used to observe the number of closed-cell in rubber foam. AH, BH and CH performed a higher number of closed-cell (Figure 1). Limiting the available space for producing bubbles allowed the full vulcanization process to inhibit bubble growth and foam expansion. As a result, 50% ADC content provided a large volume of gas during the foaming process, although the mold limitation is greater than in samples with full vulcanization. This constraint prevents the cells from growing, resulting in foams with a higher cell density and smaller cell size. [7]. In the case of a more homogeneous structure consisting of smaller cells performed foam with a higher density/fill ratio.

3.2. Density

Density is calculated from the mass of foam and divided into the volume of the foam sample. In Figure 2, cell density is higher for AF, BF, and CF than AH, BH, and CH. Thus, the full vulcanization process results in high density, leading to small amounts of cells and lower foam expansion (higher foam density). Based on research reported by Vahidifar, 2020 [15], that more homogeneous structure consisting of smaller cells has a higher density foam.

3.3. Expansion ratio

Expansion ratio performed maximum volume of foam cell to create a large cell in thin walls properties. Figure 3 showed that AH, BH and CH perform a higher expansion ratio than AF, BF, and CF. This could indicate that external stress (such as compression or resilience) is imposed and internal gas pressure within the foam cell, causing high cell wall rupture. As a result, a permanent deformation in the specimen occurred. On the other hand, the energy accumulated and released during loading and unloading cycles coincides, preventing any subsequent damage to the cell structure and a drop in gas pressure in the novel foam cells.
(a)
Figure 1. Effect of ADC content on closed-cell properties in the core layer of natural rubber foams: (a) for samples AH, BH, and CH, and (b) for samples AF, BF, CF, and C.
Figure 2. Density value of natural rubber foam (AH, BH, CH, AF, BF, CF, and C).

Figure 3. Expansion ratio of natural rubber foam (AH, BH, CH, AF, BF, CF, and C).

4. Conclusion
According to this study, the approach presented to foam our samples revealed a strategy for forming good rubber foam with significantly improved resilience and hardness while keeping its physical qualities. Rubber foam with the highest expansion ratio and cell number has a more homogenous structure with more cell connection of AH, causing a lower density value of 0.6480. In addition, AH and CH with ADC showed higher values than BH with an organic blowing agent, respectively.

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