

Research Article Experimental Investigation of Optimal Pressure Achieved through Rubber Foam Extension in the Curing Process

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Abstract:

The aim is to determine the optimal expansion pressure of the rubber foam in the mold to determine the curing time that ensures good mechanical properties. In the experiment, the temperature was examined in three levels: 130, 140, and 150°C, for 20 min. Additionally, 8 compounds were examined with either DPT or ADC blowing agents. The experiment results indicated that the compressive stress and absorbed energy showed a similar trend, which was directly changed by both temperature and blowing agent. The appropriate compounds were compound 4 with 12 phr ADC and compound 5 with 3 phr DPT with the optimal pressure of 0.22 bar and 0.17 bar, respectively. Both ensured the high absorbed energy and high compression set. By determining the optimal internal pressure, the energy consumption of the NR foaming process can be calculated. In addition, the energy consumption was significantly changed by the area ratio.

Keywords: Optimal pressure, Rubber foam, Curing process

1. Introduction

Natural rubber foams are special materials consisting of two main components: a dispersed gas phase and an elastomer matrix. This unique combination of materials gives rubber foams a range of advantageous properties that make them essential in various aspects of our daily lives. In some industrial or commercial settings, natural rubber flooring is a resilient and sustainable flooring material made from rubber tree latex. It is durable, shock-absorbing and non-slip. Due to its comfort, noise reduction and environmental friendliness, it is used in various environments including gyms, healthcare facilities, schools, private homes and industrial spaces [1]. To meet the specification, the natural rubber foam (NR foam) is the suitable choice because it has the ability to restore its original shape after being compressed or deformed. This resilience is a valuable characteristic for applications where materials need to endure repetitive loads or impacts without permanent deformation. The elastomer matrix in rubber foams provides flexibility, allowing them to be easily bent, stretched, or molded to fit specific shapes or applications. This makes rubber foams versatile and adaptable for various purposes [2].

The benefits of NR foam have been continuously examined in several studies. Pechuraia et al. [3] studied how foaming temperature (150 and 160 °C) and blowing agent concentration affected natural rubber (NR) foam curing, mechanical, and morphology. It was found that the curing time dropped at 3 phr OBSH and increased thereafter. As foaming temperature rose, cure and scorch times decreased. NR foam with large cells and non-uniform cell distribution was found. Hardness decreased when NR foam OBSH content rose. Compression set increased with OBSH to 6 phr, then decreased. Oliveira-Salmazo et al. [4] investigated elastomeric foams made from natural rubber with a medium relative density (0.3) different cell structures with regard to the shape anisotropy ratio. The foams

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with anisotropy ratios between 0.90 and 2.48 had the same density. However, the NR foam had slightly good mechanical properties. The foam composite was examined. Zhang et al. [5] used the response surface method to determine the optimal porous, sound-absorbing rubber foam composite. It was found that the ADC blowing agent and foaming temperature enable effective production of sound-absorbing materials. Petdee et al. [6] proposed the optimal NR-foam composite finally, an optimal energy absorption was found in the dinitrosopentamethylene tetramine condition with 3 phr and the curing temperature of 160 °C, providing the maximum energy absorption value of 110 J. In addition, numerical simulation was used to predict the rubber vulcanization process. The finite element method has proven to be a powerful tool for analyzing the process and identifying opportunities for improvement in the curing process [7]. Recently, Lin et al. [8] demonstrated the use of natural rubber latex foam residues in carbon nanomaterials with high added value for the design of energy storage devices.

According to the literature reviews, there are no studies on the optimal pressure achieved by the rubber foam expansion for the NR foam vulcanization process. This parameter can monitor and predict the curing time within the mold. Therefore, the aim of this article is to experimentally investigate the optimal pressure achieved by the expansion of the NR foam in the curing process. The curing temperature and blowing agent were examined and discussed. This data serves as a guide for energy saving in the curing process of the NR foam.

2. Material and Method

2.1 Raw Materials

STR 5L serves as the primary raw material for Chana Latex Co., Ltd. Within our manufacturing process, we utilize various additives to enhance rubber properties. Zinc oxide functions as a vulcanization activator, while Stearic acid serves both as an activator and a rubber grinding aid, contributing to the softness of the rubber. Sulfur serves as an essential vulcanizing agent, so ensuring the attainment of the required qualities in rubber. The hardener used for this study is diphenylguanidine (DPG). In order to safeguard light-colored rubber from oxidation processes that occur between the rubber's double bonds and oxygen, the use of Wingstay L as an antioxidant is employed.

In addition to these components, our formulations contain fillers such as kaolin and foaming agents such as Super Cell DPT and NP. The performance of these foaming agents directly affects the quality of rubber-polymer composite products. Key physical properties of the final foam products include cell structure, hardness, expansion ratio, and a decomposition temperature range of 130-165 °C, yielding a discharge rate of 140-165 mm/g, as detailed in Table 1. The sample size of rubber foam layer had a volume of 200 x 200 x 1 mm³ according to the specification of commercially available floor tiles. Moreover, the compound formulas of the NR foam samples were divided into two scenarios, including the four compounds (Compound 1 to Compound 4) with an increase in DPT and other compounds (Compound 5 to Compound 8) with an increase in ADC, the details of both blowing agent increments were listed in Table 1.

Chemical composition	Formulas (*phr)	
STR 5L (Standard Thai Rubber)	100	
ZnO (activator)	3	
Stearic acid (activator)	1	
Wingstay-L (anti-oxidant)	1	
Clay (filler)	30	
CBS (accelerators)	1	
Sulfur (vulcanizing agent)	2.5	
Supercell type DPT (blowing agent)	3/6/9/12	
Supercell type ADC (blowing agent)	3/6/9/12	
Curing temperature condition	130, 140, and 150 °C	
Curing time condition	20 min	

2.2 Manufacturing and Test Method

The manufacturing process begins with the use of 100 parts per hundred (phr) STR 5L rubber sticks. Grind these rubber sticks in a two-roll mixer at a speed of 30 rpm and a roll gap of 2-4 mm. Continue this grinding process for 5

min. After this initial grinding phase, add zinc oxide to the blender and mix it thoroughly with the gum for 1 minute. Next, add 4 phr stearic acid to the mixture and continue mixing for another 1.5 min. The finished mixture should be transferred to a roller and mixed for an extra duration of 2 min. Next, include three portions of Supercell DPT into the mixture by using the roller, ensuring thorough blending for an additional minute. Subsequently, include 1.5 portions of Supercell NP into the amalgamation and continue with the mixing process for a suitable length. Integrate a single portion of Wing Stay L into the grinding and mixing procedure. Incorporate an additional quantity of 50 portions of kaolin into the grinding amalgamation and continue with the mixing process for a cumulative duration of 10 minutes. Simultaneously, add 30 portions of paraphen oil into the mixture.

In order to enhance the optimization of the mixture, it was recommended to include 0.5 parts of dipropylene glycol (DPG) into the solution and continue the mixing process for an additional minute. The rubber mixture should be allowed to cool naturally for 30 min. Once the liquid has reached a cooled temperature, add two pieces of sulfur. Then apply force for a minute and fully integrate the components. To obtain the required properties, it was important to mix the compound sufficiently and then left it inactive at ambient temperature for 24 h. The rubber compound construction or stabilization procedure required the use of 60.5 g of the compound, which corresponded to fifty percent of the capacity of cylindrical molds measuring 4 cm in diameter, 10 cm in height, and 1 cm in depth. The compound was then introduced into a hot-press extrusion device as shown in Fig. 1(a), using a temperature range of 130–150°C as the experimental parameter. The internal pressure in the molding was recorded by the low-performance pressure sensor (FST100-1002) with an accuracy of 0.3%FS as shown in Fig. 1(b). The NR foam mass was introduced into the two-roll mill under vulcanization conditions, resulting in the extrusion of plate samples. The NR foam mass was used to form approximately 50% of the total volume of the mold. For each test, three samples were prepared and the experiment is repeated three times to ensure accuracy and consistency.



(a) small hydraulic press



(b) schematic diagram of pressure measurement

Fig. 1. The hot press extrusion apparatus

This study aimed to examine the physical and mechanical characteristics of the generated NR foam in order to ascertain the optimal performance of various compounds. The examination of the microstructures and porosities of the rubber sponge was conducted using a scanning electron microscope (SEM) provided by Corded Electric Company. The SEM was operated at a voltage of 5V and a magnification of 1600x.

The experiment included conducting a compression set test on cylindrical specimens. This was achieved by subjecting the specimens to a consistent compressive strain of 25% for a duration of 22 hr. The experimental procedure adhered to the guidelines outlined in ASTM D395-03 (Test method B) [9]. Furthermore, the calculation of the compression set % is derived from Eq. (1).

$$Compression \, set = \frac{t_o - t_f}{t_o - t_n} \times 100 \tag{1}$$

The expansion rate is an indicator used to quantify the ratio of the volume of the NR-foam compound before and after vulcanization, relative to the molding volume. The determination of the Foam expansion rate is obtained from Eq. (2) [10]

Expansion rate =
$$\frac{H_f - H_o}{H_o} \times 100$$
 (2)

ASTM 575-91 [11] covers energy received testing. It is crushed into a circle (Cycle) at 10 mm/min using an NRI-TS500-50 global mechanical testing equipment [12]. In cycle mode, the rubber is compressed to 100% shrinkage and stretched for three cycles. It may be discovered by comparing the force-elongation graph area when the item is loaded and when it is not. The hysteresis number for each load depends on its speed, previous loads, and current load. Thus, the energy absorbed is calculated according to Eq. (3). Energy absorbed is hysteresis loss (J), a fitting curve brought curves A and B to find the Equation in excel plot. The integral method is determined under diagram area.

Energy absorbed = $\oint CurveAdx - \oint CurveBdx$

(3)

3. Result and Discussion

In the experimental results, the effect of curing temperature and blowing agent on the expansion rate, compression set and energy absorbed were investigated and discussed. Moreover, the optimal internal pressure was also determined in order to calculate the energy consumption in the NR-foam vulcanization process.

3.1 Effect of curing temperature and blowing agent on the mechanical properties

In the context of tile flooring, it is crucial to consider specific mechanical properties, namely expansion rate, compression set, and energy absorbed, as they directly reflect their practical applicability [6]. The curing temperature had a significant effect on the expansion rate, compression set, and energy absorbed. For the compound with ADC (1–4), the expansion rate at the specific curing temperature was proportional to ADC, while the compound with DPT (5–8) showed the opposite trend, as shown in Fig. 2. NR-foam expansion refers to the process of expanding or increasing the volume of a material made from rubber foam. The expansion of rubber foam can be achieved through both heat and pressurized expansion [13]. It was explained that the behaviour of the vulcanized NR foam with DPT had a larger number of open cells than that of ADC, as shown in Fig. 3. The number of open cells depends on the decomposition temperature of the blowing agent [1, 2, 13]. Therefore, the compounds with DPT, which has a lower decomposition temperature than ADC, have a higher expansion rate than ADC, as shown in Fig. 2(a).

For the compression set, it refers to the ability of foam to recover their original shape and thickness after being subjected to compression for a period of time. Compression set is a measure of a foam's resilience and durability [1, 2]. When the curing temperature increased, the compression set of compounds mixed with ADC steadily decreased. On the other hand, the compression set of compounds mixed with DPT gradually rose, as illustrated in Fig. 2(c). The main reason might be the factors affecting compression set in foam include the foam's material composition, density and cell structure [13]. Fig. 3(b) shows the high-quality foams with an open-cell structure, which are designed to have lower compression set values and are therefore suitable for applications where long-term resilience and durability are important. The experimental result was similar to the previous works [4, 6].

The final indicator was the energy absorbed. It is crucial in flooring applications where safety and protection are paramount. NR-foam with good energy absorbed characteristics can help reduce the risk of injury by absorbing and dissipating impact energy [6]. The curing temperature had a slight effect on the energy absorbed for the compounds with ADC except the curing temperature of 150 °C. For the compounds with DPT, the energy absorbed was directly changed by the curing temperature. The maximum values of absorbed energy occurred at a curing temperature of 150 °C, as illustrated in Fig. 2 (c). The impact energy or force reduction was found to be proportional to the size of the void within the cellular structure of the NR foam, as shown in Fig. 3. This result was consistent with the effect of cell size and cell density on the foam formation process [1, 2]. However, in the NR foaming process, it was difficult to determine the accuracy and precision values of the curing time. In this study, the curing time was set at 20 minutes. It was found that some compounds with lower ADC content may not be sufficiently cured, as shown in Fig. 3(a). Therefore, this study deals with the evaluation of the internal pressure generated by the expansion of the NR foam.



Fig. 2. Mechanical properties of compounds



130 °C

140 °C

(a) ADC

150 °C



Fig. 3. Morphological analysis for different blowing agents

3.2 The Optimal Internal Pressure

In this experiment, the effective compounds of each blowing agent were selected for floor tile application through engineering optimization [6]. Compound 4 with DPT and compound 5 with ADC were used as experimental input in the internal pressure determination test. The internal pressure profile for different blowing agents were shown in Fig. 4. The internal pressure is evaluated based on the steady state curing time. Curing time refers to the time required for the internal pressure to reach a stable or unchanging state.

For the compound 4 (ADC of 12 phr), the curing time was 17 min that was evaluated from the steady-state internal pressure of 0.22 bar. The supported information is shown in Fig. 3(a). The compound with a curing temperature of 150 °C had a more open cell structure, which was larger than that of 130 °C and 140 °C. In addition, the area ratio of the curing temperature of 150 °C was higher than that of 130 °C and 140 °C, as shown in Table 2.

For compound 5 (DPT of 3 phr), the curing times were achieved at all cure temperatures. The curing times at temperatures of 130 $^{\circ}$ C, 140 $^{\circ}$ C and 150 $^{\circ}$ C were 14, 19 and 20 min, respectively. The optimal pressure was 0.17 bar. The reason for describing the phenomenon was the same as for compound 4 (ADC of 12 phr), as shown in Fig. 3(b). In addition, the area ratios of all curing temperature were more than that of the undercure compounds with ADC at curing temperatures of 130 $^{\circ}$ C and 140 $^{\circ}$ C, as shown in Table 2.

In the energy analysis, the energy consumption of the NR foaming process can be calculated by evaluating the optimal internal pressure. In addition, the energy consumption was significantly changed by the area ratio. The area ratio was assumed to reflect the NR foam density [13]. The compound had the lowest energy consumption with a DPT of 3 phr at the curing temperature of 150 °C. The main reason was the higher area ratio. In this study, it was recommended to use the compound with DPT in NR foam flooring. It can provide higher absorbed energy, higher expansion rate and lower compression set (Fig. 2). The suitable temperature was 150 °C. In addition, the internal pressure inside the mold was the important indicator for evaluating the curing time in the NR foaming process.



Fig. 4. Internal pressure profile for different blowing agents

Blowing agent	Curing temperature (°C)	Area ratio	Energy consumption (kWh)
ADC	130	0.121±0.09	-
	140	0.131±0.06	-
	150	0.185 ± 0.07	1.85
DPT	130	0.163±0.09	2.00
	140	0.172 ± 0.08	1.95
	150	0.193±0.05	1.75

Table 2: Area ratio and Energy analysis at steady state

4. Conclusion

The study focuses on the investigation and advancement of the foam rubber layer. Specifically, it examines the impact of blowing agents and curing temperatures on the physical and mechanical characteristics of NR-foam, with a particular emphasis on its used in floor tiles. Furthermore, an investigation was conducted to determine the appropriate formulation of NR-foam that has exceptional physical and mechanical capabilities. The internal pressure inside the mold was evaluated in order to determine the energy consumption in the curing process. The observed outcomes indicated that the choice of blowing agent and the temperature at which curing took place had a notable impact on the physical and mechanical characteristics of NR-foams. ADC compound and DPT compound had optimal

pressures of 0.22 bar and 0.17 bar, respectively. As a result of the excellent physical and mechanical properties, the DPT blowing agent and 150°C curing temperature were recommended as the optimal parameters for the curing process. The compound had the lowest energy consumption with a DPT of 3 phr at a curing temperature of 150°C, which equated to 1.75 kWh. Moreover, the internal pressure profile in both combinations might provide crucial insights into the duration required for foam creation to reach the desired level of curing. The aforementioned results provide significant insights that may inform and shape the future advancements in foam rubber product development.

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Nomenclature

- A tensile force, N
- *B* retraction force, N
- H height, m
- t thickness, m

Subscripts

- f final
- n the spacer bar used
- o original

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