

EFFECTS OF FOAMING PARAMETERS ON PHYSICAL PROPERTIES OF MICROWAVE-VULCANIZED NATURAL RUBBER LATEX FOAMS (IVCST2021)

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Abstract

This work reported the use of microwave vulcanization coupled with Dunlop process to prepared natural rubber latex foam (NRLF). Optimization of microwave heating for curing step was firstly carried out using a commercially available domestic microwave oven. The processing window and optimum condition for making NRLF were obtained. The optimum power and time of the irradiation were found at 600 W and 6 min, respectively. Using the optimum microwave vulcanization, effects of stirring time (2, 4, 6, 8, and 10 min) and speed (650, 950, 1250, 1550, and 1850 rpm) of foaming step on cell structure, bulk density, compressive properties, and hardness of NRLFs were investigated. Generally, the increases in the stirring time and speed in foaming step both gave rise to the volume of the air incorporated into the latex compound. This resulted in the increases in the bulk density and the number of cell but, the decrease in foam cell size. The stirring speed in a range of 950 to 1550 rpm was successfully used for preparing the NRLF. Too low and too high foaming speeds caused unstable latex compounds and failed to produce NRLF. The foam cell structure changed dramatically as the foaming speed increased. The foam mechanical properties, both compressive strength and hardness, were found to be linearly related to the foam bulk density.

Keywords: Compressive strength; Dunlop process; Hardness; Microwave heating; Morphology; Natural rubber latex foam

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Introduction

Natural rubber latex foam (NRLF) products have been used in several applications such as packaging, construction, and home furniture (Oliveira-Salmazo, *et al.*, 2016; Salmazo, *et al.*, 2016; Suksup, *et al.*, 2019; and Sirikulchaikij, *et al.*, 2020). Unique properties of the natural rubber foam are lightweight, good thermal insulation, good sound absorption, excellent elasticity, and tunable cushioning performance (Suksup, *et al.*, 2019).

Two main methods used in the production of NRLF are Dunlop process and Talalay process. The Dunlop process is more widely used than the Talalay process in the NRLF production. This is because the Dunlop process is a simpler method with a lower production cost as compared to those of the Talalay process (Sirikulchaikij, *et al.*, 2020). The two methods hold the same basic common steps which are 1). air incorporation and 2). vulcanization.

In Dunlop process, the air incorporation step is done by mechanically stir the air into the latex compound. The compound usually contains, but not limit to, foam stabilizing agent, gelling agents, vulcanizing agent, activators, accelerators. The foam structure is generated during this step before vulcanization with heat. The foam structure and foam density of NRLF depend on several parameters of the air incorporation step (Hui Mei, *et al.*, 2010) including the amount of the compound ingredients (surfactant, gelling agent, etc.) (Suethao *et al.*, 2021) and stirring time and speed of the machine used to incorporate the air into the latex compound.

Vulcanization is the step to transform the aerated latex compound into a solidified rubber foam. Typically, the compound may be heated at 120°C for 90 min using a conventional hot air oven or a steam oven (Sirikulchaikij, *et al.*, 2020). However, in the recent years, microwave heating has been slowly replacing a conventional heating technique. Microwave heating possesses several significant advantages over the conventional heating method including better uniformity of generated heat, faster heating rate, lower energy consumption, lower heating time, and overall lower processing cost. (Ahmad Zauzi, *et al.*, 2019).

Microwave heating has been used in many steps in rubber processing such as in drying, vulcanization, and de-vulcanization. Microwave irradiation heating mechanism greatly differs from conventional heating in that it generates heat from inside to outside (Ariff, *et al.*, 2020). The use of microwave-assisted vulcanization in the making of NRLF is limited and has not been widely studied. The change in heating mechanism when use

microwave irradiation may affect the delicate balance of the latex foam before it is fully set. A systematic study is needed in order to understand the relationships of processing parameters in each step and the foam structure as well as its properties.

The objectives of this work were, firstly to determine the microwave irradiation processing window and secondly to investigate the effects of stirring time and speed in foaming step on bulk density, cell structure, and mechanical properties. This study was expected to provide the improved environmentally friendly process for fabricating NRLF by combining Dunlop process and microwave heating technique.

Materials and Methods

Materials

Chemicals and their contents used in the formulation of natural rubber latex foam compounds are listed in Table 1. Natural rubber latex (60% high ammonium latex, HA latex) and chemicals including potassium oleate (K-oleate; surfactant), sulfur (vulcanizing agent), zinc diethylpyrocarbonate (ZDEC; primary accelerator), zinc 2-mercaptobenzothiazone (ZMBT; secondary accelerator), Wingstay L (antioxidant), zinc oxide (ZnO; activator), sodium silicofluoride (SSF; primary gelling agent), and diphenyl guanidine (DPG; secondary gelling agent) were supplied by Chemical and Materials Co., Ltd.

Preparation of natural rubber latex foam

The preparation of NRLF can be divided into 5 steps (ammonia reduction, foaming, gelling, vulcanizing, and drying process). Natural rubber latex (60% high ammonium latex) was stirred by a mechanical stirrer at a speed of 300 rpm for 30 min to reduce the ammonia content in the latex. Chemical ingredients for foaming process i.e., K-oleate, ZDEC, ZMBT, and Wingstay L were incorporated into the reduced ammonia latex. In this

Table 1. Formulation of NRLF compound

Ingredients	Content (phr)
60% HA Latex	100
10% K-oleate	0.15
50% Sulfur	2
50% ZDEC	2
50% ZDMT	2
50% Wingstay L	2
50% ZnO	5
33% DPG	1.4
12.5% SSF	0.25

foaming step, stirring times were varied from 2, 4, 6, 8, and 10 min at a constant stirring speed of 1250 rpm. Then ZnO, DPG, and SSF were added into the NRL compound at a speed of 700 rpm for 1 min. After that, NRL compound was gelled by SSF. The gelled foam was poured into a silicone mold (5.5 cm × 5.5 cm × 2.5 cm) at room temperature and rest for 1 h. The foamed compound then was vulcanized using a commercially available domestic microwave oven (Samsung, MS23K3513AW) at a constant power of 600 W for 6 min. Finally, the NRLF was washed and dried in a hot air oven at 70°C for 24 h.

Similar process was used to study the effects of stirring speed of the foaming step. The speed was varied from 650, 950, 1250, 1550, and 1850 rpm for a constant foaming time of 8 min. Other parameters were kept constant.

Characterizations

Bulk density: A bulk density or a foam density (ρ_{foam}) determination was carried out by measuring the weight and volume of a sample. The bulk density of natural rubber latex foam can be determined using the following Equation (1)

$$\rho_{foam} = \frac{M}{V} \quad (1)$$

Where: M is mass of specimen (kg)
 V is volume of specimen (m³)

Morphology: Cell morphology of NRLF was observed using a scanning electron microscope (SEM, JEOL/JSM6010LV) with an accelerating voltage of 10 kV. The cross-sectional areas of the samples were prepared using a sharp blade and then sputtered coated with gold for 3 min.

Cell size distribution and number of cell per unit volume: Cell size or cell diameter of NRLFs was obtained from the analysis of SEM micrographs using an image analyzer software ImageJ. SEM micrographs of the cross-sectional area of the NRLF samples were used. The micrographs were taken at the magnification of 50x. The area used for the analysis was at the center of the specimen. The average cell size of NRLF samples was evaluated on a specimen of which at least 100 cells were accounted for the calculation. (Harnnarongchai *et al.*, 2015).

The number of cell per unit volume (N) of NRLF was evaluated by the average cell size data from ImageJ software and calculated using the equation (2) as given below:

$$N = \frac{6}{\pi d^3} \left(\frac{\rho_{rubber}}{\rho_{foam}} - 1 \right) \quad (2)$$

Where, N is number of cells per unit volume,
 d is average cell diameter (cm),
 ρ_{rubber} is the density of the solid rubber (1.09 g/cm³) (Sirikulchaikij, *et al.*, 2020),
 and
 ρ_{foam} is the density of the rubber foam (g/cm³) or the bulk density

Compressive strength

Compressive strength of NRLF determination was carried out according to the ASTM D3574 using a universal testing machine (UTM, INSTRON/5565) with a load cell of 5 kN. A rectangular NRLF specimen used was approximately 50 mm × 50 mm × 25 mm in size. The foam specimen was compressed to 50% of its original thickness at a crosshead speed of 50 mm/min. At least three specimens were tested, and the average values were reported.

Hardness

The hardness of NRLF was determined according to ASTM D2240 using a durometer hardness (Shore OO, X.F). The hardness was measured at the top surface of an NRLF specimen. The area used was 25 mm × 50 mm (width × length). The thickness of the specimen was approximately 25 mm. At least three specimens were tested.

Results and Discussion

In this work, we used the Dunlop process together with microwave-assisted curing for NRLF fabrication. We firstly optimized microwave irradiation conditions (power and time). Then the optimal microwave irradiation condition was used to investigate the effect of the stirring time and speed of the foaming step on the NRLFs' structure and properties.

Microwave irradiation optimization for curing NRLF was performed using different powers (450, 600, and 800 W) and times (4, 6, and 8 mins.). The sample was prepared in a rectangular silicone mold with the size of 5.5 cm × 5.5 cm × 2.5 cm (width × length × thickness). The latex compound was poured into the mold and let rest for 1 h without a cover. Then the filled mold was heated with microwave irradiation. Figure 1 shows the image of a dissected fully cured NRLF sample. It could be seen that the foam possessed fine and uniform cells throughout the sample thickness. A closer look shown in Figure 2 revealed that there was a skin layer which may be characterized as a dense, thin



Figure 1. The appearance of the dissected fully cured NRLF prepared using microwave irradiation power and time of 600 W and 6 mins, respectively



Figure 2. Close-up image of a dissected NRLF.

Table 2. Physical appearance of NRLF at various microwave irradiation conditions

Power (W)	Time (mins.)		
	4	6	8
450	Incomplete cured with gel-like edges		
600	√ Fully cured		
			
800	√ Fully cured	Over cured and degraded	
			

layer on the top of the sample. The skin top layer gave the overall appearance of the foam sample as a dense and smooth block. When the cross-section of the sample was considered, the foam cells appeared to be relatively homogeneous with only the thin layer near the top surface that the cells appeared to be smaller than the rest of the sample. The different microwave irradiation times and power used in the curing step gave the samples with different degrees of curing as shown in Table 2.

From Table 2, it can be seen that at a low microwave irradiation output power of 450 W, the samples were only partially cured even with the irradiation of 8 mins. The uncured portions were runny and gel-like, locating around the edges on the outside of the samples. The cores of the samples were cured. When the irradiation power increased to 600 W the foam was fully cured when microwave irradiated for 6 mins. As the power increased to 800 W, the foam could be fully cured in as little time as 4 mins. However, as the irradiation time increased to 6 and 8 mins, the size of foam cells became noticeably larger at the core and eventually turned brown. These observations of the change in cell size

and color indicated the degradation of the rubber foam samples. The core of the rubber foam became brown first. This was well agreed with the heating characteristic of microwave irradiation that heated from the inside out.

From this result, the processing window for curing of the NRLF could be obtained. The microwave curing conditions may be used as a guideline for fabricating NRLF products. It is important to note that the NRLF products with different sizes, shapes, and volumes may require some tuning to get the best curing result. For this study, the results indicated that the optimum microwave curing condition of a rectangular block NRLF was with the irradiation power of 600 W and time of 6 mins. where the foams with good foam structure and no degradation were obtained. Comparing to the conventional curing technique that use hot air oven at 120°C for 90 mins, the microwave curing can significantly save time and energy as well as greatly increase the production rate.

This work also aimed to investigate the two important parameters in the Dunlop process for

making rubber foam from rubber latex using microwave-assisted vulcanization. The two parameters were stirring time and speed in the foaming step. In this step, air was whipped into the latex creating air-in-liquid colloidal dispersion. The stability of the colloid depends on various factors and is crucial for controlling the foam structure in the final product. The volume of the air incorporated into the latex was expected to affect the foam structure. Longer foaming time may increase the air volume in the foam but can also destabilize the colloid and, consequently, alters the final foam product's properties. The results reported here discussed the effects of stirring time and speed of the foaming step on foam (bulk) density, structure, compressive strength, and hardness.

Effect of stirring time of the foaming step (foaming time)

The effect of foaming time on the properties of NRLFs is summarized in Table 3.

Bulk density

From Table 3, it can be seen that the bulk density decreased as the foaming time increased. This was because the longer whipping time (foaming time) allowed a greater amount of air to be

incorporated into the compound (Hui Mei, *et al.*, 2010 and Singh, 2013). As a result, the foams prepared using longer foaming time expanded to a greater degree/volume while the weight of the samples was relatively constant. The change in foam cell characteristics i.e., foam cell morphology, size, and number were also expected with the change in the foam density.

Morphology

Figure 3 shows SEM micrographs of the NRLFs. The images gave an insight into the detail of the foam structure and how they changed as the foaming time increased. In general, the semi-open cell foam structure was observed in all conditions. The cells of the foam were more or less spherical with different sizes. The cell wall appeared to be interconnected with nonuniform wall thickness. Similar structure was also reported by Sirikulchaikij *et al* (Sirikulchaikij *et al.*, 2020) where the Dunlop process was used to prepare NRLF.

From these SEM images, it could be seen that as the foaming time increased, the cell size of NRLF decreased, at the same time the number of cells increased as a function of foaming time. The analysis of these SEM micrographs with the image analyzer revealed that the average cell diameter

Table 3. Properties of NRLFs prepared using different foaming times and at a constant speed of 1250 rpm

Foaming time (min)	Bulk density (kg/m ³)	Cell size (μm)	Number of cell (×10 ³ cells/cm ³)	Compressive strength (kPa)	Hardness (Shore OO)
2	420.80 ± 4.03	197.68	155.55	263.30 ± 9.60	55.13 ± 0.12
4	397.95 ± 8.55	170.13	223.15	198.66 ± 9.73	53.87 ± 1.45
6	367.43 ± 6.66	166.98	208.71	171.76 ± 9.89	52.07 ± 2.50
8	355.89 ± 2.99	136.71	362.59	156.01 ± 4.12	48.60 ± 2.62
10	345.23 ± 2.20	127.88	423.55	154.69 ± 1.67	47.80 ± 1.83

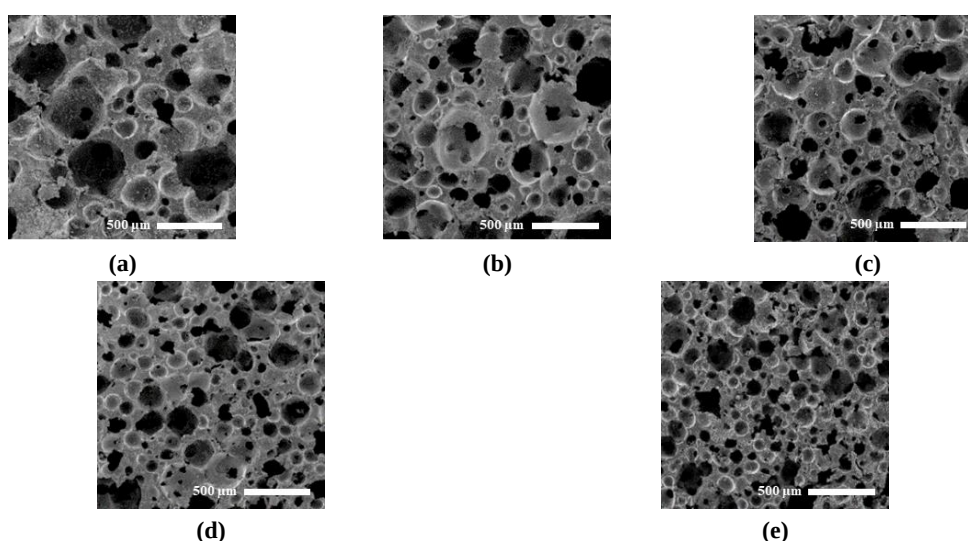


Figure 3. SEM micrographs of NRLFs were prepared using a constant foaming speed of 1250 rpm and different foaming times (a) 2 min, (b) 4 min, (c) 6 min, (d) 8 min, and (e) 10 min

decreased from 197.68 to 127.88 μm or about 35% as the foaming time increased from 2 to 10 mins. The distribution of the cell size in Figure 4 reveals that the cell size of the foam became more uniform as the foaming time increased.

The cell structure of the foam prepared using the Dunlop process was developed during the foaming step. This step involved a mechanically mixing of latex at a high stirring speed. This stirring action helped incorporating the air into the latex. Air bubbles were formed. The amount of air in the latex depended on the stirring time and speed. Longer foaming time allowed more air to be incorporated with the rubber latex and the more mechanical mixing action also generated a greater number of foam cells. It could be hypothesized that the large foam cells were initially formed and then broke down into smaller ones as the mixing action continued. The smaller foam cell also resulted in a shorter cell wall which was less likely to collapse. On the other hand, the greater number of cells meant that the cell wall may become thinner and less stable. In such cases, the bubbles merging could occur (Suethao *et al.*, 2021). Therefore, the cell foam became smaller but being greater in number, the final properties of the foam depended on the balance of these two characteristics.

Compressive strength

Compressive strength at 50% compressive strain of NRLF prepared with different foaming times is also listed in Table 3. The compressive strength of NRLF decreased with increasing

foaming time. The highest compressive strength was 263.30 kPa obtained when the foam was prepared using a foaming time of 2 mins. The compressive strength decreased to 154.69 kPa or approximately 40% lower as the foaming time increased up to 10 mins. This was likely due to the change in the structure of the foams and their density. As previously shown, both higher volume of air (lower in foam density), the greater number of foam cells and thinner cell wall can contribute to the lower of the compressive strength. This finding was also recognized by other researchers. Samsudin *et al.* reported that the NRL foam with higher density usually possessed a greater compressive strength (Samsudin, *et al.*, 2019).

Hardness

The hardness of NRLF is slightly reduced with increasing foaming time. Similar to the compressive strength, a hardness of NRLF was also dependent on density. Pechurai *et al.* also reported a similar finding, where they observed that NR foam with lower density possessed lower hardness (Pechurai *et al.*, 2015).

From this result, it could be concluded that the mechanical properties of NRL foam were strongly governed by the foam density. However, this was not the case for all NRL foam. Other researchers also reported that the mechanical properties of NR foam also depended on its morphology. Sirikulchaikij *et al.* reported that the foam with a smaller cell size gave better mechanical properties (Sirikulchaikij *et al.*, 2019).

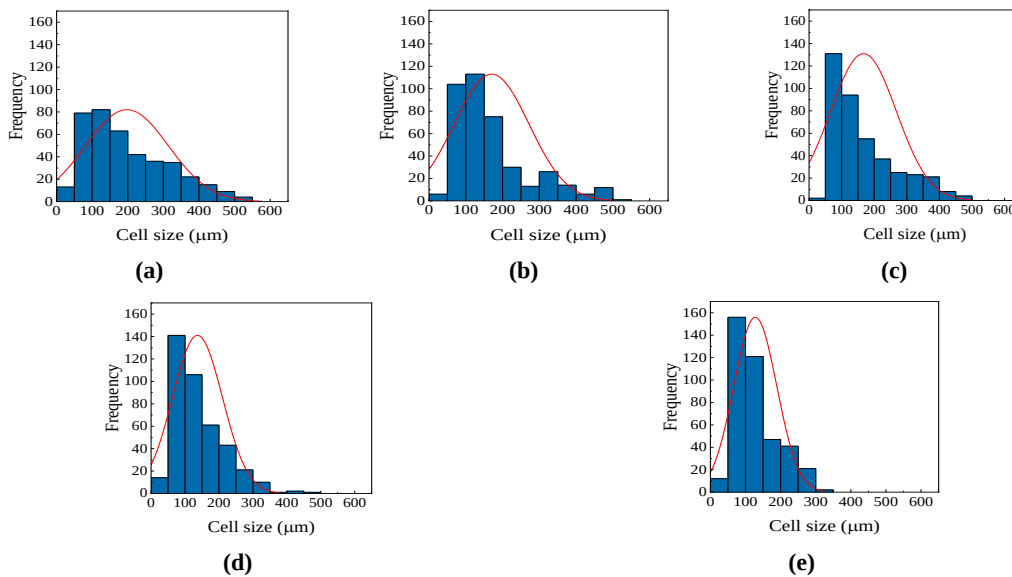


Figure 4. Cell size distribution of NRLF at a foaming speed of 1250 rpm and different foaming times (a) 2 min, (b) 4 min, (c) 6 min, (d) 8 min, and (e) 10 min

Effect of stirring speed of foaming step (foaming speed)

In the Dunlop process, stirring speed during the foaming step is one of the most important parameters to develop the character of a cell foam of NRLF. In this study, five different foaming speeds were used in the NRLF preparation (650-1850 rpm). However, only 3 speeds were used successfully. Too low and too high of the foaming speed created unstable foam bubbles which broke down before the

foam can be settled as shown in Figure 5 (a) and (b). In the case of the low foaming speed of 650 rpm, the air was not enough to create stable foam cells throughout the sample. The top surface of the foam was so dense that it trapped air and moisture inside the sample. When heated, the sample burst due to the internal pressure built up as the water vapor could not escape during vulcanization. On the other hand, the latex compound prepared using a foaming speed of 1850 rpm became a gooey rubber paste after adding SSF in the gelling process (see Figure 5(b)). Too high of the foaming speed probably destabilized the colloidal latex compound. We could establish a processing window for the foaming speed for our system to form these results.

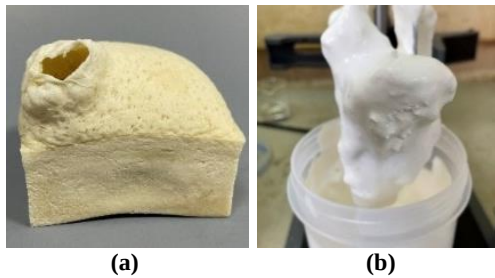


Figure 5. Physical appearance of NRLF during foam production at (a) 650 rpm and (b) 1850 rpm

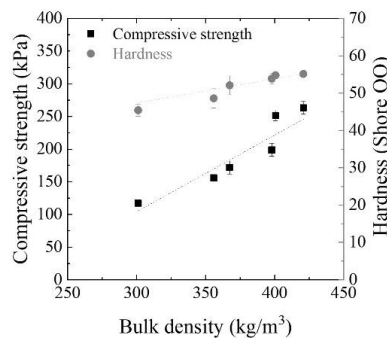


Figure 6. Relationship of bulk density and mechanical properties

Bulk density

Effect of stirring speed in foaming step on bulk density along with other properties are reported in Table 4. It can be seen that higher speed during foaming step caused the lowering in the bulk density of NRLF. The lower in the bulk density could be due to that the higher foaming speed was more efficient in term of aeration. The lower bulk density obtained at the higher foaming speed indicated that a greater volume of the air was incorporated into the latex at the same amount of time.

Morphology

SEM results in Figure 7 revealed that cell size of the foams decreased as the foaming speed increased from 950 to 1250 rpm. Foam cell size measurement (Table 4) showed that the average cell diameter decreased from 201.50 to 136.71 μm or 32% lower. However, the cell structure was noticeably changed as the foaming speed increased to 1550 rpm. The foam cell became rather irregular in shape, larger in size, and less uniform. The cell

Table 4. Properties of NRLFs prepared using different foaming speeds and at a constant time of 8 min

Foaming time (min)	Bulk density (kg/m ³)	Cell size (μm)	Number of cell ($\times 10^3$ cells/cm ³)	Compressive strength (kPa)	Hardness (Shore OO)
950	400.73 $\pm$ 14.00	201.50	135.79	251.16 $\pm$ 7.62	54.80 $\pm$ 0.40
1250	355.89 $\pm$ 2.99	136.71	362.59	156.01 $\pm$ 4.12	48.60 $\pm$ 2.62
1550	301.14 $\pm$ 8.11	181.72	121.56	117.03 $\pm$ 5.08	45.40 $\pm$ 1.60

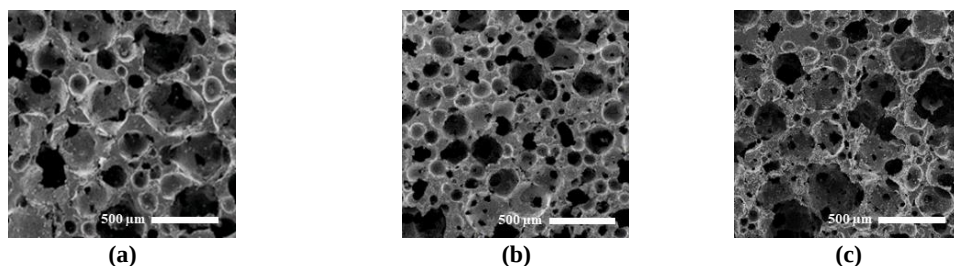


Figure 7. SEM micrographs of NRLF at a foaming time of 8 min and various foaming speeds (a) 950 rpm, (b) 1250 rpm, and (c) 1550 rpm

structure seemed to be ruptured with the presence of smaller cells inside the cell wall itself. It could be speculated that at 1550 rpm, the forming speed was too high which generated an excessive force that ruptured the large bubbles and, at the same time, may cause the merging of the small ones. The wider cell size distribution shown in Figure 8(c) confirmed this speculation. This structure could lead to a lowering in the mechanical properties of this NRLF.

Compressive strength

The compressive strength of NRLFs prepared using different foaming speeds of 950, 1250, and 1550 rpm are presented in Table 4. At a low foaming speed of 950 rpm, the foam possessed the highest compressive strength of 251.16 kPa at 50% compressive strain. As the foaming speeds increased to 1250 and 1550 rpm, the compressive strength of the foams became 38 and 53 % lower than that of the foam prepared at 950 rpm, respectively. The result inferred that the compressive strength of NRLF was mainly determined by the foam density rather than the foam cell size or structure. The compressive strength decreased as a function of foam density.

Hardness

Increasing foaming speed resulted in a reduction of the hardness of NRLF as displayed in Table 4. This was mainly due to the increasing amount of air inside NR with foaming speed, leading to a lowering density of NR foam. In addition, the cell size of the foam dropped with increasing speed.

It was clear that both compressive strength and hardness of the NRLF strongly depended on the bulk density. Figure 6 shows linear relationships between the bulk density to the compressive strength and hardness. The linear relationship was not found between the cell size or the cell number and the mechanical properties. The observed linear relationships may be true only over a narrow range

of the foam density that we could produce using this technique. We speculated that different foaming techniques may result in further cell structure and density. Other relationships of the cell structure, foam density, and properties may be observed.

Conclusions

This study reported the success of the use of microwave irradiation in curing step of NRLF fabrication. The processing window and the optimum condition of microwave curing were obtained. We have illustrated that microwave heating is one of the techniques that should be adopted in the near future as it could save a significant amount of time and energy during the vulcanization process. Additionally, the effect of the two important parameters i.e., time and speed used in the foaming step in the Dunlop process on foam structure and properties were reported. The relationship between the cell characteristics, bulk density, hardness, and compressive strength of NRLF was established. Hardness and compressive strength depended mainly on the bulk density. The final foam product properties and performance strongly depended on the foam structure generated during the foaming step. Understanding the parameters affecting the foam structure and properties could lead to the better control and design the foam product to give desired properties suitable for specific applications.

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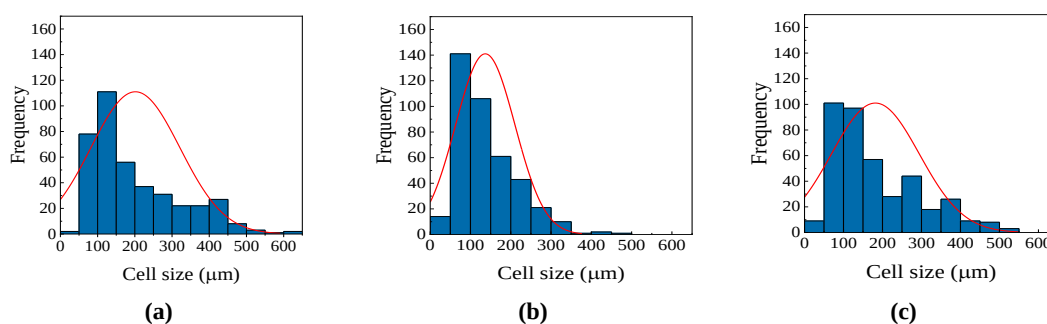


Figure 8. Cell size distribution of NRLF at foaming time 8 min and various foaming speeds (a) 950 rpm, (b) 1250 rpm, and (c) 1550 rpm

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