Research article

Combination Effects of Calcium Carbonate and Crumb Rubber Fillers on the Properties of Natural Rubber Latex Foams

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Abstract

The preparation and mechanical properties of natural rubber latex foam (NRLF) using calcium carbonate ($CaCO_3$) or combinations of $CaCO_3$ and crumb rubber (CRM) as fillers were investigated. The $CaCO_3$ contents were varied as 10, 20, and 30 phr. The ratios of $CaCO_3$/CRM at 0.5/1.0, 0.75/0.75 and 1.0/0.5 were used. The experimental findings were that $CaCO_3$ was well dispersed and blended with the natural rubber latex. The density of the NRLF increased with the increase in $CaCO_3$ content. The foam containing less than 20 phr of $CaCO_3$ showed higher tensile strength and elongation at the break than natural rubber latex foam without $CaCO_3$. The percentage of compression set and depression of NRLF increased with increasing $CaCO_3$ content. When the mixture of $CaCO_3$ and CRM was used as a filler in NRLF, it was found that both $CaCO_3$ and CRM were well dispersed and mixed with natural rubber latex. The NRLF containing the mixture of $CaCO_3$ and CRM was more flexible than the foam containing only $CaCO_3$. Furthermore, the NRLF with $CaCO_3$ and CRM at the ratio of 0.75/0.75 showed more uniform bubble shape and higher density than foam with other $CaCO_3$/CRM ratios. The tensile strength of the foam decreased with decreasing CRM content, while the percentage of depression was low when the proportion of the CRM was reduced. Furthermore, the foam containing $CaCO_3$/CRM ratio of 0.75/0.75 showed the lowest compression set.

Keywords

calcium carbonate;

crumb rubber;

natural rubber;

natural rubber latex foams

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1. Introduction

Natural rubber latex foam (NRLF) is a product made from natural latex with porous characteristics. NRLF can be compressed or bent without losing its shape. It is commonly used to supply cushioning or softness in molded products, such as a cushion, pillow, mattress, and so on. NRLF products are products that can be made without the use of very advanced technology. Their production involves adding value to para-rubber, and can thus support an increase in the production and consumption of para-rubber in the country. Each NRLF product has different properties and prices depending on the purpose of use. In order to obtain the required properties, there are a number of factors involved and must be taken into account such as chemical type, chemical content, manufacturing processes, etc.

Since the products made from NRLF are soft, their applications are limited. Researchers have developed different NRLF formulations that are more rigid and can thus be manufactured into a wider range of products. This work has often involved the addition of fillers, the most widely used of which are silica [1] and calcium carbonate [2-4]. These fillers, in addition to increasing the hardness of the NRLF, are also cost-reducing agents the latter factor being very important for production on the high-capacity industrial scale. However, the use of silica and calcium carbonate is still limited. When used in high quantities, the produced NRLF possesses too much hardness, and the elastic properties or the rubbery properties are reduced. Therefore, to obtain a low-cost NRLF product with a variety of properties as needed, researchers have turned to natural materials. These can be used in combination with industrial fillers to improve NRLF properties.

Currently, there are many researchers interested in the development and improvement of NRLF processes and products, focusing on cost reduction and better product properties. The cost-reducing fillers commonly used in NRLF are both synthetic and natural. In particular, natural fillers have been studied as replacements for synthetic fillers. Natural fillers, in addition to their potential to reduce costs, are also environmentally friendly. Examples of natural fillers used in NRLF include ash [5], bran oil [3], rice husk powder [6], eggshell powder [7], kenaf fiber [8-10], kenaf bark [11], bagasse fiber [12], and oil palm fibers [13]. In addition to using synthetic and natural substances as fillers in NRLF, it was found in 2018 that crumb rubber (CRM) or rubber powder (RP) could be used as a filler to reduce costs and reinforce NRLF [14]. Rattanapan *et al.* [14] reported that the tensile strength, elongation at break, and shrinkage of NRLF increased as the particle size of CRM decreased while the tensile strength and density of the NRLF increased with an increase in CRM loading.

CRM is a product that is obtained from used rubber products or rubber waste, such as car tires and crumb rubber from the production of rubber products. This rubber waste is crushed into powder to obtain the desired CRM particle size [15]. CRM obtained from the tire industry is especially abundant. If crumb rubber could be utilized in large quantities, it could reduce environmental problems. Rubber tires are not biodegradable and many countries do not have systems for the disposal of tires in place. Researchers have therefore studied the use of CRM as a filler including the use of modified molasses carbon as filler in natural rubber [16], or mixing CRM with recycled plastics for roofing materials [17]. CRM was used as a filler in rubber blends of EPDM with epoxidized natural rubber [15]. As a result of the use of CRM as a filler in EPDM/ENR blends, Mooney viscosity, hardness, and compression set of EPDM/ENR blend increased with increasing CRM content while the tensile strength and thermal aging resistance decreased with increasing CRM content [15]. Furthermore, CRM from SBR rubber was mixed with recycled polypropylene [18] and polypropylene.

Based on the aforementioned finding, this research proposes that CRM should be used as a cost-reducing filler in NRLF products as a way of adding value to waste materials. However, the use of CRM only as a filler in the NRLF may affect the quality of the NRLF or the production process. The combination of a commercial filler with CRM as a further filler in NRLF is interesting and is the point of focus of this research. In order to add value to the CRM and develop a prototype material for new products, the effect of calcium carbonate and CRM ratio on the properties of NRLF are investigated and presented in this article.

2. Materials and Methods

2.1 Materials and formulation

$CaCO_3$ and a mixture of $CaCO_3$ and CRM were used as fillers in NRLF. The formulations and materials are shown in Table 1. High ammonia natural rubber latex (LNR) with a dry rubber content of approximately 60 wt%, manufactured by the T.T. Latex&Products Co., Ltd., (Nakhon Sri Thammarat, Thailand), was used as the raw material for the preparation of NRLF. Latex chemicals (sulfur, Wingstay L, potassium oleate, zinc diethyldithiocarbamate (ZDEC), zinc-2-mercaptobenzthiazole (ZMBT), diphenylguanidine (DPG), sodium silicofluoride (SSF), and $CaCO_3$ were purchased from Bayer Thai Co., Ltd. CRM was supplied by Saeng Thai Rubber Co., Ltd. The specific gravity and the average particle size of CRM were 1.15 and 40 mesh, respectively. The commercial grade $CaCO_3$ used was supplied by Bayer Thai Co., Ltd. The specific gravity and particle size of the commercial $CaCO_3$ powder were 2.73 and 45 µm, respectively (data obtained from the technical data sheet of this material).

Table 1. Formulation of natural rubber latex foam filled with $CaCO_3$ and CRM

Ingredient	Formulation (phr)	Formulation (phr)
Natural rubber latex	100	100
K-Oleate	1.0	1.0
Sulphur	5.0	5.0
ZDEC	1.0	1.0
ZMBT	1.0	1.0
Wingstay L	1.0	1.0
ZnO	5.0	5.0
DPG	1.0	1.0
SSF	1.2	1.2
$CaCO_3$	10, 20, 30	-
*$CaCO_3$/CRM	-	10

* $CaCO_3$/CRM (0.5/1.0, 0.75/0.75, and 1/0.5 wt%) by a total combination between $CaCO_3$ and CRM equal to 10 phr

2.2 Sample preparation

Firstly, high ammonia natural rubber latex (LNR) was filtered and weighed according to the formulation. Then, latex and latex chemicals (ZMBT, ZDEC, Sulphur, Wingstay L, and potassium oleate) were stirred for about 4 h using a mechanical stirrer at low speed. After 4 h of continuous stirring, the NRLF compound was foamed using a stand mixer (OTTO Mixer HM-273). Either $CaCO_3$ (0, 10, 20, and 30 phr) or $CaCO_3$/CRM (0.5/1.0, 0.75/0.75, and 1/0.5 wt%) was slowly added to the NRLF compound and the mixture was beaten using a stand mixer for about 4 min until the compound was homogenized. After that, the NRLF compound was beaten intensively for about 2 min until the volume of the compound increased up to three times the initial volume. The foaming speed was lowered to obtain a fine and even foam once the desired volume had been obtained. Next, the primary gelling agent (zinc oxide, ZnO) together with diphenyl guanidine (DPG) was added to the foam, and the beating was continued for another 1 min. Next, the secondary gelling agent (sodium silicofluoride, SSF) was added and the foam was beaten for 1 min. Subsequently, the ungelled foam was quickly poured into the desired aluminum mold and allowed to gel for 3 min at ambient temperature. The gelled foam was then cured in a steam cure at 100°C for 30 min. The $CaCO_3$-filled NRLF and $CaCO_3$/CRM-filled NRLF were stripped from the mold and washed thoroughly with distilled water to remove the excessive non-reacted materials. After washing, the cured NRLF was dried in a hot air oven at 60°C for 24 h. The well-dried foam appeared to be lighter in color.

2.3 Measurement of foams

2.3.1 Physical characteristics

The morphology and the porosity of each NRLF were inspected using a stereomicroscope (Nikon ECLIPSE E200) and SEM micrograph (ZEISS, MERLIN COMPACT). The samples were cut with a blade into 10 mm x 10 mm x 2 mm slices. For the SEM micrograph, the foam surfaces were installed on aluminum stubs and sputter-coated with a thin layer of gold to avoid electrostatic charging and poor image resolution during an examination.

2.3.2 Tensile properties

Tensile tests were carried out according to ASTM D 412 using a universal testing machine, Hounsfield-H10 KS. Dumbbell shaped samples were cut from each NRLF sample using a Pneumatic Shape Cutting Device 403S-S Series. Five samples were required from each loading of $CaCO_3$ and $CaCO_3$/CRM. The tensile test was performed at ambient temperature with a crosshead speed of 500 mm/min. Tensile strength and elongation at break were obtained from tensile testing and the average results were reported.

2.3.3 Density

The density of $CaCO_3$-filled NRLF and $CaCO_3$/CRM-filled NRLF were determined by calculation from the mass and volume of a specimen as shown in equation 1. Samples used in this test method were of regular shape and not less than 1000 mm^3 in volume (as per ASTM D3574). Five samples of each $CaCO_3$ and $CaCO_3$/CRM loading were measured, and an average of three measurements was reported.

$$\text{Density (g/cm}^3\text{)} = (M/V) \tag{1}$$

where M is the mass of the specimen (g) and V is the volume of the specimen (cm)^3.

2.3.4 Compression set

The compression set properties of $CaCO_3$-filled NRLF and $CaCO_3$/CRM-filled NRLF were evaluated according to ASTM D3574. Samples used were of regular shape with dimensions of 50 mm × 50 mm × 25 mm, and had parallel top and bottom surfaces and essentially perpendicular sides. Five specimens per sample were tested. Specimens were placed in the test apparatus and deflected to 50 ± 1% of their original thickness. Within 15 min, deflected specimens and the apparatus were placed in mechanically convected air oven for 22 h with a test temperature of 70 ± 2°C. Specimens were immediately removed from the apparatus and measured after 30 min recovery. Compression set percentage were obtained from this testing. Five samples of each $CaCO_3$ and $CaCO_3$/CRM loading were tested. The compression set, expressed as a percentage of the original thickness, was calculated as shown in equation 2.

$$C_t = [(t_o - t_f)/ t_o] \text{ x } 100 \qquad (2)$$

where: C_t = compression set expressed as a percentage of the original thickness, t_o = original thickness of test specimen, and t_f = final thickness of test specimen.

2.3.5 Depression

Depression is a test to measure the percentage of deflation of the NRLF. Depression, expressed as a percentage of the original height, was calculated as shown in equation 3.

$$\text{Depression (\%)} = ((C - D) / C] \text{ x } 100 \qquad (3)$$

where: C = mold height (mm), and D = average height of the sample test (mm).

2.3.6 Hardness

A Shore C hardness tester (LX-C-1, SHAHE, China) was used to measure the hardness of the sample. Samples used were of regular shape and had dimensions of 50 mm x 50 mm x 25 mm with parallel top and bottom surfaces and essentially perpendicular sides. The testing temperature was 25°C. Five samples were tested for each loading condition.

3. Results and Discussion

3.1 The effect of $CaCO_3$ on properties of NRLF

3.1.1 Physical characteristics, hardness, and densities

From the use of calcium carbonate as a filler in the NRLF, it was found that calcium carbonate was able to disperse well in latex. The NRLF exhibited a smooth surface and no noticeable shrinkage was observed. However, the foam sank slightly in the middle after the vulcanization in a steam oven (see Figure 1a). The cell characteristics of the NRLF can be observed in Figure 1b. It was tested using a microscope at 40 times magnification. It was found that when the calcium carbonate content increased, the image was clearer, indicating that the cells of the NRLFs were more open-celled. Figure 1c shows the NRLFs loaded with 10, 20, and 30 phr $CaCO_3$. From these results, it is obvious that shapes of the cells changed with increased $CaCO_3$ content. When the $CaCO_3$ loading was increased, the $CaCO_3$ began to agglomerate on the cell walls. The NRLF loaded with less $CaCO_3$ showed an almost smooth surface and well-defined cell wall. All NRLFs consisted of spherical and polyhedral cells.

This observation indicates that the foam cells tended to tightly press together with the increase of $CaCO_3$ loading. An increase in $CaCO_3$ content also increased the density of the NRLF as shown in Figure 2a. The effect of $CaCO_3$ on hardness is shown in Figure 2a. The hardness (Shore C) mainly describes the ability of the rubber to resist pressing [19]. Figure 2a shows the hardness of the $CaCO_3$-filled NRLF at different $CaCO_3$ loadings. The addition of $CaCO_3$ to NRLF resulted in increased hardness. The higher $CaCO_3$ load caused the filler to form aggregates, which led to the formation of a coalescent structure resulting in higher stiffness. For this reason, the density of NRLF was also higher with increasing $CaCO_3$ content.

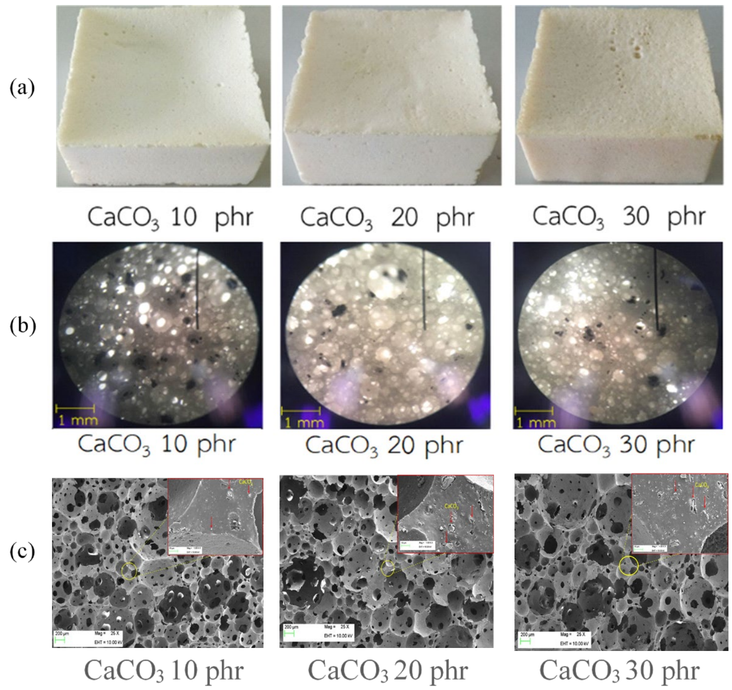


Figure 1. (a) Photographs of $CaCO_3$-filled NRLF, (b) Cell characteristics of $CaCO_3$-filled NRLF using an optical microscope, and (c) SEM micrographs of $CaCO_3$-filled NRLF

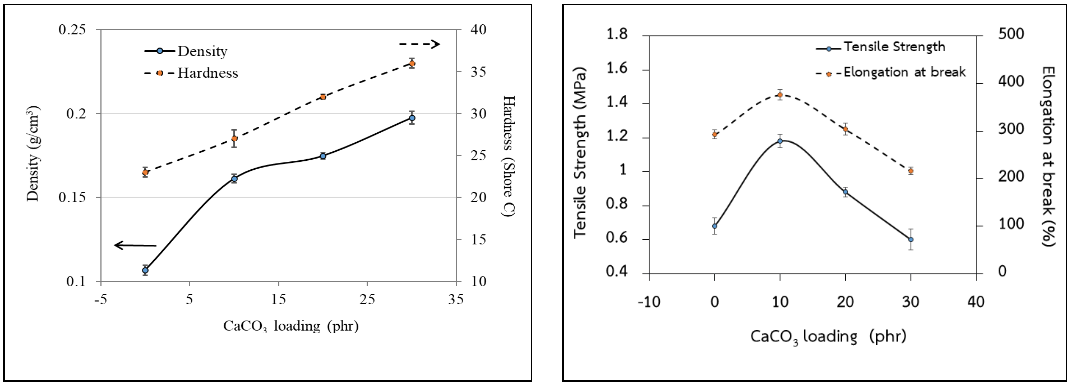
3.1.2 Tensile properties

Figure 2b shows the effect of $CaCO_3$ loading on the tensile strength and elongation at break of NRLFs. The tensile strength and elongation at break of the NRLFs increased once the $CaCO_3$ was introduced into the NRLF at 10 phr of $CaCO_3$, and continuously decreased as the $CaCO_3$ loading increased. However, the use of $CaCO_3$ loading at 20 phr still showed higher tensile strength and elongation at break than NRLF with $CaCO_3$ at 0 phr. This can be understood because as the $CaCO_3$ content increased, the hardness of the NRLF increased (Figure 2a) and its elasticity was reduced [20]. Aggregation is a disadvantage, affecting the tensile strength and elongation at break. The results were similar to those of Putkham *et al*. [21] in which silica aerogel were used as a filler in NRLF.

3.1.3 Compression properties

Figure 2c shows the effect of calcium carbonate content on the percentage of permanent deformation after pressing (% compression set) and the percentage of collapse (% depression). The compression set is a measure of the elastic behavior of the material. The compression set value in Figure 2c shows that the NRLF ($CaCO_3$ 0 phr) had the lowest compression set, indicating that the NRLF had the highest elasticity. Figure 2c shows the compression set value for $CaCO_3$-filled NRLF. As the $CaCO_3$ loading increased, the compression set increased. This was due to the NRLF trapping filler agglomerates, thereby restricting the molecular chain, decreasing elasticity, and increasing stiffness. Regarding the percentage of collapse (% depression), it was found that when the calcium carbonate

(a)



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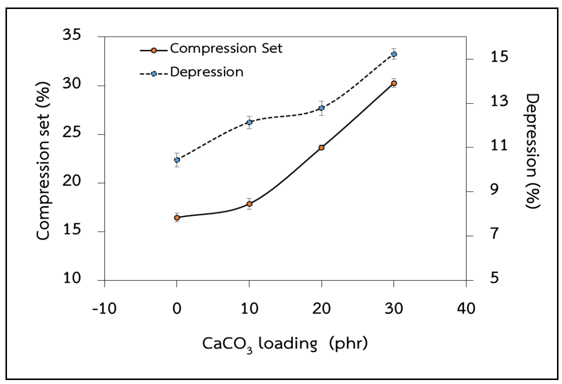


(c)

Figure 2. Properties of $CaCO_3$-filled natural rubber latex foam: (a) Densities and hardness, (b) Tensile strength and Elongation at break, and (c) Compression set and Depression

content increased, the percentage of depression of the NRLF increased. Due to the increased viscosity of the $CaCO_3$ latex, dispersion was more difficult and as a result the of $CaCO_3$ was irregularly dispersed. The hardness of the NRLF increased and its elasticity decreased. The percentage of depression of the NRLF corresponded to the effect of density. With an increase in the $CaCO_3$ content, the greater weight of the filler was further pressed down by gravity, giving rise to a slight percentage of depression of the NRLF before the foam shape was formed. The percentage of depression of NRLF was in the range of 9-15%. From these experimental results, there was no significant difference. The increase of $CaCO_3$ content resulted in a faster setting time of the NRLF. $CaCO_3$ has a lower pH than latex rubber. Adding $CaCO_3$ into the latex rubber during the foaming process decreases the pH of the latex, and also results in a higher latex viscosity. The stabilizer dispersed in the latex surrounds more $CaCO_3$ particles, so less stabilizer is distributed on the surface of the rubber particles. As a result, the NRLF sets faster [2].

As the $CaCO_3$ content is increased, the NRLF can therefore be formed before it collapses. However, the percentage of depression of the NRLF still occurs and tends to increase as the $CaCO_3$ content increases. This is because increasing the amount of $CaCO_3$ results in a reduction of the proportion of rubber or the elastic part of the rubber. As the proportion of rubber decreases or the amount of $CaCO_3$ increases, the NRLF becomes less flexible, which results in the collapse of NRLF.

3.2 The effect of ratio between $CaCO_3$ and CRM on properties of NRLF

The effects of the mixture of $CaCO_3$ and CRM on the NRLF properties were observed. The size of the CRM at 40 mesh provided better and more suitable properties of the NRLF than other sizes of CRM as suggested by Rattanapan *et al*. [14]. Therefore, the CRM size of 40 mesh was selected for this experiment. The content of the mixture of $CaCO_3$ and CRM was 10 phr. The $CaCO_3$/CRM ratios between $CaCO_3$ and CRM were 0.5/1, 0.75/0.75, and 1/0.5 by weight.

3.2.1 Physical characteristics, hardness, and densities

The study of the use of $CaCO_3$/CRM as fillers in NRLF revealed that the foaming of the NRLF obtained from both $CaCO_3$/CRM was able to disperse well and compatible with the natural latex. The foam products formed are shown in Figure 3a. Moreover, it was found that the higher the amount of $CaCO_3$ used, the harder was the latex foam (Figure 2a), It was also observed that, in all case, the foam sank slightly in the middle. When comparing the physical characteristics of $CaCO_3$/CRM-filled NRLF (Figure 3a) and $CaCO_3$-filled NRLF (Figure 1a), it was found that using CRM as a co-filler could prepare a more complete product. Additionally, the resulting NRLF was softer and more flexible than the foam filled with the $CaCO_3$. Figures 3b and 3c show the morphology structure of $CaCO_3$/CRM-filled NRLFs. The foam containing $CaCO_3$/CRM ratio at 0.75/0.75 %wt showed more uniformity than the foams containing other $CaCO_3$/CRM ratios (Figure 3c).

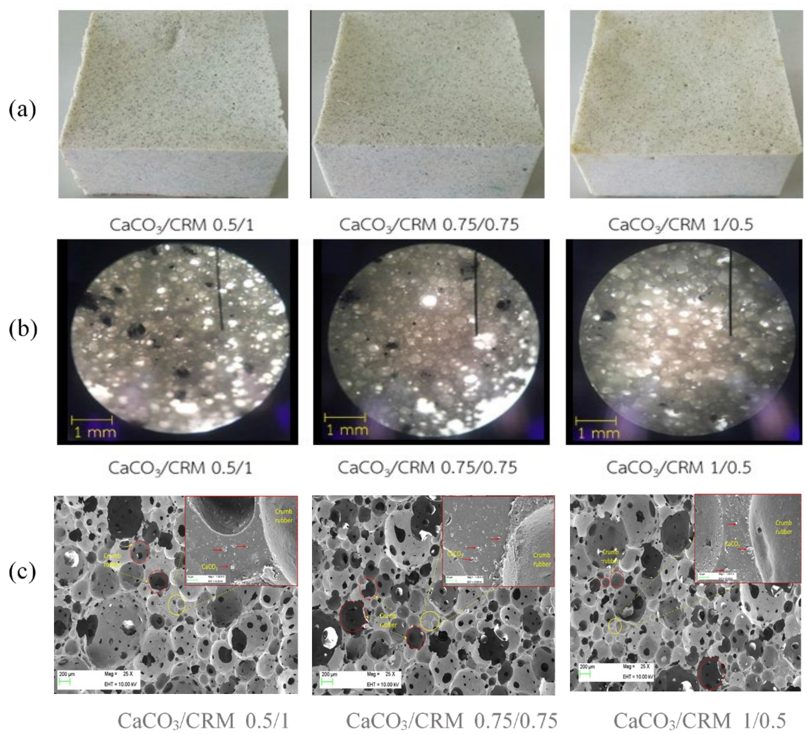


Figure 3. (a) Photographs of $CaCO_3$/CRM-filled NRLF, (b) Cell characteristics of $CaCO_3$/CRM-filled NRLF using a microscope at 40X magnification, and SEM micrographs of $CaCO_3$/CRM-filled NRLF

From SEM micrograph (Figure 3c), it was found that CRM dispersed within the holes or foam bubbles, while $CaCO_3$ is embedded in the cell walls of the foam. It is expected that when only one type of filler was incorporated, it tends to form agglomerates at high loading. As a result, the reinforcement effect would reduce. Therefore, the use of co-fillers helps the CRM and $CaCO_3$ to be better dispersed in the foam. It can be seen in the density and hardness test results shown in Figure 4a that the foam produced from the fillers in the proportion of $CaCO_3$/CRM at 0.75/0.75 had higher density and hardness than foam that incorporated other proportions of the fillers. However, the densities and hardness of the $CaCO_3$/CRM filled-NRLFs, which ranged from 0.1721-0.1818 g/cm^3 (density) and 25-28 shore C (hardness), were slightly different, or showed no significant difference.

3.2.2 Tensile Properties

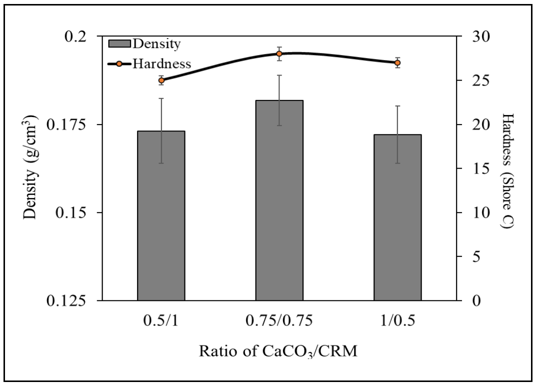
Figure 4b shows the effects of varying the ratio of $CaCO_3$ and CRM on the tensile properties of NRLFs through the range of proportions: 0.5/1, 0.75/0.75, and 1/0.5. It was found that when the crumb ratio was decreased, the tensile properties decreased. This finding was consistent with the results reported by Prasertsri *et al*. [15]. The use of CRM/$CaCO_3$ as a filler in the NRLF gives the resulting foam softer and more elastic characteristics but the particle size of CRM (400 μm) compared to the particle size of the $CaCO_3$ filler (<50 μm) is still considerably larger. CRM may cause defects in the foam, giving its tensile properties and elongation at break values that are lower than those of NRLF with $CaCO_3$ only. This may be explained by the incorporation of the filler, which reduces the mobility of the flexible rubber chains. From the SEM micrograph (Figure 3c), most $CaCO_3$ is embedded and dispersed on the cell walls, while the CRM is scattered within the air bubbles of the foam. Therefore, as the $CaCO_3$ ratio increases, the cell walls contain more $CaCO_3$. This may be responsible for the decrease in the tensile strength of the foam. In any case, the elongation at break values of the $CaCO_3$/CRM filled NRLFs, which ranged from 112-140 %, were only slightly different or showed no significant difference.

3.2.3 Compression properties

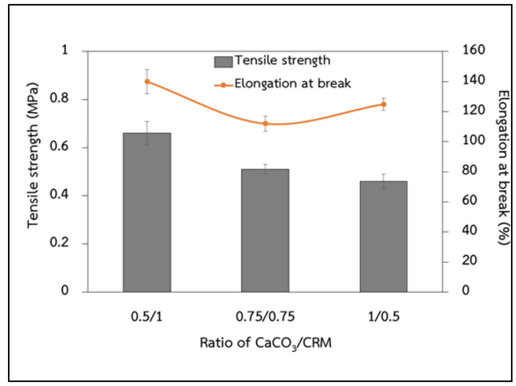
Figure 4c shows the effects of the ratio between $CaCO_3$ and CRM on the percentage of compression set and the percentage of depression of the NRLFs. It was found that the proportion of 0.75/0.75 gave the least percentage of collapse. This is consistent with the results of the experiments depicted in Figures 3b and 3c; the foam of this optimized NRLF is more uniform and the foam bubbles have more open cells than the foams with other proportions, and as a result, the collapse (% depression) of the NRLF is reduced, too. When the CRM is decreased, the percentage of compression set is decreased. It was shown that the use of CRM in a greater proportion than $CaCO_3$ resulted in a decrease in the elastic properties of the NRLF, which was consistent with that found by Prasertsri *et al*. [15].

4. Conclusions

The NRLFs containing either $CaCO_3$ or the mixture of $CaCO_3$ and CRM were successfully prepared. The NRLF showed a smooth surface, no shrinkage, but some collapses were observed. The tensile strength and elongation at break of NRLFs were increased once the $CaCO_3$ was introduced to the NRLF at 10 phr of $CaCO_3$ and continuously decreased as the $CaCO_3$ loading increased. The percentage of compression set and percentage of depression of NRLF increased when $CaCO_3$ content was increased. A mixture of $CaCO_3$ and CRM was well dispersed in the natural rubber latex



(a)



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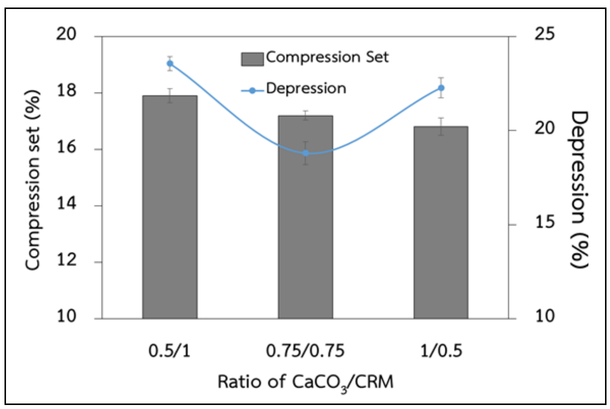

(c)

Figure 4. Properties of $CaCO_3$/CRM-filled NRLF: (a) Densities and hardness, (b) Tensile strength and Elongation at break, and (C) Compression set and Depression

and it could be used as filler in the preparation of NRLF product. The foam containing $CaCO_3$ and CRM with the ratio of 0.75/0.75 showed more uniformity and higher density than the foam containing other $CaCO_3$/CRM ratios. When the proportion of CRM to $CaCO_3$ was decreased, the tensile properties were decreased. The lowest percentage of depression was obtained when the ratio of $CaCO_3$ to CRM was 0.75/0.75. The compression set value was reduced when the CRM ratio was decreased.

5. Acknowledgements

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