

## Morphology and Properties of Agarwood-waste-filled Natural Rubber Latex Foam

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Agarwood (*Aquilaria crassna*) (ACW) waste is widely available as a by-product of agarwood essential oil production. In this study, ACW waste was ball milled into ACW powder (passed through 120 mesh) and used as filler in natural rubber latex foam (NRLF) prepared by the Dunlop method. The effects of the ACW filler on cell morphology and properties of the NRLF were determined. It was found that the ACW filler loading affected cell morphology of the NRLF. The cell size of the ACW-filled NRLF increased with ACW loadings of 1.5 parts per hundred parts of latex (phr) and 2.5 phr, compared with that of control NRLF. A bimodal cell size distribution (with large and small cells) was dominant in the ACW-filled NRLF at loadings of 3.5 phr, 4.5 phr, 5.5 phr, and 6.5 phr. The cell walls also became thicker, causing inferior compression set behavior. In addition, the density and hardness of the ACW-filled NRLF increased with ACW filler loading.

*Keywords:* Rubber foam; Agarwood waste; *Aquilaria crassna*; Natural rubber latex

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### INTRODUCTION

Agarwood is resinous hardwood from trees of the genus *Aquilaria*. Most *Aquilaria* species are found in Southeast Asian countries (Siripatanadilox 2007), such as Malaysia, Thailand, Vietnam, and Indonesia. There are five *Aquilaria* species recorded in Thailand: *A. subintegra*, *A. crassna*, *A. malaccensis*, *A. hirta*, and *A. rugosa* (Eiadthong 2007). *Aquilaria malaccensis* is more widely traded than *A. crassna* in Thailand (Siripatanadilox 2007). However, farmers in eastern Thailand have mostly cultivated *A. crassna*, which, in Trat province, has support for distribution, seedling production, planting, and agarwood industry and business (Jamroenprucksa 2007). Agarwood (*Aquilaria crassna*) (ACW) is among the high-value fragrant woods from which essential oils are extracted. The desired compounds in this essential oil are neopetasane, dihydrokaranone,  $\beta$ -agarofuran, and agarospirol, which contribute to the fragrance (Thuy *et al.* 2019) and are used in perfumes. Additionally, agarwood essential oil has antimicrobial activity against *Staphylococcus aureus* and *Candida albicans* (Wetwitayaklung *et al.* 2009), pharmacological activity, and medical benefits (Dahham *et al.* 2016; Ahmed and Shafiul Islam 2020).

There are various ways to make ACW oil, such as water distillation, subcritical water extraction (Yoswathana *et al.* 2012), and supercritical fluid carbon dioxide extraction (Wetwitayaklung *et al.* 2009). The ACW oil industries in Trat and Chanthaburi provinces of Thailand use conventional aqueous distillation. The production of essential oil from agarwood starts with cutting the agarwood into chips. Subsequently, the chips are dried, milled, and fermented in water for approximately 5 d to 10 d, followed by distillation for 5 d to 10 d (Jamroenprucksa 2007; Jindawech *et al.* 2015; Moungrimuangdee *et al.* 2016).

The extraction of ACW oil produces a lot of solid waste, which is used to make frankincense and incense products. Sauki *et al.* (2013) investigated utilizing the waste from distillation of ACW oil by converting it into a profitable oil well cement additive. Such reuse or recycling of waste reduces pollution and protects the environment. Moreover, products from ACW waste are interesting because of the fragrance. Aromatic products from natural rubber latex (NRL) filled with ACW waste were the focus of this study. The recent sharp decrease in demand for NRL (BOI 2018) has caused a decrease in the prices, and rubber farmers in Thailand have suffered from this.

Natural rubber latex comes from rubber trees (*Hevea brasiliensis*), which have been widely cultivated in southern and eastern Thailand (Kasikranan 2012). Natural rubber latex can be used as a raw material to directly produce some rubber products, such as gloves, condoms, balloons, catheters, baby soothers, dental dams, and foam rubber (Yip and Cacioli 2002). Adding value to ACW waste and to NRL would benefit the environment, rubber farmers, and Thailand overall.

Natural rubber latex in the form of NRL foam (NRLF) is used in porous rubber products, such as pillows, cushions, and mattresses. Bio-based fillers for NRLF have been investigated, including rice husk (Ramasamy *et al.* 2012, 2013a) and kenaf (Abdul Karim *et al.* 2016). The investigations found that rice husk powder filler increased the density, hardness, tensile strength, and filler–matrix interaction in NRLF. In contrast, the kenaf powder filler decreased tensile strength of NRLF. The kenaf-filled NRLF had irregular morphology, and the filler did not bind well with the matrix. These alternative fillers are low-cost, lightweight, less energy intensive, and environmentally friendly. However, ACW waste has not yet been tested as a candidate filler for NRLF, but the eventual development of fragrant products using ACW-filled NRLF is a good motivation. In addition, ACW waste has been tested as filler in a polymer composite membrane, and the mechanical properties of the membrane were improved by the ACW waste filler (Shahrudin and Jasni 2014). Therefore, this preliminary study examined the potential and the filler effects of ACW in NRLF. The morphology and properties of ACW-filled NRLF were investigated.

## EXPERIMENTAL

### Materials

#### *Natural rubber latex*

High ammonia latex concentrate (HA latex) NRL was obtained from Chalong Latex Industry Co., Ltd. (Songkhla, Thailand). It had 60% dry rubber content (DRC), 61.5% total solids content (TSC), and 0.7% ammonia by weight. Sulfur, potassium oleate, poly(dicyclopentadiene-co-*p*-cresol) (Lowinox CPL), zinc diethyldithiocarbamate (ZDEC), zinc 2-mercaptobenzthiozole (ZMBT), zinc oxide (ZnO), diphenyl guanidine (DPG), and sodium silicofluoride (SSF) used in this study were supplied by Sunny World Chemicals Co., Ltd. (Bangkok, Thailand).

#### *ACW waste*

The ACW waste was received from the Agro-Production Community Enterprise Community of Trat Province, Thailand. The ACW waste was a by-product from ACW oil extraction by aqueous distillation. The chemical composition of the as-received ACW waste was examined by X-ray fluorescence spectrometry (XRF, PW2400, Philips, Amsterdam, The Netherlands)

A 10% ACW filler dispersion was then prepared by adding bentonite (5 g), vultamol (5 g), and distilled water (440 mL) into 50 g of as-received ACW waste. Subsequently, the mixture was ball milled for 72 h and sieved through 120 mesh.

### Foam Sample Preparation

The formulation and materials used in this study are shown in Table 1. First, the HA-type NRL was filtered, measured, and stirred using a mechanical stirrer for approximately 30 min. Next, potassium oleate soap, vulcanizing agent (sulfur), and ZMBT together with ZDEC and antioxidant (CPL) were added and stirred at 120 rpm, 240 rpm, 360 rpm, and 120 rpm for approximately 1.5 min, 1.5 min, 1.5 min, and 1.5 min, respectively. Then, the 10% dispersion of ACW waste powder was added and stirred at 120 rpm for approximately 2 min.

Next, DPG, together with zinc oxide (ZnO), was added as the primary gelling agent to the foam, and beating was continued for another 90 s by stirring at 240 rpm. Immediately after, SSF (the secondary gelling agent) was added, and the foam was beaten for another 30 s by stirring at 240 rpm.

Finally, the un-gelled foam was quickly poured into an aluminum mold and allowed to gel for 7 min at ambient temperature. The gelled foam was then vulcanized by steam at 100 °C for 1 h. Once the foam was cured, it was stripped from the mold and washed thoroughly with de-ionized water to remove potassium oleate soap and excess unreacted chemicals.

After washing, the cured NRLF was dried in a hot-air oven at 70 °C for 5 h. The well-dried foam was off-white. The same procedure was used to produce the control sample of NRLF without ACW waste powder filler.

The following steps were the same as in the preparation of the NRLF control sample without ACW waste powder loading. The proportions (in parts per hundred parts of latex (phr)) of all the chemicals are shown in Table 1.

**Table 1.** Formulation of NRLF Filled with ACW Powder

Ingredient	Total Solids Content (%)	Formulation (phr) <sup>1</sup>
NRL (HA latex) <sup>2</sup>	60	100
Sulfur	50	2.5
Zinc oxide	50	5
Lowinox CPL <sup>3</sup>	50	1
ZDEC <sup>4</sup>	50	1
ZMBT <sup>5</sup>	50	1
DPG <sup>6</sup>	33	0.83
SSF <sup>7</sup>	20	0.12
Potassium oleate	10	0.5
ACW <sup>8</sup>	10	0, 1.5, 2.5, 3.5, 4.5, 5.5, 6.5

<sup>1</sup> Amounts are given as parts per hundred parts of latex (phr).

<sup>2</sup> Natural rubber latex

<sup>3</sup> Poly(dicyclopentadiene-co-p-cresol)

<sup>4</sup> Zinc diethyldithiocarbamate

<sup>5</sup> Zinc 2-mercaptobenzthiozole

<sup>6</sup> Diphenylguanidine

<sup>7</sup> Sodium silicofluoride

<sup>8</sup> Agarwood (*Aquilaria crassna*) (ACW) waste

The other latex chemicals (*i.e.*, Lowinox CPL, ZMBT, ZDEC, DPG, and SSF) were manufactured by Zarm Scientific and Supplies Sdn. Bhd., Malaysia.

## Visual Inspection of Foaming and Gelling Stages

Bubble formation was inspected visually, and the gel time for each experimental condition was measured. Macroscopic foam surfaces after vulcanization were photographed with a cell phone camera using 4× magnification.

## Morphology Characterizations of ACW-filled NRLF Samples

Scanning electron microscopy (SEM) was used to study the surface morphology of the ACW-powder-filled NRLF samples. First, the ACW-powder-filled NRLF samples were sputter-coated with a thin layer of gold to avoid electrostatic charging during imaging. Then, the coated foam samples were mounted on aluminum stubs. From the SEM micrographs, the rubber-filler interactions, the ACW dispersion, and the pore morphology of the foams were assessed.

## Properties of ACW-filled NRLF Samples

The densities of the ACW-filled NRLF samples were measured by the displacement method in accordance with ASTM D1056 (2014).

The hardness of an ACW-filled NRLF sample was measured using a Shore durometer (LX-AO) (DUNDOO, LX Series Analog Shore Durometer, China). Five points for each ACW loading were measured, and the average results were reported.

In the compression set test, the ACW-filled NRLF samples were compressed to 50% of their original thickness ( $t_0$ ) at  $70\text{ °C} \pm 1\text{ °C}$  for 22 h ( $t_s$  is the thickness at 50%). Next, the compression was released, and the test sample was allowed to rest for 30 min at room temperature before the final thickness ( $t_f$ ) was measured. The compression set percentage ( $C_d$ ) was determined as follows (Eq. 1):

$$C_d = (t_0 - t_f) / (t_0 - t_s) \times 100\% \quad (1)$$

## Swelling of ACW-filled NRLF Samples

Swelling testing was used to assess rubber-filler interactions as in a prior study (Muniandy *et al.* 2012). The ACW-filled NRLF samples with dimensions of 30 mm × 5 mm × 2 mm were accurately weighed, immersed in toluene, and allowed to swell in a closed bottle for 72 h at room temperature (25 °C). When the sample was removed, its surface was quickly wiped dry, and the sample was weighed. It was then dried in an oven at 70 °C for 15 min and weighed again. The weight of toluene uptake per gram of rubber hydrocarbon ( $Q$ ), was determined according to Eq. 2.

$$Q = \frac{\text{Swollen weight} - \text{Dried weight}}{\text{Original weight} \times (100 / \text{Formula weight})} \quad (2)$$

The rubber-filler interactions were estimated using the Lorenz and Park equation (Eq. 3):

$$\frac{Q_f}{Q_g} = ae^{-Z} + b \quad (3)$$

where the subscripts  $f$  and  $g$  refer to filled and gum vulcanizates, respectively,  $Z$  is the ratio of filler to rubber by weight in the vulcanizate, and  $a$  and  $b$  are constants.

## RESULTS AND DISCUSSION

### Visual Inspection of Foaming and Gelling Stages

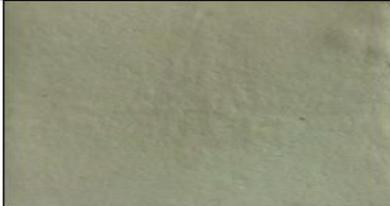
Visual inspections in the foaming and gelling stages of the NRLF without ACW filling (0 phr, or control NRLF) and with ACW filling (ACW loadings of 1.5 phr, 2.5 phr, 3.5 phr, 4.5 phr, 5.5 phr, and 6.5 phr) are shown in Table 2. The control NRLF had fine bubbles of uniform size and a specular surface after vulcanization, and the cases with 1.5 phr ACW and 2.5 phr ACW in NRLF were similar in these respects. However, gel time increased with ACW loading, and the number density of the foam cells increased while they decreased in size, relative to the unfilled control NRLF. Classical nucleation theory of polymer foams proposes two types of cases, homogenous and heterogeneous, and the addition of solid particles to a solution leads to heterogeneous nucleation (Mokhtari Motameni Shirvan *et al.* 2016). The homogenous bubble nucleation site in control NRLF was potassium oleate, whereas the filled NRLF had both potassium oleate and ACW particles as homogenous and heterogeneous bubble nucleation sites, respectively. The small loading levels of 1.5 phr and 2.5 phr did not yet cause much heterogeneous nucleation, therefore maintaining fine and uniform bubbles. However, the greater ACW filler loadings of 3.5 phr to 6.5 phr yielded non-uniform bubbles in the foaming stage and long gel times, which may be caused by the heterogeneous nucleation occurring simultaneously with homogenous nucleation. The surfaces after vulcanization showed enlarged pores.

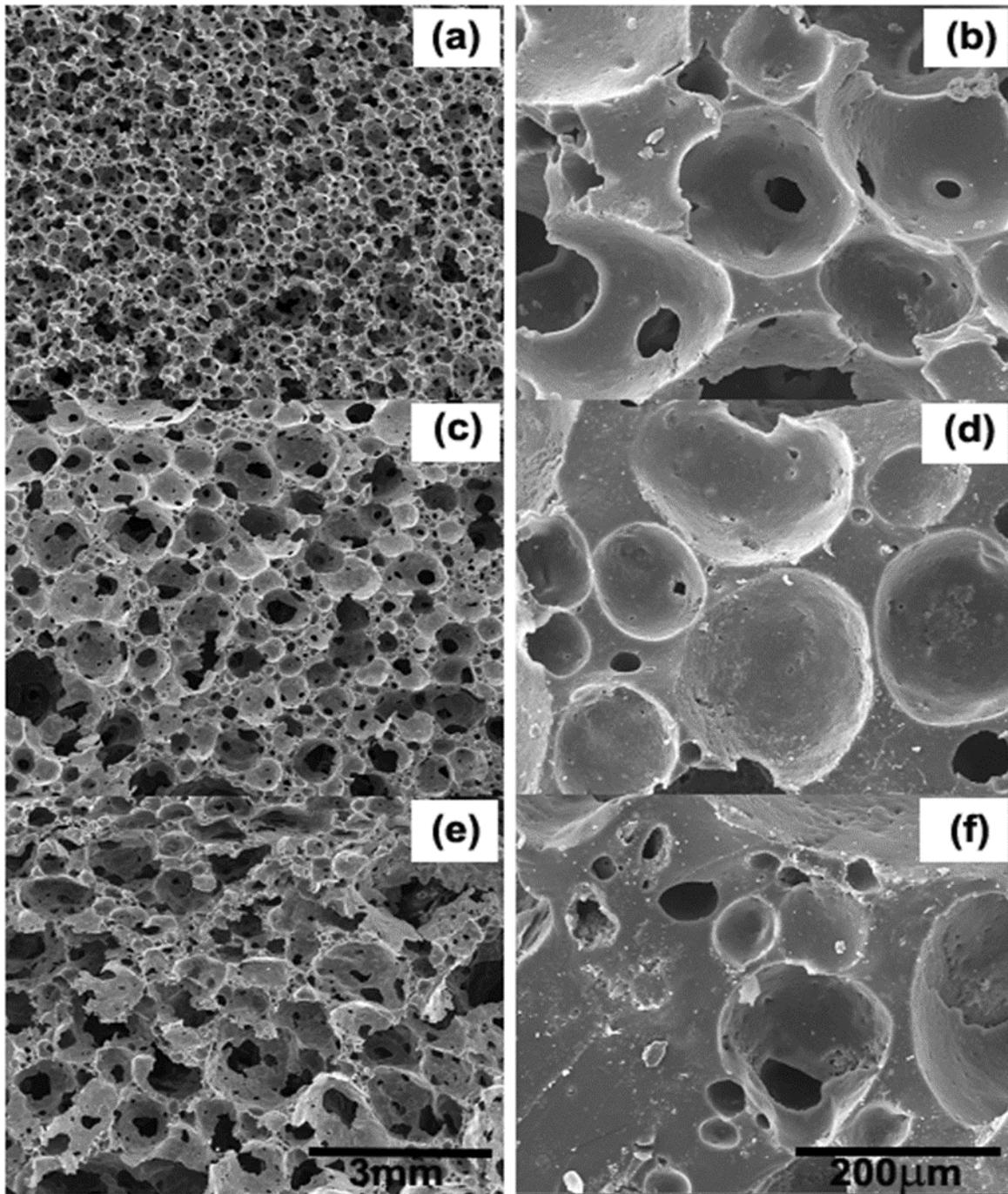
### Morphology Characterizations of ACW-filled NRLF Samples

Figures 1(a) and 1(b) present micrographs of the control NRLF, clearly revealing open and closed cell morphologies. The micrographs of the ACW-filled NRLF at the low loading of 1.5 phr ACW are shown in Fig. 1(c) and 1(d), in which polyhedral and semi-closed cells are apparent. The cell sizes in the 1.5 phr ACW-filled NRLF were heterogeneous and larger than those of the control NRLF.

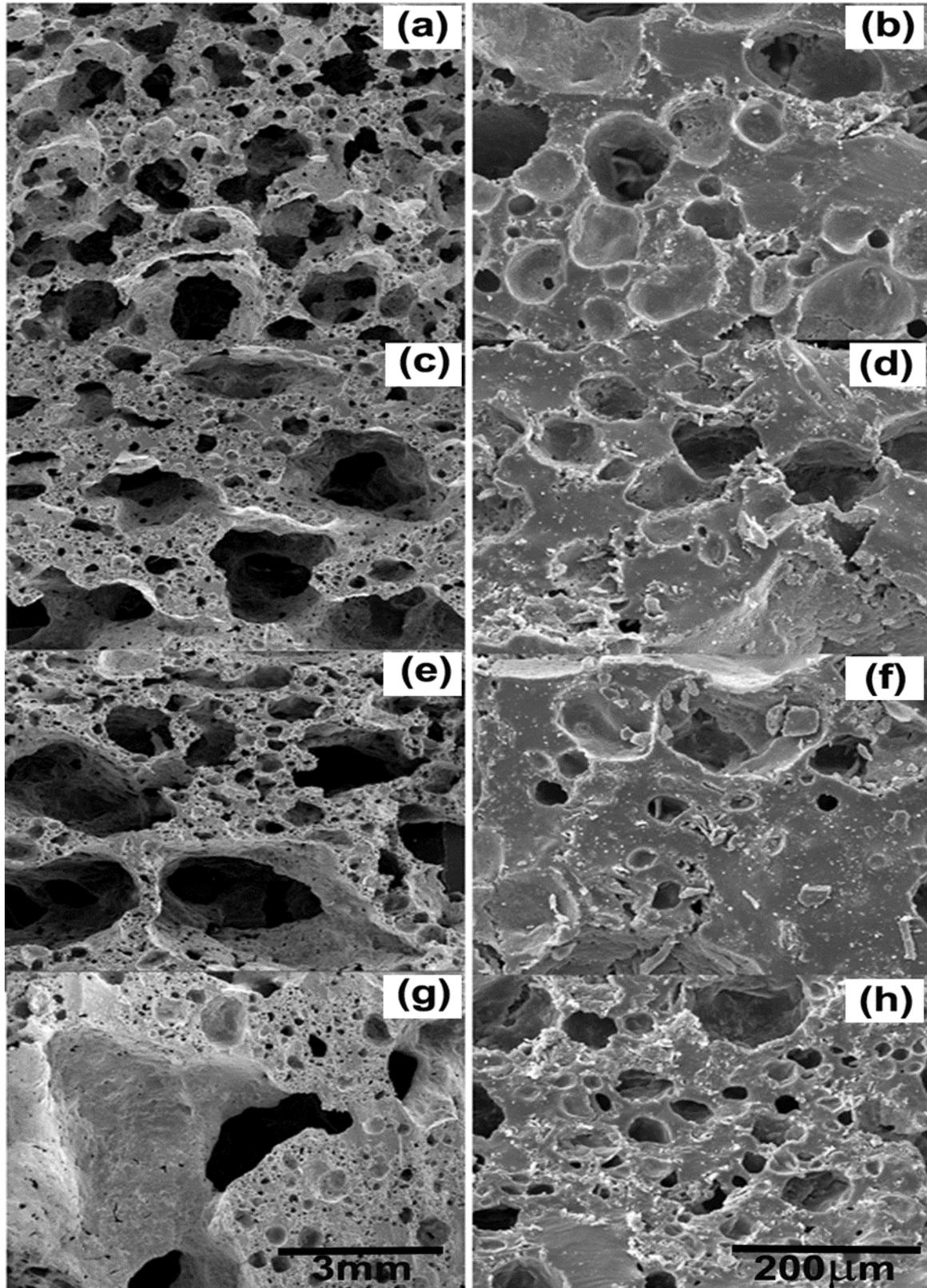
The cells in the 2.5 phr ACW-filled NRLF were larger than those with the 1.5 phr ACW filler or in the control NRLF, and they were mostly open, as shown in Figs. 1(e) and 1(f). Thick cell walls started to form in the foams with ACW loadings of 3.5 phr or greater, as shown in Figs. 2(a) and 2(b). A bimodal cell size distribution was seen in these foams. Cell walls in the 3.5 phr ACW-filled NRLF were thicker than those in the 1.5 phr and 2.5 phr ACW-filled NRLF or the control NRLF. Cell wall thickness in the ACW-filled NRLF increased with ACW loadings of 4.5 phr, 5.5 phr, and 6.5 phr, and there was a bimodal cell size distribution, as shown in Figs. 2(c) to 2(h). Various factors influence the cell morphology of foams, such as temperature, pressure, type of polymeric material, the formulation used (Ariff *et al.* 2008), filler type (Cao *et al.* 2005), and amount of filler (Tangboriboon *et al.* 2015). A nanoparticle filler yielded heterogeneous nucleation sites during cell formation, which reduced the cell size compared to a foam without the filler (Cao *et al.* 2005). Vahidifar *et al.* (2016) stated that increasing viscosity and curing density inhibits bubble growth, leading to reduced cell size. That result is different from this current work, in which the cell size increased as ACW filler loading increased. The cell size increase with any parameter helps gas release during the foaming process (Vahidifar *et al.* 2016). In this work, the obtained micrographs clearly suggested that the amount of ACW filler was an important factor affecting cell morphology, as in the prior study of Tangboriboon *et al.* (2015).

**Table 2.** Foaming and Gelling Inspections

ACW Filler Loading in the Foam (phr)	Foam Characteristics	Gel Time (min)	Foam Surface after Vulcanization
0	Fine and uniform bubbles	1	
1.5	Fine and uniform bubbles	2	
2.5	Fine and uniform bubbles	3	
3.5	Non-uniform bubbles	5	
4.5	Non-uniform bubbles	5	
5.5	Non-uniform bubbles	6	
6.5	Non-uniform bubbles	7	



**Fig. 1.** Cell morphologies of NRLF samples: control at (a) 15x and (b) 250x, ACW filler loading of 1.5 phr at (c) 15x and (d) 250x, and ACW filler loading of 2.5 phr at (e) 15x and (f) 250x



**Fig. 2.** Cell morphologies of NRLF samples with ACW filler loadings of 3.5 phr at (a) 15x and (b) 250x, 4.5 phr at (c) 15x and (d) 250x, 5.5 phr at (e) 15x and (f) 250x, and 6.5 phr at (g) 15x and (h) 250x

### Density of ACW-filled NRLF Samples

Figure 3 presents the effects of ACW filler loading on the density of filled NRLF. The density of the control NRLF (without ACW filler) was  $161.6 \text{ kg/m}^3$ . The densities of the NRLF with ACW filling in the range of 1.5 phr to 3.5 phr were lower than that of the control NRLF. The densities of the 1.5 phr, 2.5 phr, and 3.5 phr ACW-filled NRLF were  $143.4 \text{ kg/m}^3$ ,  $153.3 \text{ kg/m}^3$ , and  $161.1 \text{ kg/m}^3$ , respectively. Typically, NRLF with filler would have a greater density than that without filler (Najib *et al.* 2009), in contrast to these results.

Upon increasing the amount of ACW filler to 4.5 phr, 5.5 phr, and 6.5 phr, the density of the ACW-filled NRLF exceeded that of the control NRLF. Additionally, the density of the ACW-filled NRLF increased steadily with filler loading.

The chemical crosslinking strongly affects foam density, as it can generate a network of tightly packed polymer chains (Ariff *et al.* 2008; Najib *et al.* 2009). Thinner cell walls can indicate a lower crosslinking density (Najib *et al.* 2009). It is possible that increasing the loading of ACW filler contributed to chemical crosslinking and thereby increased the cell wall thickness (as shown in Fig. 2(c) to 2(h)) and the density of the ACW-filled NRLF.

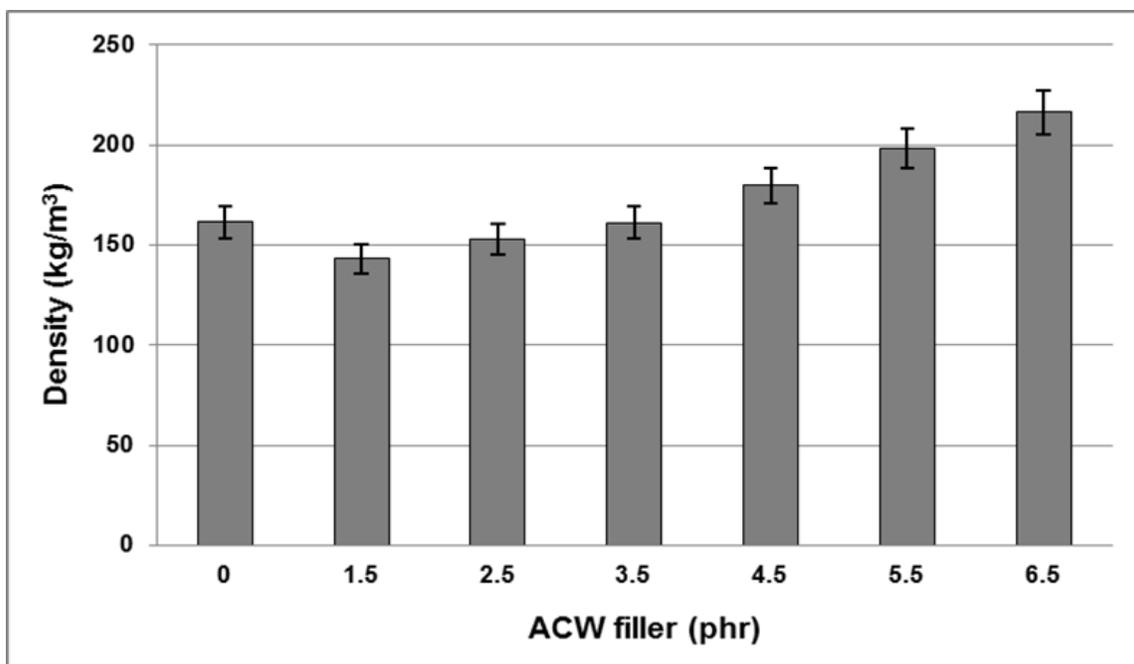


Fig. 3. Density of ACW-filled NRLF at various ACW filler loadings

### Hardness of ACW-filled NRLF Samples

Figure 4 presents the effects of ACW filler loading on the hardness of the NRLF. The control NRLF had the lowest hardness, below the limit of Shore scale A. The hardness of ACW-filled NRLF increased with increasing filler loading. This result indicates that ACW filler contributes to harder NRLF, which is consistent with effects of rice husk and kenaf fillers (Ramasamy *et al.* 2013a,b; Kudori *et al.* 2019).

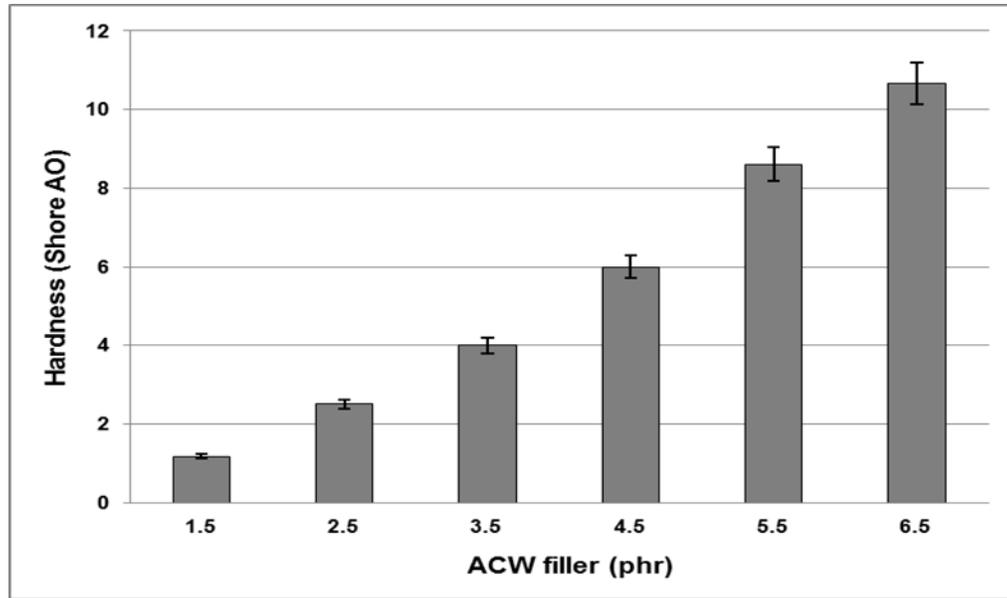


Fig. 4. Hardness of ACW-filled NRLF at various ACW filler loadings

### Compression Set of ACW-filled NRLF Samples

The dimensional durability and stability of ACW-filled NRLF under stress was determined in terms of the compression set. Figure 5 shows that the compression set of the ACW-filled NRLF increased with increased filler loading. High ACW filler loadings of 4.5 phr, 5.5 phr, and 6.5 phr yielded high compression sets ( $\geq 20\%$ ), while the compression set was 10.5% for both the 2.5 phr and 3.5 phr ACW-filled NRLF samples. The lowest ACW loading of 1.5 phr yielded a compression set of 9.5%, which was the least among the filled NRLF. However, the control NRLF had an even lower compression set (8.4%), less than any of the ACW-filled NRLF samples.

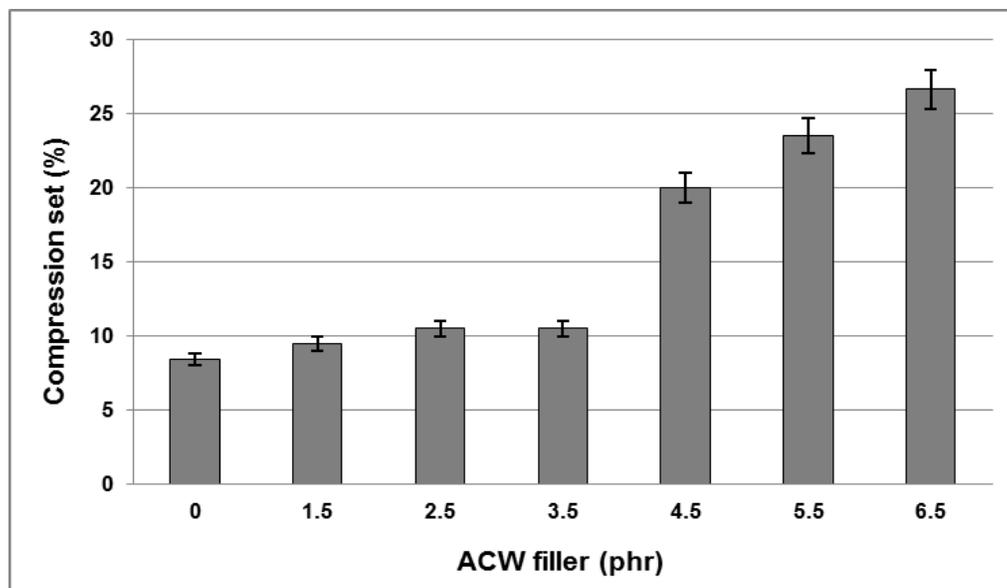


Fig. 5. Compression set values of control NRLF (without filler) and NRLF with various ACW filler loadings

Both cell wall thickness and cell size of the foam affect the compression set. Zakaria *et al.* (2007) suggested that a foam with thinner cell walls and larger cell sizes has a more elastic behavior and better compression set behavior than that with thicker cell walls and larger cell sizes. In this work, the control NRLF with comparatively thin cell walls and small cell sizes had good compression set behavior. The compression set behavior of the 1.5 phr, 2.5 phr, and 3.5 phr ACW-filled NRLF was slightly inferior, with thicker cell walls and larger cell sizes, as shown in Figs. 1 and 2. The cases with 4.5 phr, 5.5 phr, and 6.5 phr ACW filler loadings had bimodal cell size distributions and thicker cell walls, as shown in Fig. 2, leading to clearly inferior compression set behavior. Nevertheless, the compression sets of the 1.5 phr, 2.5 phr, 3.5 phr, and 4.5 phr ACW-filled NRLF are acceptable according to Thai industrial product standards (TIS 173-2519), for NRLF (Industrial product standards Act, 1976), in which the compression set must not exceed 20%. Such acceptable loadings could be used to make fragrant foam rubber products, including mattresses and pillows.

### Swelling of ACW-filled NRLF Samples

Figure 6 displays the trend of rubber-filler interactions ( $Q_f/Q_g$ ) in the ACW-filled NRLF samples. A lower  $Q_f/Q_g$  indicates stronger filler-matrix interactions (Ismail *et al.* 1999). The results showed that ACW filler decreased the  $Q_f/Q_g$  ratio, which consistently decreased with the filler loading. This result explains why the ACW filler provided good rubber-filler interactions in the ACW-filled NRLF. A similar study with rattan powder as the filler in natural rubber compounds has been reported (Muniandy *et al.* 2012). Increasing the amount of ACW filler reduced filler-filler interactions but increased filler-matrix interactions. This increased crosslink formation. These results are further supported by morphological evidence (Ramasamy *et al.* 2012).

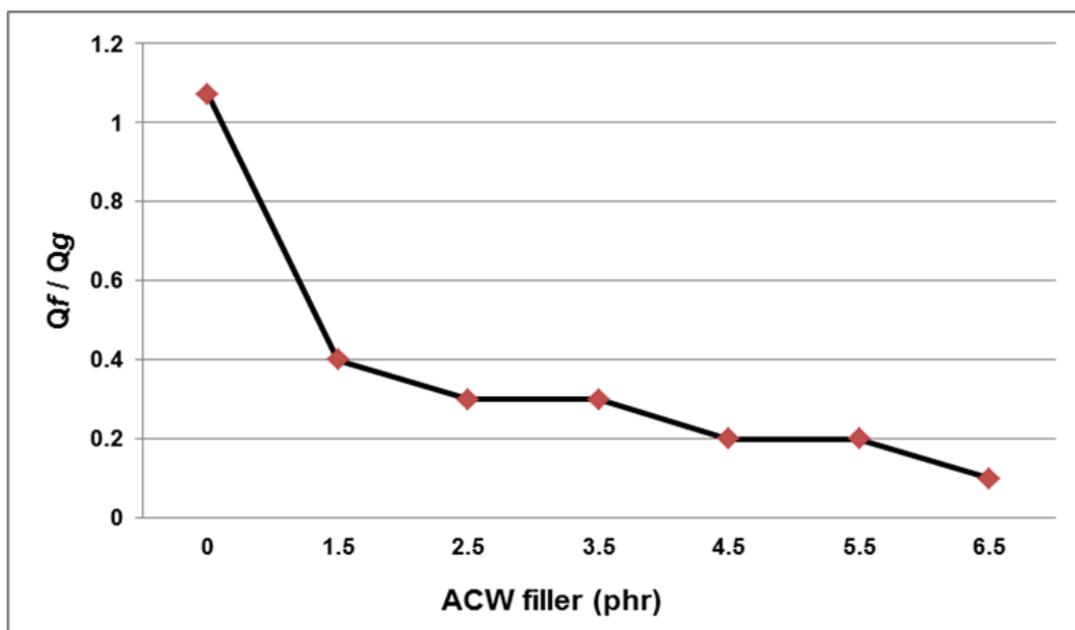


Fig. 6. Rubber-filler interactions in the NRLF at various ACW filler loadings

## CONCLUSIONS

1. Increasing the loading of fragrant ACW filler in NRLF from 1.5 phr to 6.5 phr affected the cell morphology of the foam. The cell size in ACW-filled NRLF increased with filler loadings of 1.5 phr and 2.5 phr, compared with that of the control NRLF without filler.
2. A bimodal cell size distribution, with large and small cells in the ACW-filled NRLF, was mainly formed at greater (3.5 phr, 4.5 phr, 5.5 phr, and 6.5 phr) loading levels. The cell walls also became thicker. In addition, the density and hardness of the ACW-filled NRLF increased with filler loading.
3. The compression set of the ACW-filled NRLF samples was poor at the 5.5 phr and 6.5 phr loadings, exceeding 20%. However, the compression set with the 1.5 phr, 2.5 phr, 3.5 phr, and 4.5 phr loadings was acceptable according to the Thai industrial product standard (TIS 173-2519) for NRLF (Industrial product standards Act, 1976).

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