Effect of Partial Replacement of Kenaf by Empty Fruit Bunch (EFB) on the Properties of Natural Rubber Latex Foam (NRLF)

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Kenaf was replaced by various amounts of empty fruit bunch (EFB) in natural rubber latex foam (NRLF). Five different compositions of kenaf/EFB (7/0, 5/2, 3.5/3.5, 2/5, 0/7 phr) were prepared by using the Dunlop method. The comparison of tensile properties, morphology, foam density, compressive strength, hardness, swelling, compression set, and accelerated aging of natural rubber latex foam (NRLF) were studied. The tensile strength, modulus at 100% (M_{100}), foam density, compressive strength, hardness, and compression set decreased with increasing EFB loading. However, the elongation at break and swelling percentage of NRLF increased as the content of EFB decreased. Morphological studies showed that a higher amount of EFB weakened the interaction between EFB and NRLF matrix and increased the formation of cell windows. The EFB-filled NRLF also showed better tensile retention compared to kenaf filled NRLF.

Keywords: Natural rubber latex foam; Kenaf; Empty fruit bunch; Tensile properties

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INTRODUCTION

The use of lignocellulosic materials such as wood or cellulose in the production of natural rubber-based product is becoming more common (Ismail *et al*. 2012; Ramasamy *et al*. 2012; Norjulia *et al*. 2016). Lignocellulosic materials have many advantages compared to synthetic materials such as being less abrasive to the equipment, low density, renewable character, and environmental friendliness (Xu *et al*. 2012). Natural fibers promote the ‘green’ economy based on energy efficiency. The organic waste that results from the processing can be used as a fuel and generate the electricity. The selection of natural fibers usually depends on its geographical availability (Pickering *et al*. 2016). For example, Europe is known for flax fiber (Foulk *et al*. 2002), while the fibers attracting the biggest interest in Asian regions are jute, kenaf, and rice husk (Tripathy *et al*. 2000; Aji *et al*. 2009; Akil *et al*. 2011).

Kenaf (*Hibiscus cannabinus*) originated from West Africa. It is a warm seasonal plant and can grow under a wide range of weather conditions, either in heavy rainfall or high solar radiation (Ashori *et al*. 2006; Akil *et al*. 2011). Kenaf can be harvested after 3 months of plantation and absorbs more carbon dioxide (CO_2) than other crops (Saba *et al*. 2014). The stem and bark of the kenaf have distinct qualities and can be used in various products, for example, car accessories, animal feed, biofuels, and paper-based products (Ismai *et al*. 2011). Kenaf has been established in Malaysia as a new industrial crop with high potential for product development (Subramaniyan *et al*. 2013).

The oil palm tree (*Elaeis guineensis*) originated from the Palmae family and the coastal strip of Africa (Anuar *et al*. 2018). Oil palm is a renewable resource that is abundantly available in Malaysia and an important part of the economy (Rosli *et al*. 2017). Malaysia has become one of the biggest producers and exporters of oil palm products through giant government agencies such as FELDA, FELCRA, and RISDA (Faizi *et al*. 2017). Unfortunately, palm oil generates a huge amount of biomass wastes of approximately 50 to 70 tonnes from every hectare, and most of the biomass is in the form of empty fruit bunch (EFB) (Islam *et al*. 2017). Each EFB has a thickness of 130 mm, weighs approximately 3.5 kg, and has an irregular shape (Chang 2014). The composition of the EFB depends on the plant age, soil condition, and testing method (Hassan *et al*. 2010).

The usage of kenaf in NRLF has been reported (Karim *et al.* 2016). It was found that the incorporation of kenaf in NRLF tended to reduce tensile properties due to the weaker rubber-filler interaction. Generally, natural fibers had some unfavorable effects on fiber dispersion in the polymer matrix. This was due to the nature of natural fibers, which are hydrophilic, as they are derived from lignocellulose. Different approaches have been applied to improve the natural fiber reinforced in polymer composites. Employing combinations of different types of natural fibers, *i.e.* as hybrid composites, is one approach that has been used to improve the properties of the materials. Using various types of complementary fibers offers a range of properties that are quite difficult to attain by using only one type of reinforcement (Ismail *et al*. 2011). Several studies have reported the hybridization of kenaf and EFB in a polymer composite. Islam *et al.* (2017) studied the physical and thermo-mechanical properties of kenaf and EFB in polylactic acid (PLA). They found that the fiber can support the other fiber to achieve high mechanical properties of the material. Hanan *et al*. (2018) studied the mechanical performance of oil palm/kenaf fiber reinforced epoxy-based bilayer hybrid composites, showing that the higher loading of kenaf to EFB improved the tensile and flexural properties. The present study incorporated kenaf/EFB with NRLF natural rubber latex foam (NRLF). The mechanical properties and microstructure of the kenaf/EFB-filled NRLF was studied.

EXPERIMENTAL

Materials and Formulation

The Dunlop method was being used in this study. The advantages of using Dunlop method are that it requires less massive capital investment in its equipment, and it can achieve higher durability and environmentally sustainability in the product. The formulations of the foam are given in Table 1.

Natural rubber latex (High Ammonia (HA) latex) and latex chemicals (sulphur, antioxidant, potassium oleate, zinc diethyldithio-carbamate (ZDEC), zinc 2-mercaptobenzhiozate (ZMBT), zinc oxide, diphenylguanidine (DPG), and sodium silicofluoride (SSF)) were supplied by Zarm Scientific & Supplies Sdn. Bhd., Bukit Mertajam, Malaysia. Kenaf was supplied by the National Kenaf and Tobacco Board, Kota Bharu, Malaysia. Oil palm empty fruit bunch in a fibrous form was obtained from Sabutek (M) Ltd, Teluk Intan, Perak, Malaysia.

Table 1. Formulation of Kenaf Core and Bast Filled NRLF

Ingredient	Total Solid Content (TSC%)	Parts per hundred rubber (pphr)
HA latex	60	100
Sulphur	50	2.2
Antioxidant	50	1
Potassium Oleate	20	4.5
ZDEC	50	0.9
ZMBT	50	0.9
Zinc Oxide	50	3
DPG	40	0.3
SSF	25	1.2
Kenaf / EFB	-	7/0, 5/2, 3.5/3.5, 2/5, 0/7

TSC%: percentage by weight of the whole, which is non-volatile at definite temperature

Sample Preparation

The procedure is outlined in Fig. 1. High ammonia latex concentrate (HA latex) was filtered, weighed, and stirred by a mechanical stirrer for 10 min at low speed. A vulcanizing agent (sulphur) and accelerator (ZMBT and ZDEC) were added and stirred for 15 min. Kenaf powder and EFB (7/0 phr) were added slowly in the mixer for 1 h.

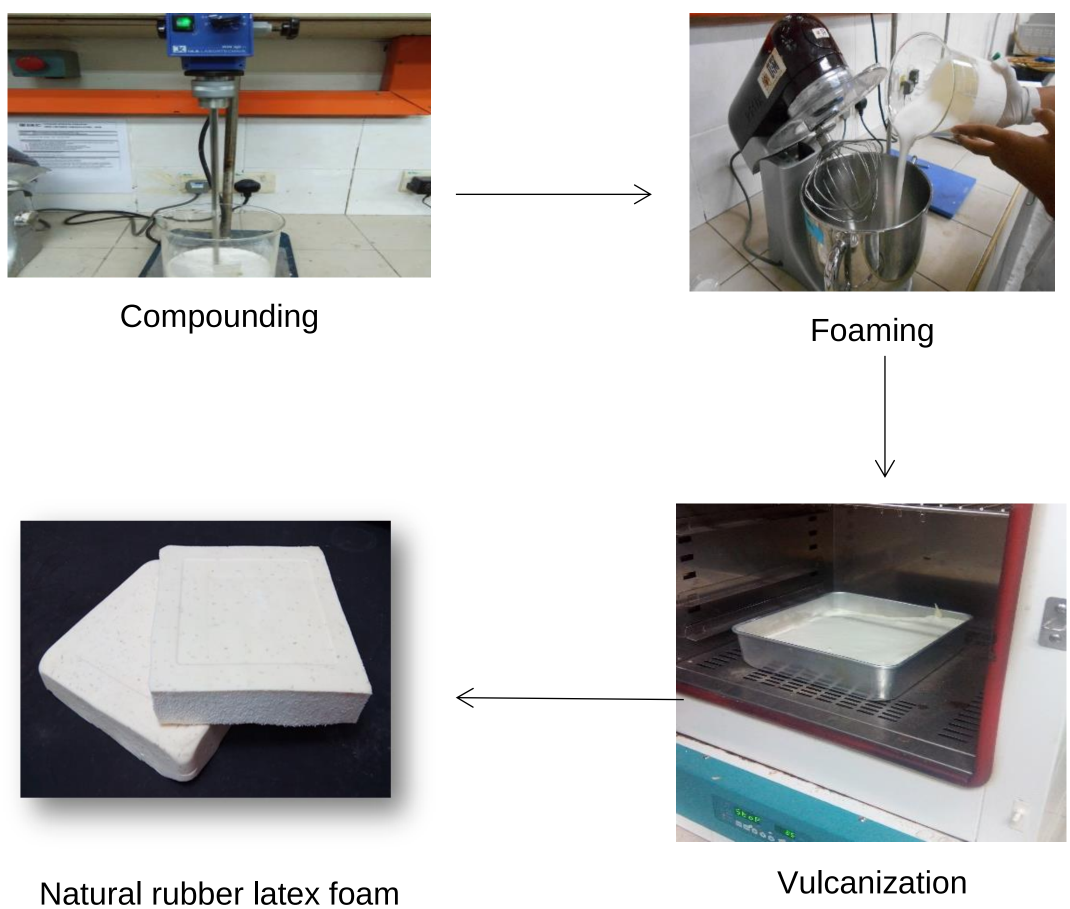

Fig. 1. The steps for preparation of NRLF by using Dunlop method

The foaming agent (potassium oleate) was added slowly for 6 h at 10 rpm. The NRLF compound was intensely beaten for 5 min using a stand mixer (Kenwood, kMix) until the volume was increased to three times the initial volume. The speed was lowered once the desired volume for a fine and even foam was achieved. Next, the primary gelling agent (zinc oxide (ZnO), and diphenylguanidine (DPG)) were added for 1 min; a secondary gelling agent (sodium silicofluoride (SSF)) was added for 1 min. The un-gelled foam was placed on the desired aluminium mould for 3 min. The gelled foam was cured in a hot air oven at 105 °C for 2 h. The cured NRLF was stripped from the mould and washed thoroughly with distilled water to remove excessive non-reacted material. Then, it was oven-dried at 80 °C for 8 h. These steps were repeated for 5/2, 3.5/3.5, 2/5, and 0/7 phr of kenaf/EFB filled NRLF**.**

Measurement Tensile Properties

Tensile properties were measured by using an Instron universal testing machine, model 3366 (Norwood, MA, USA) according ASTM D3574-03(E) (2003) with the crosshead speed 500 mm/min. A tensile test was conducted on dumbbell shaped from the sheet of the NRLF with a thickness approximately 2 mm by using a Wallace die cutter. Data such as tensile strength, tensile modulus, and elongation at break can be obtained from the tests.

Morphology

The microstructure of kenaf, EFB, and the surface morphology of kenaf/EFB-filled NRLF were observed using a field emission scanning electron microscope (FESEM; Supra 35VP, Zeiss, Oberkochen, Germany). The samples were sputter-coated with a thin layer of gold and surrounded with aluminium stubs. From the resulting SEM micrographs, the rubber-filler interactions, kenaf/EFB dispersion, and pore morphology of the foams were evaluated.

Foam Density

The foam density measurement was determined using a density balance. The density was calculated as shown in Eq. 1. Specimens used in this test method were in a cuboid shape with not less than 16 cm^3 in volume. Five samples of each kenaf loading were measured and the average was reported.

$$Density\ (\frac{g}{cm^2}) = \frac{mass\ of\ specimen\ (g)}{volume\ of\ specimen\ (cm^2)} \tag{1}$$

Compressive strength

Compression testing was carried out on the Instron 3366 universal testing machine at crosshead speed of 50 mm/min according ASTM D3574-03 (2003). The samples were rectangular with dimensions of 50 mm × 50 mm × 25 mm. The specimens were compressed up to 50% of their original thickness.

Hardness

The hardness of kenaf/EFB-filled NRLF was measured by using the foam and sponge rubber durometer 302SL (PTC Instruments, Los Angeles, USA), which classified cellular rubbers of the foam type as “x-soft”, “soft”, “medium”, or “firm” (Table 2). The dimensions of tested samples were 55 mm × 30 mm × 25 mm (length × width × thickness).

Table 2. Foam and Sponge Rubber Durometer 302SL Value Ranges

Class	Value Range
X-Soft	19-27
R32-Soft	27-42
R33-Medium	42-66
R34-Firm	66-90
R35-Xfirm	90-100

Swelling

Swelling was tested according ASTM D471-06 (2006). Kenaf/EFB-filled NRLF samples with dimensions of 30 mm × 5 mm × 2 mm were weighed, immersed in toluene, and allowed to swell in a closed bottle for 72 h. The samples were removed, and the surfaces of the swollen samples were wiped and weighed. The swelling (%) was calculated according to Eq. 2.

$$Swelling\ (\%) = \frac{Swollen\ weight\ (g) - initial\ weight\ (g)}{Initial\ weight (g)} \times 100 \quad (2)$$

Compression Set

The testing was carried out according to ASTM D1055-97 (1997). The specimens were cut in rectangular shapes with the dimensions 50 mm × 50 mm × 25 mm. The samples were placed between two plates of compression devices and compressed to 50% of their original thicknesses. Within 15 min, the compressed specimen, along with the apparatus, was placed in the air oven at 70 °C for 22 h. Specimens were immediately removed from the apparatus and the thicknesses were measured after a 30-min recovery. The recovery (%) was calculated according to Eq. 3.

$$Recovery\ (\%) = \frac{Final\ thickness}{Original\ thickness\ of\ specimen} \times 100 \quad (3)$$

Accelerated Aging

Accelerated aging was carried out in the instrument's tensile mode. Aging properties were tested according to the ASTM 1055-97 (1997). Five samples were subjected to 100 °C for 48 hours prior to tensile testing using the Instron 3366. The crosshead speed was set at 500 mm/min. The retention of each property was calculated by using Eq. 4.

$$Retention\ (\%) = \frac{value\ after\ aging}{value\ before\ aging} \times 100 \quad (4)$$

RESULTS AND DISCUSSION

Tensile Properties

Figure 1 shows the tensile strength of partial or complete replacement of kenaf by EFB-filled NRLF. The foam without any filler showed lower tensile strength compared to foam filled with 7 phr of kenaf. It was found that the incorporation of kenaf in NRLF increased the value of tensile strength. However, the tensile strength decreased as EFB is being introduced in NRLF. This could be attributed to the poor dispersion of EFB in

NRLF and agglomeration when EFB content was higher. The particulate and rough surface of EFB contributes to the strong filler-filler interaction compared to the filler-matrix interaction. Table 3 shows that the percentage of lignin for EFB is higher than the kenaf. Therefore, the EFB has higher tendency to agglomerate, which reduces the contact area of fiber with matrix.

Figure 1 shows that kenaf-filled NRLF had higher tensile strength than the EFB-filled NRLF. Thus, the type of fiber affected the tensile strength of the NRLF. This could be explained by the different cellulose content of the fibers. Cellulose is important for load transfer in fiber (Tezara *et al*. 2016). Kenaf has a higher percentage of cellulose (54.1%) than EFB (41.3%), as shown in Table 3. Cellulose fiber is made of bundles of cellulose molecules in the form of microfibrils that are arranged linearly, with crystalline regions alternating with amorphous regions. They form intra- and intermolecular hydrogen bonds. The fibrous structure and strong hydrogen bonds result in higher tensile strength (Karina *et al*. 2007). Subsequently, the higher percentage of cellulose increased the tensile strength of kenaf-filled NRLF.

The elongation at break of the composites are presented in Fig. 2. The elongation at break of kenaf or EFB filled NRLF was lower compared to the pure NRLF. This can be explained by the incorporation of filler, which restricts the mobility of flexible rubber chain. Furthermore, the elongation at break increased as EFB content filled NRLF also increased. The addition of EFB in kenaf-filled NRLF improved the flexibility of the foam. This might be due to the lower cellulose content, which contributes to the high elongation at break of EFB-filled NRLF. The elongation at break of EFB was 10 times higher than kenaf (Anuar *et al.* 2018). The EFB-filled NRLF had a higher elongation at break than kenaf-filled NRLF.

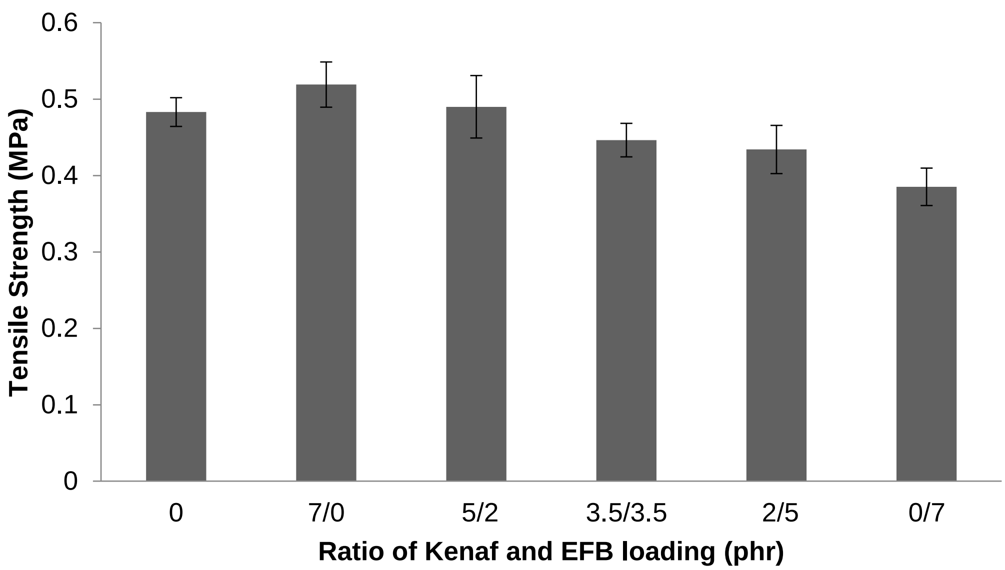

Fig. 2. Effect of kenaf/EFB loading on the tensile strength of natural rubber latex foam

Figure 3 presents the modulus at 100% (M_{100}) with partial or complete replacement of kenaf by EFB-filled NRLF. Kenaf-filled NRLF had a higher M_{100} than EFB-filled NRLF, which might be due to cellulose content in kenaf. The foam without filler had lower M_{100} compared to the foam that contained kenaf or EFB in NRLF. Kenaf-filled NRLF had higher M_{100} compared to EFB-filled NRLF. This also might be due to cellulose content in kenaf. Crystalline cellulose increases the stiffness of the foam by restricting the mobility of the rubber chain (Chen 2014). Kenaf-filled NRLF had a higher M_{100} compared with the EFB-filled NRLF.

Table 3. Chemical Composition of Kenaf and EFB (Anuar *et al*. 2018)

Component	Kenaf (%)	EFB (%)
Holocellulose	86.3	62.6
α-Cellulose	54.1	41.34
Lignin	20.35	27.71
Ash	2.21	5.29

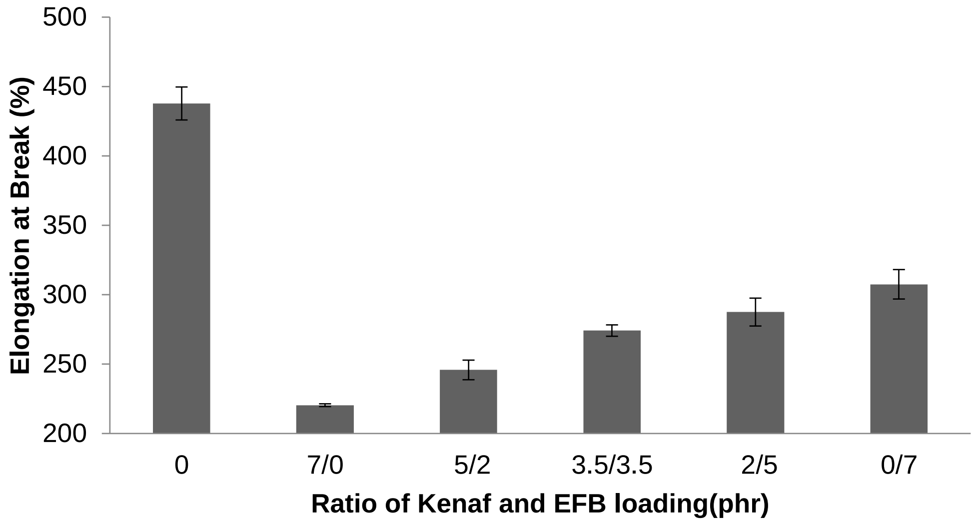

Fig. 3. Effect of partial replacement of kenaf with EFB on elongation at break of natural rubber latex foam (NRLF)

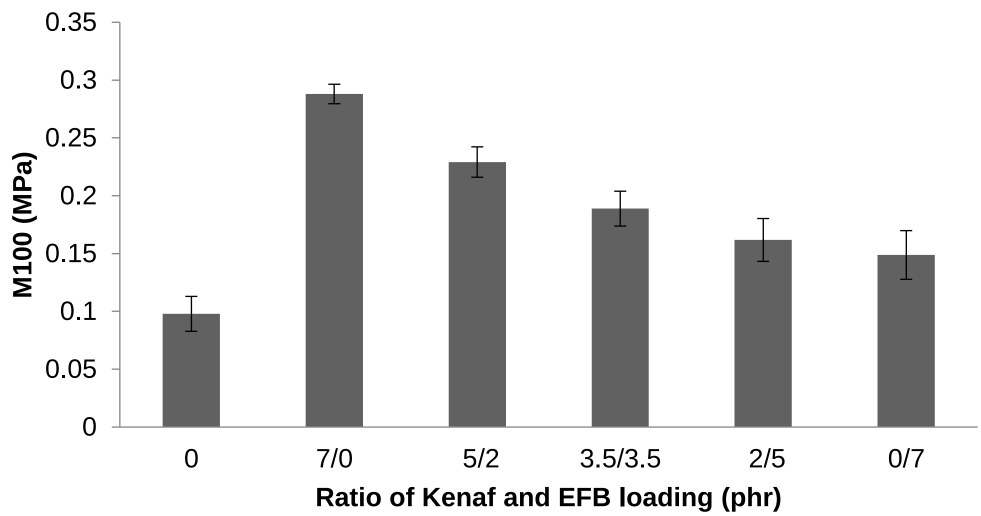

Fig. 4. Effect of partial replacement of kenaf with EFB on modulus of NRLF

Morphology

Figure 5 (a) presents the surface micrograph of EFB magnification at 100x.

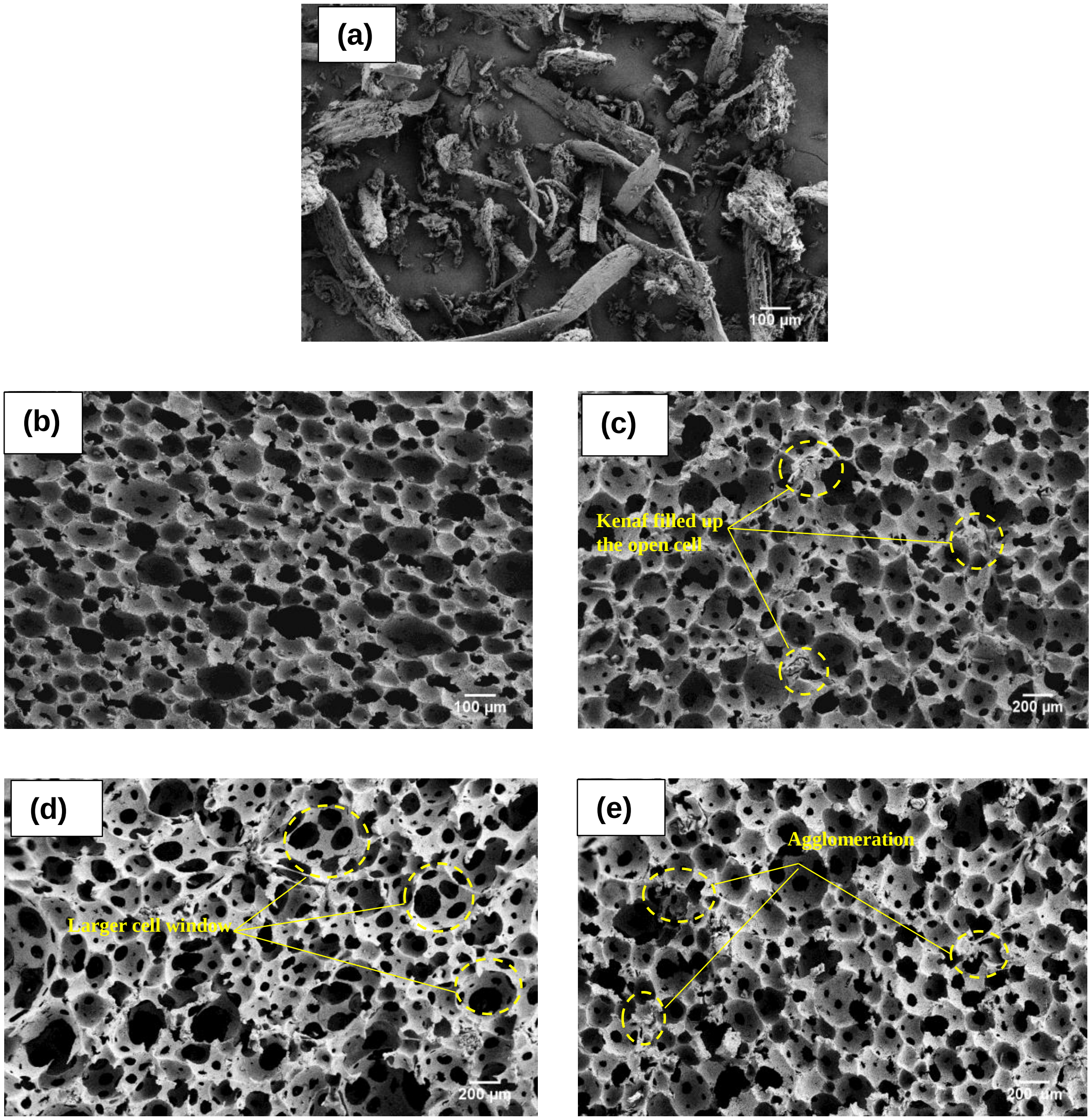

Fig. 5. Surface micrograph (a) EFB magnification at 100 x, (b) pure NRLF, (c) 7/0 of kenaf/ EFB, (d) 0/7 of kenaf/EFB and (e) 3.5/3.5 of kenaf/EFB

The EFB had the combination of particulate and fibrous shapes with a rough surface. The tendency of EFB to agglomerate lessens it ability to enhance the mechanical properties because of the confinement in the interfacial area.

Figure 5 (b) shows a micrograph of pure NRLF, while part (c) shows a micrograph of 7/0 of kenaf/EFB filled NRLF. It shows that the fibrous of kenaf filled up the open cell of the NRLF. Figure 5 (d) shows the formation of larger cell window once the EFB is being introduced at 7 phr. The small cell windows coalesce with each other and form larger windows and makes the cell walls become thinner compared to the other loading. These findings reveal that the higher amount of EFB weakens the interaction between EFB-NRLF and enhances the formation of cell window. Figure 5 (e) presents a surface micrograph of 3.5/ 3.5 of kenaf-filled NRLF. The filler failed to distribute well and formed agglomerates when the amount of kenaf and EFB was equal. This might be due to the presence of EFB, as the structure of the EFB has the combination of particulate and fibrous shapes with rough surfaces as discussed in Fig. 5 (a), resulting in filler-filler interaction.

Foam Density

The foam density of partial or fully replacement of kenaf by EFB-filled NRLF is shown in Fig. 6. The foam density of pure NRLF was lower compared to that of kenaf or EFB-filled NRLF. This may be due to the addition of kenaf into NRLF and its effects on mass of the NRLF itself. The foam density was slightly reduced as the kenaf was replaced by the EFB. This observation was attributed to the structure of NRLF. The addition of EFB increased the size of window formation, and more spaces were created in the foam, which made the foam lighter. The cell walls of the foam also became thinner at 0/7 phr of kenaf/EFB loading, as shown in Fig. 5(d), which contributed to the lower density. Hence, the presence of more windows and thinner cell walls reduces the density of the foam. In addition, the density of fiber itself affects the density of the foam. The density of kenaf is 1.62 g/cm^3, which is higher than EFB (1.52 g/cm^3) (Anuar *et al*. 2018). Subramaniyan *et al*. (2013) reported that the density of the filler affects the density of the foam. The foam filled with kenaf had a higher density than the one filled with EFB.

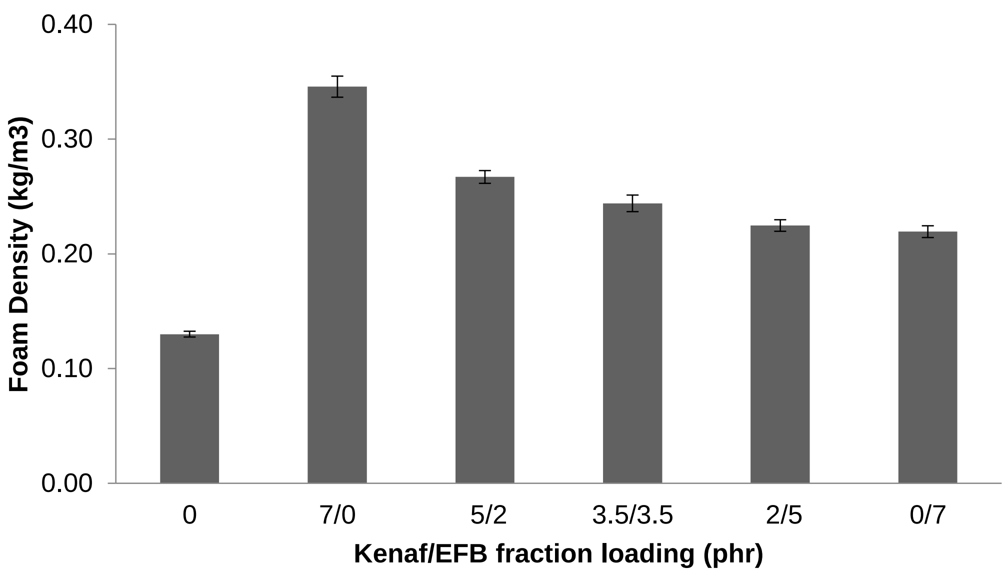

Fig. 6. Foam density of partial or fully replacement of kenaf by EFB-filled NRLF

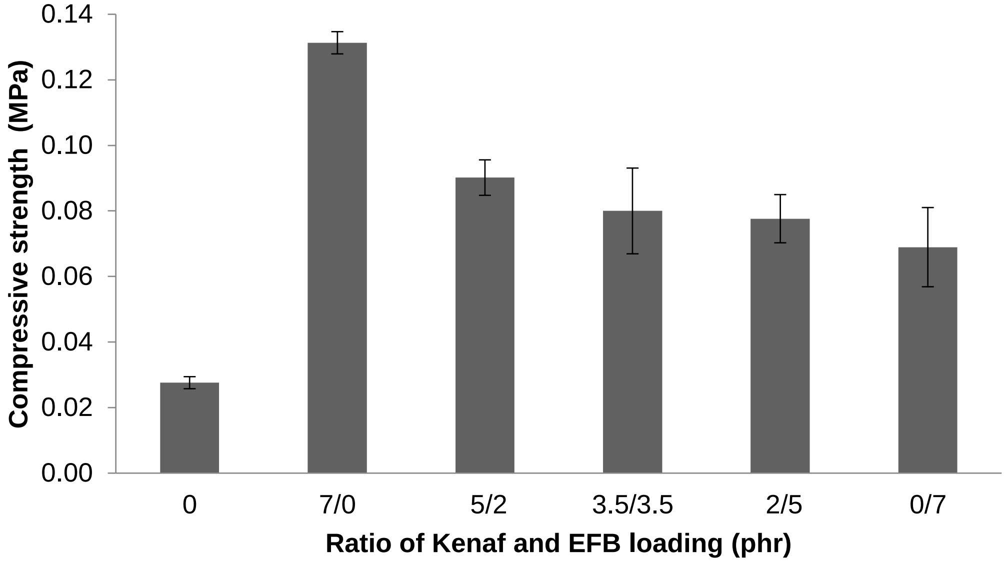

Fig. 7. Compression test of partial or fully replacement of kenaf by EFB-filled NRLF at 50% strain

Compressive Strength

Figure 7 shows the compressive strength with partial or full replacement at 50% of kenaf by EFB-filled NRLF. The compressive strength of pure NRLF was less compared to the kenaf- or EFB-filled NRLF. The compressive strength was higher than the kenaf- or EFB-filled NRLF. This may be because the addition of filler increased the stiffness of the foam. The compression strength of foam was reduced as EFB loading increased. The EFB showed poor dispersion in the matrix when the EFB loading increased, which caused agglomeration and minimized the surface area for interaction between filler and matrix. More cell windows were formed in EFB-filled NRLF, which led to less formation of matrices, as the load was unable to distribute over the foam network once the force was applied. The foam became susceptible to a higher load, and less force was needed to compress at 50% strain. A foam with low density tends to have lower compressive strength, as the density of the foam is related to the cell wall (Zakaria *et al*. 2007). The thinner cell wall at 0/7 phr of kenaf/EFB was easy to bend, resulting in less compressive strength. Therefore, as kenaf was replaced by the EFB-filled NRLF, the compressive strength was reduced.

Hardness

Figure 8 represents the hardness of partial or full replacement of kenaf by EFB-filled NRLF. The hardness of NRLF decreased with increasing EFB loading. The value range of hardness of kenaf-filled NRLF at 7/0 kenaf/EFB was 89, while EFB-filled NRLF at 0/7 kenaf/EFB fraction loading (phr) was 65. All values were in the range of R34-Firm, as shown in Table 2. Kenaf-filled NRLF had higher hardness than the EFB due to the cellulose percentage in kenaf, which had higher cellulose content. Higher cellulose increased the stiffness of the foam due to its regular arrangement and results in a high degree of crystallinity. In addition, the structure of the cell plays an important role in hardness (Dahlia *et al*. 2009). The larger cell window exhibited less foam per cubic centimetre and reduced hardness. EFB showed the larger cell window with less foam density, resulting in a lower hardness value.

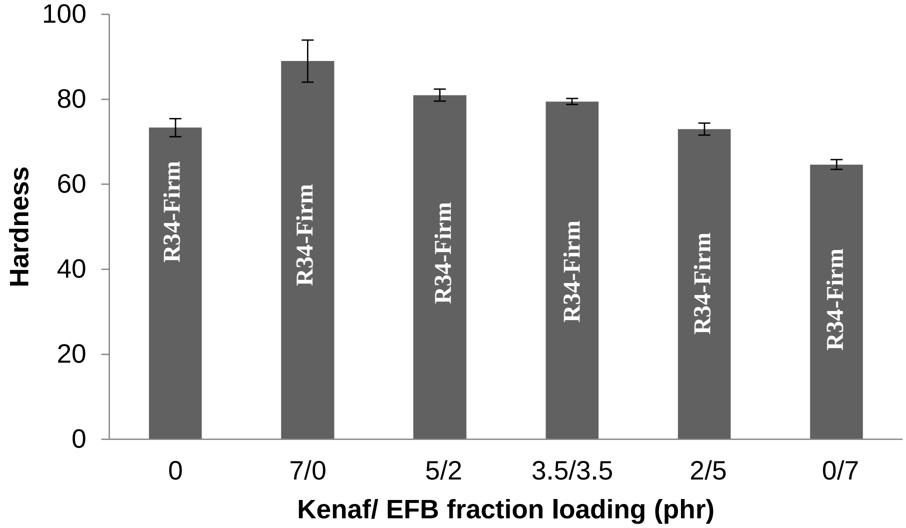

Fig. 8. Hardness of partial or fully replacement of kenaf by EFB-filled NRLF

Swelling

Figure 9 illustrates the swelling with partial or full replacement of kenaf by EFB-filled NRLF. The swelling percentage of pure NRLF was less compared to the kenaf- or EFB-filled NRLF. This might be due to the high free volume in pure NRLF. Furthermore, it also showed the increasing trend in swelling percentage after replacing kenaf with EFB. The toluene uptake into kenaf/EFB-filled NRLF increased with increasing EFB loading. There was a higher swelling percentage as kenaf was fully replaced by EFB in NRLF. This might be due to the poor dispersion of EFB in NRLF, which promotes poor rubber filler interaction in foam. Thus, higher toluene can be diffused in the NRLF. Furthermore, EFB had less crystallinity than kenaf, as discussed in tensile properties. Less crystalinity of EFB increased the ability of the toluene to diffuse into the NRLF. Hence, EFB-filled NRLF had a higher swelling percentage than the kenaf-filled NRLF.

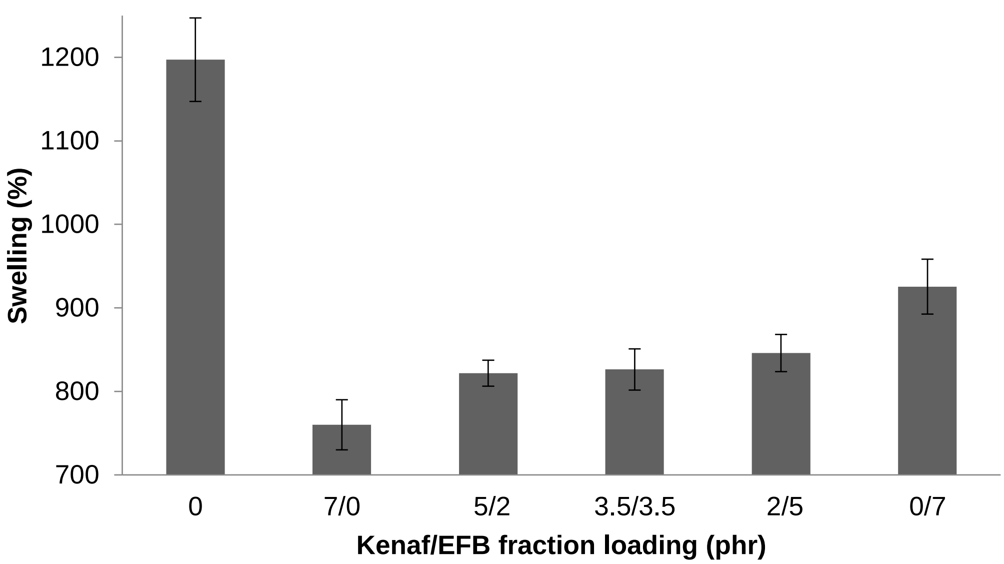

Fig. 9. Swelling percentage of partial or fully replacement of kenaf by EFB-filled NRLF

Compression Set

As shown in Fig. 10, the compression set of partial or fully replacement kenaf by EFB-filled NRLF increased with the increasing EFB. This might be due to the better dispersion of kenaf at 7 phr loading, which contributed to the smaller deformation. As the kenaf became evenly distributed, the tendency between particles to agglomerate decreased, which weakened the filler network and increased the elasticity behaviour of foam. Figure 11 shows the recovery percentage of partial or fully replacement kenaf filled NRLF. The recovery percentage of pure NRLF was higher compared to the kenaf- or EFB-filled NRLF. Furthermore, the structure of foam affected the compression set of the foam (Tangboriboon *et al*. 2014). Larger sized cell windows in 0/7 phr of kenaf/EFB-filled NRLF exhibited thinner cell walls, which might be easily ruptured once the consistent strain, load, and heat are applied. The foam had less cell wall to act as a strain recovery, resulting in less recovery percentage. Thus, the distribution of filler and structure of foam affected the value of compression set and recovery percentage of the foam.

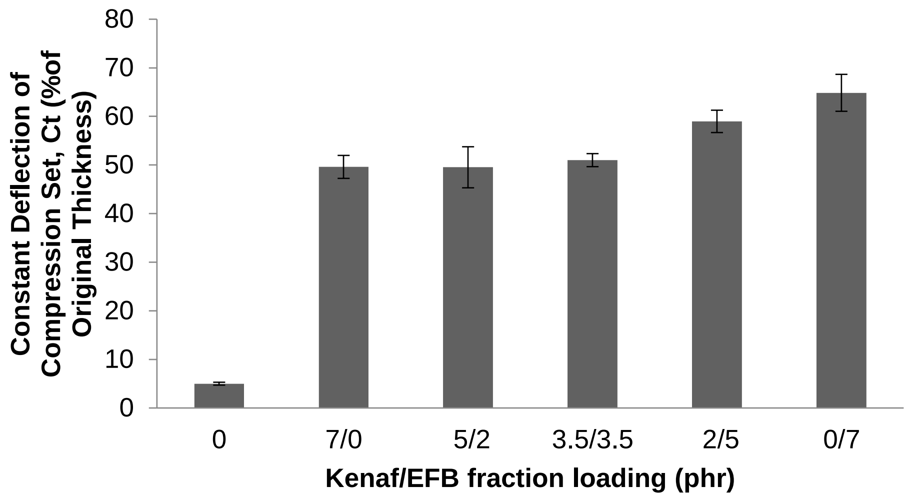

Fig. 10. Compression set of partial or fully replacement kenaf by EFB-filled NRLF

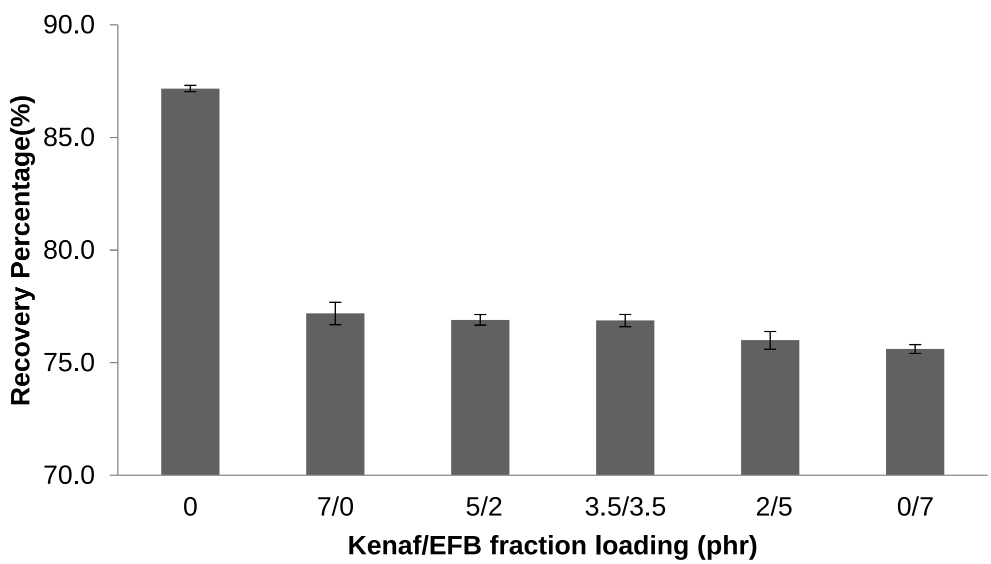

Fig. 11. Recovery percentage of partial or fully replacement of kenaf by EFB-filled NRLF

Accelerated Aging

The resistance of foam to thermal aging is required for better service performance. The effects of tensile strength, elongation at break, and M_{100} with partial or full replacement of kenaf by EFB-filled NRLF are shown in Figs. 12 and 14.

Based on Figs. 12 and 13, the retention of tensile strength and elongation at break of partial or fully replacement before and after aging of EFB filled NRLF was low compared to kenaf-filled NRLF. This was due to the structure and morphology of the foam. The structure of EFB filled NRLF had bigger cell windows compared to kenaf filled NRLF.

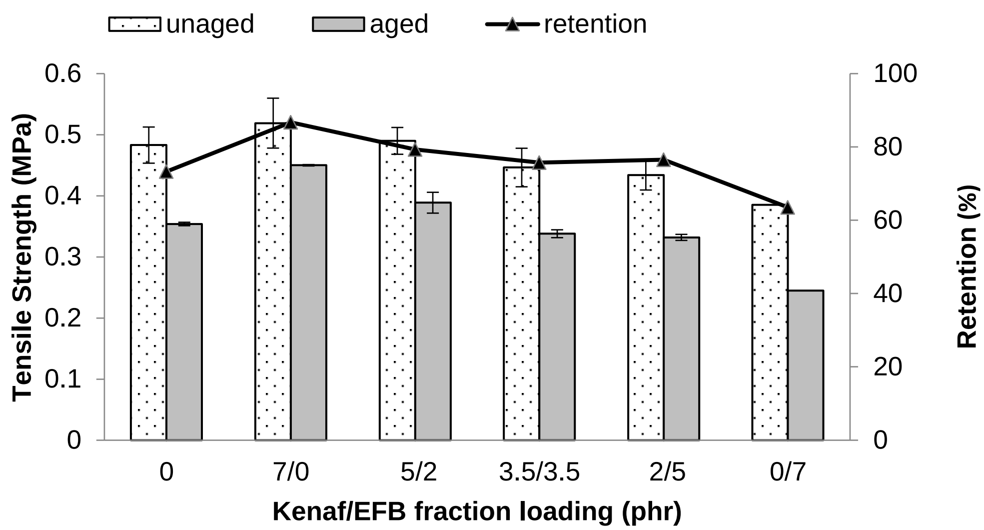

Fig. 12. Tensile strength with partial or full replacement kenaf by EFB-filled NRLF after the aging process

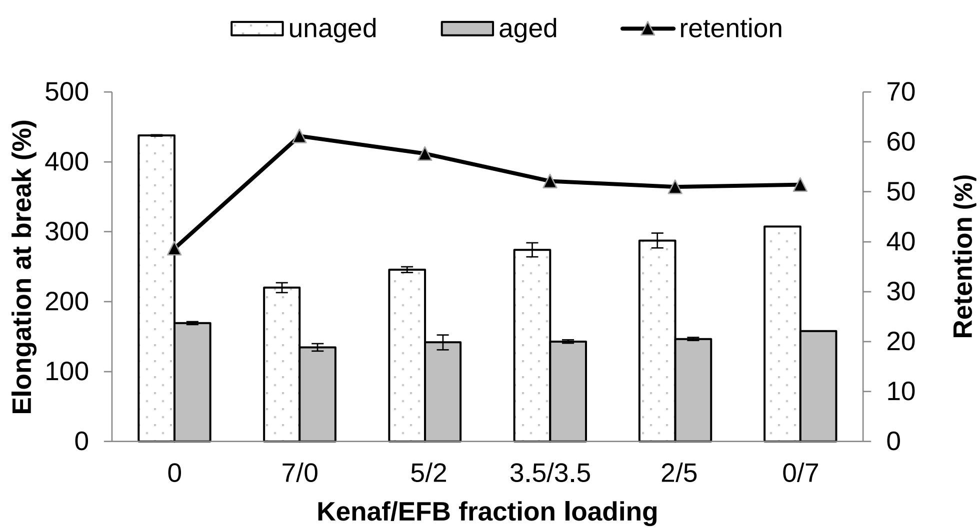

Fig. 13. Elongation at break with partial or full replacement kenaf by EFB-filled NRLF after the aging process

As stress is applied to the foam during tensile test, the cell windows will not be able to contribute its strength to the foam. As more bigger cell windows were formed within the foam, the tensile strength and elongation at break decreased. Thus, the retention of tensile strength and elongation at break of partial or fully replacement before and after aging of EFB filled NRLF was less compared to kenaf-filled NRLF.

Figure 14 presents the modulus at 100% (M_{100}) with partial or full replacement of kenaf and EFB-filled NRLF. M_{100} and the percentage of retention increased as EFB loading increased. This result might be due to the large pendant group of lignin crosslinking during aging, resulting in stiffer and more rigid foam. It reduced the elasticity of the foam, which resulted in a higher modulus but lower elongation at break.

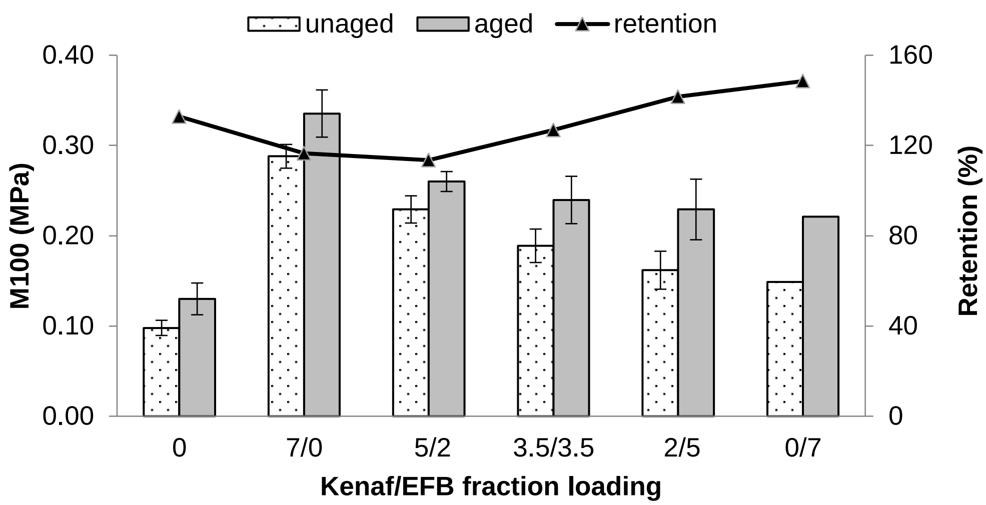

Fig. 14. Modulus at 100% (M_{100}) with partial or full replacement kenaf by EFB-filled NRLF after the aging process

CONCLUSIONS

1. The tensile strength, M_{100}, foam density, compression strength, compression set, and hardness decreased as the EFB loading increased.
2. There was notable difference in the morphology of kenaf/EFB-filled NRLF, which affected the tensile strength properties, compressive properties, hardness, and foam density. The size of cell windows in NRLF tended to increase as kenaf was fully replaced by the EFB.
3. Full replacement of kenaf by EFB resulted in a higher retention of tensile strength and M_{100}, which led to better aging resistance.

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