Kenaf Core and Bast Loading *vs.* Properties of Natural Rubber Latex Foam (NRLF)

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Natural rubber latex foam (NRLF) based on kenaf core and bast were prepared by using the Dunlop method. Mechanical properties, foam density, compression, hardness, thermal aging, and microstructural characteristics of kenaf-filled NRLF with different loadings of kenaf core and bast were analysed. The tensile strength, elongation at break, and compressive strength of kenaf bast filled NRLF samples decreased as the content of kenaf was increased. However, the modulus at 100%, hardness, and density of NRLF increased with increasing kenaf contents. The addition of kenaf core into NRLF increased the swelling percentage and aging of the foam compared to the bast. Scanning electron microscopy (SEM) results indicated that the fibrous kenaf bast had a stronger adhesion compared to the particulate kenaf core, resulting in higher tensile strength, elongation at break, and compression strength.

Keywords: Kenaf core; Kenaf bast; Natural rubber latex; Foam

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INTRODUCTION

Natural rubber latex (NRL) comes from *Hevea brasiliensis* trees. It has outstanding mechanical properties and makes for an excellent rubbery material. The production of porous rubber products from natural latex, known as latex foam, was founded by Schidrowitz and Goldsbrough in the year 1914 (Ramasamy *et al*. 2013). Recently, the development of natural rubber latex foam (NRLF) has become popular in the rubber industry. The main advantages of NRLF include having a porous structure, which gives a high level of comfort capability, and a high resilience against repeated force. Natural rubber latex foam can be applied in various applications such as cushioning in automobile seats and furniture, inner footwear soles, pillows, mattresses, and upholstery foam (Lim 2010).

The combination of natural fibers in polymer leads to engineering green composites (Han *et al*. 2012). Recently, the utilization of natural fibers in polymers has attracted the interest of researchers around the world due to its prospects for certain applications especially in aerospace and the biomedical industry (Namvar *et al.* 2014). The advantages of natural fibers are their excellent mechanical properties, especially high specific tensile properties, and their nonabrasive nature on equipment, which makes them preferred in various applications such as packing, construction, and automobiles (Aizan *et al*. 2013). Because natural fibers offer an environmental advantage with competitive specific strengths and moduli, natural fibers are becoming favoured over synthetic fibers (Joshi *et al*. 2004).

Kenaf (*Hibiscus cannabis* L.) originates from West Africa and grows in tropical, mild climates with heavy rainfall (Ashori *et al*. 2006). Kenaf grows rapidly under good meteorological conditions with maximum heights up to 3.7 m to 5.5 m after 4 to 5 months. Kenaf plants also can absorb higher percentages of carbon dioxide (CO_2) compared to any other crop (Saba *et al.* 2015). The kenaf plant has a single, straight stalk with branches. The stems of kenaf can be classified into two layers; inner, wood-like core, and outer, fibrous bark. Stalks of kenaf are 60% to 65% core and 35% to 40% bast (Akil *et al*. 2011). Over the past few years, kenaf has been attracting attention and has been considered for applications such as pulp, paper, textile, and thermal insulation boards (Ayadi *et al*. 2017). This is due to the 80% cellulose content of kenaf plants, which contributes to the strength and stiffness of the fibers, and the 20% lignin content, which is thermally stable and responsible for UV degradation of the fibers.

The preference for natural fibers is due to their cost effectiveness and environmental friendly raw material. Furthermore, natural fiber also can be derived from a renewable resource (Ismail *et al.* 1997). Loh *et al.* (2016) studied the mechanical properties on kenaf reinforced polypropylene (PP) composites. They obtained comparable properties to those kenaf filled polypropylene. Shahril *et al.* (2017) reported the effect of kenaf loading in natural rubber composite for engine rubber mounting. They found that the optimum value of kenaf loading is 4%, which gives a higher tensile strength and modulus. Some researchers also have already reported the incorporation of fibers into natural rubber latex foam (Bashir *et al*. 2017). The use of baked starch to reinforce NRL has been studied in some detail. It improves the water resistance and flexibility of starch-based products (Shey *et al*. 2006). Karim *et al*. (2016) studied the properties and characterization of kenaf-filled NRLF. The result showed that, as the filler loading increased, the tensile strength, elongation at break, and compressive strength were all reduced. Despite that, to the best of our knowledge, there have been no research studies that have compared the properties of two main components; bast and core filled in NRLF.

Development of kenaf based on kenaf-core and -bast filled NRLF in this work is a novel direction. Thus, this work aims to compare the tensile properties, density, compression, swelling percentage, accelerated aging, and micro structural character of kenaf core and bast filled NRLF.

EXPERIMENTAL

Table 1. Formulation of Kenaf Core and Bast Filled NRLF

Ingredients	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 Core or Bast	-	1/3/5/7

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

Materials and Formulation

The formulations used in this study are shown in Table 1. Natural rubber latex (High Ammonia (HATZ) type) and latex chemicals (sulphur, antioxidant, potassium oleate, zinc diethyldithiocarbamate (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 fibers in core and bast forms were supplied by the National Kenaf and Tobacco Board (Kota bharu, Malaysia). They were ground and sieved into an average particle size of 250 µm.

Sample Preparation

High ammonia latex concentrate (HATZ) type NRL was filtered, weighed, and stirred by a mechanical stirrer for 10 min at low speed. After that, a vulcanizing agent (sulphur) and an accelerator (ZMBT and ZDEC) were added and stirred for 15 min. After that, kenaf powder core or bast was added slowly in the mixer for 1 h. The foaming agent (potassium oleate) was added slowly for 6 h at 10 rpm. The foaming process of NRLF compound was carried out by using a stand mixer (Kenwood, kMix). The NRLF compound was intensely beaten for 5 min 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. Then, the primary gelling agent (zinc oxide (ZnO), and diphenylguanidine (DPG)) were added for 1 min. Next, the secondary gelling agent (sodium silicofluoride (SSF)) was added for 60 sec. The un-gelled mixture was then placed on the desired aluminium mould for about 3 min for gelling purposes. The gelled foam was cured in a hot air oven at 105 °C for 2 h. The kenaf core or bast filled NRLF was stripped from the mould and washed thoroughly with distilled water to remove excess non-reacted material. The cured NRLF was dried in the oven at 80 °C for 8 hours. These steps were repeated without kenaf core or bast for preparing the control sample.

Measurement of Tensile Properties

The stress-strain properties of the kenaf core and bast filled NRLF were measured using an Instron Universal Testing Machine, model 3366 (Norwood, MA, USA) according ASTM D412 and with a 500 mm/min cross-head speed. Five samples were cut from each of the kenaf loadings in dumbbell shapes by using a Wallace die cutter. Tensile properties such as tensile strength, elongation at break, and tensile modulus were obtained and average results were reported.

Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) was used to study the surface morphology of the kenaf core and bast filled NRLF samples. 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 and kenaf dispersion as well as the pore morphology of the foams were evaluated.

Foam Density

The foam density measurements were carried out by using a density balance. Density of the kenaf core and bast filled NRLF was determined by using a formulation 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.

$$\text{Density (g/cm}^3) = \frac{\text{mass of specimen (g)}}{\text{volume of the specimen (cm)}^3} \quad (1)$$

Compression Test Measurement

The Instron 3366 was used to carry out the compression tests for the kenaf core and bast filled NRLF in accordance with ASTM D3574 (2003). The dimensions for the sample were 50 mm x 50 mm x 25 mm. A testing speed of 50 mm/min was used, and the test was performed at a test temperature of 25 °C. Five specimens for each loading were tested. The specimens were compressed up to 50% of their original thickness. The stress *versus* kenaf loading at 50% compression were obtained.

Hardness

Hardness of kenaf core and bast 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 x 30 mm x 25 mm (length x width x 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 Test

Kenaf core and bast filled NRLF samples with dimensions of 30 mm x 5 mm x 2 mm were weighed, immersed in toluene, and allowed to swell in a closed bottle for 72 hours. The samples were then removed and the surfaces of the swollen samples were wiped and weighed. The swelling (%) was calculated according to Eq. 2.

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

Compression Set Measurement

The testing was carried out according to ASTM D1055-97 (1997). Five specimens were used in this testing with the dimensions 50 mm x 50 mm x 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

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

Accelerated Aging

Accelerated aging was carried out in the instrument's tensile mode. 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.

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

RESULTS AND DISCUSSION

Tensile Properties

Figure 1 shows the tensile strength for different loadings of kenaf core or bast in NRLF. The tensile strength of kenaf core or bast decreased with increasing filler loadings. This result is in agreement with Ismail *et al.* (2002) for other filled composites. The decrease in tensile strength with kenaf core loading is caused by the agglomeration of the filler, as is shown later in the morphology study. The agglomeration leads to an increase in stress concentration. Hence, kenaf core was unable to disperse uniformly in NRLF as its loading increased. The decrease in tensile strength of kenaf bast in NRLF could be attributed to the size of the pores. The pore size of kenaf bast filled NRLF increased and the fibers became less interconnected between each other.

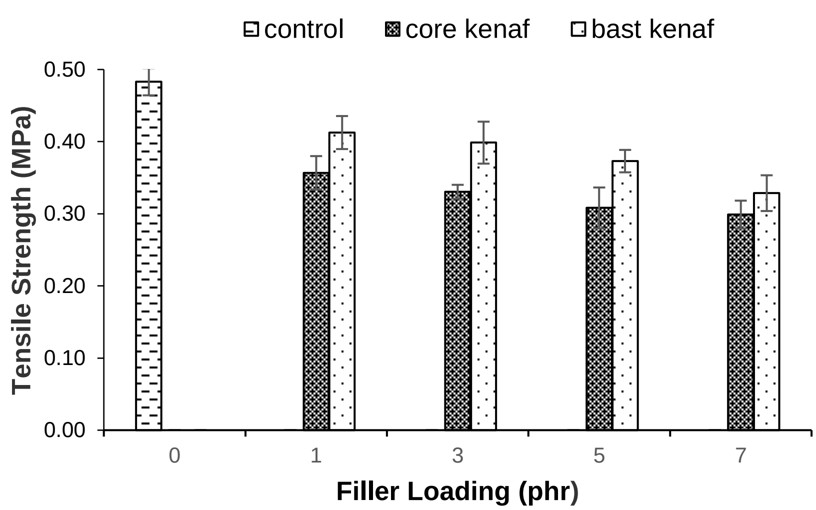

Fig. 1. The effect of filler loading on tensile strength of kenaf core or bast filled NRLF

Figure 1 also shows that the kenaf bast had higher values of tensile strength compared to the kenaf core. This might be due to the structure of the kenaf bast, which contains more cellulose (55%) than does the core fibers (49%). This is shown in Table 3 (Nayeri *et al*. 2013). Cellulose is a main structural component that provides strength and stability to the fibers. Cellulose structure consists of large linear polymer chains with many hydroxyl groups. Furthermore, the shape of the fiber also plays an important role in promote the higher tensile strength of bast. Bast fibers have a fibrous shape, which resulted in good adhesion, hence producing good physical interactions between kenaf and NRLF. However, core fibers have a more particulate shape and tend to agglomerate, as shown in an SEM micrograph that will be discussed later.

Figure 2 shows the results of elongation at break of core and bast kenaf filled NRLF. The elongation at break decreased as the loadings of kenaf increased. Increasing the kenaf loading resulted in stiffer and harder NRLF. This reduced the elasticity of the foam, which led to lower elongation at break values. Figure 3 shows how the modulus at 100% increased as the kenaf loading was increased. Higher loadings of kenaf resulted in higher stiffness. The NRLF penetrated between the kenaf's aggregates and stacks, acting as the filler network, which increased the stiffness of the core or bast filled NRLF. The elasticity of the foam decreased and the tendency of the foam to cause failure became high after the force was applied (Karim *et al.* 2016). The addition of kenaf core and bast in NRLF reduced the elasticity of the foam, but it increased the rigidity. Figures 2 and 3 also show that the kenaf bast filled NRLF had a higher elongation at break and modulus at 100% compared to kenaf core filled NRLF. The structure of the cellulose in kenaf bast has highly crystalline, compact structures which also promotes a higher stiffness. The kenaf bast filled NRLF was less deformed as stress was applied to the foam.

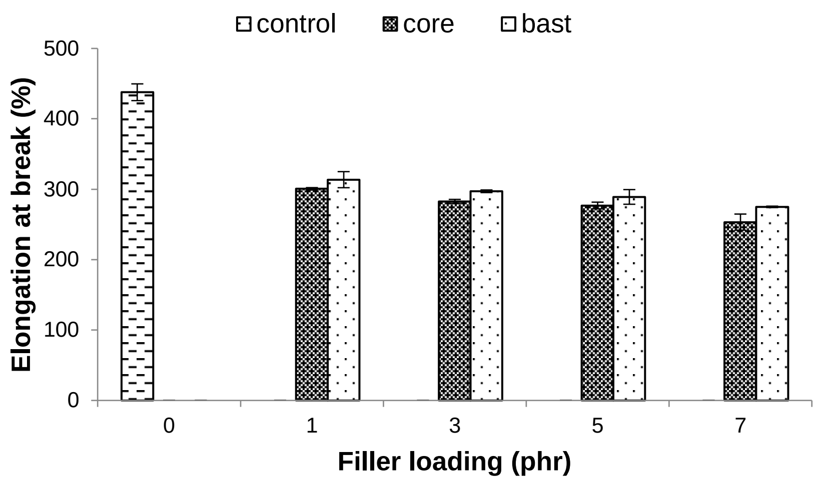

Fig. 2. The effect of filler loading on elongation at break of kenaf core or bast filled NRLF

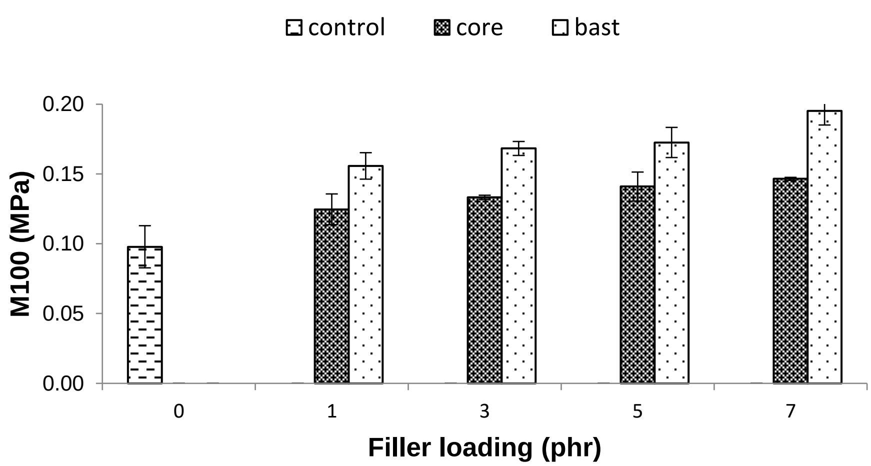

Fig. 3. The effect of filler loading on modulus 100% elongation (M_{100}) of kenaf core or bast filled NRLF

Table 3. Chemical Composition and Physical Properties of Kenaf Core and Bast Fibres (Nayeri *et al.* 2013)

Macrofibril size/ Chemical content	Bast	Core
Fibril length, *L*(mm)	2.22	0.75
Fibril width, *W* (lm)	17.34	19.23
L/*W*	128	39
Lumen diameter (lm)	7.5	32
Cell wall thickness (lm) Cellulose (%)	36	1.5
Lignin (%)	14.7	19.2
Hemicellulose (%)	26.2	29.7
Ash content (%)	5.4	1.9
Holocellulose (%)	86.8	87.2
α-cellulose (%)	55	49

Morphology

Figure 4 shows SEM images of kenaf core and bast powders. Figure 4(a) shows that the kenaf core had in an irregular and particulate shape. The stronger tendency of kenaf core particles to agglomerate with each other resulted in a less interconnected structure of rubber. As shown in Fig. 4(b), the kenaf bast had a fibrous shape. The lengths of these fibrous shapes were higher compared to those of the particulate shapes, resulting in greater reinforcement of the filler.

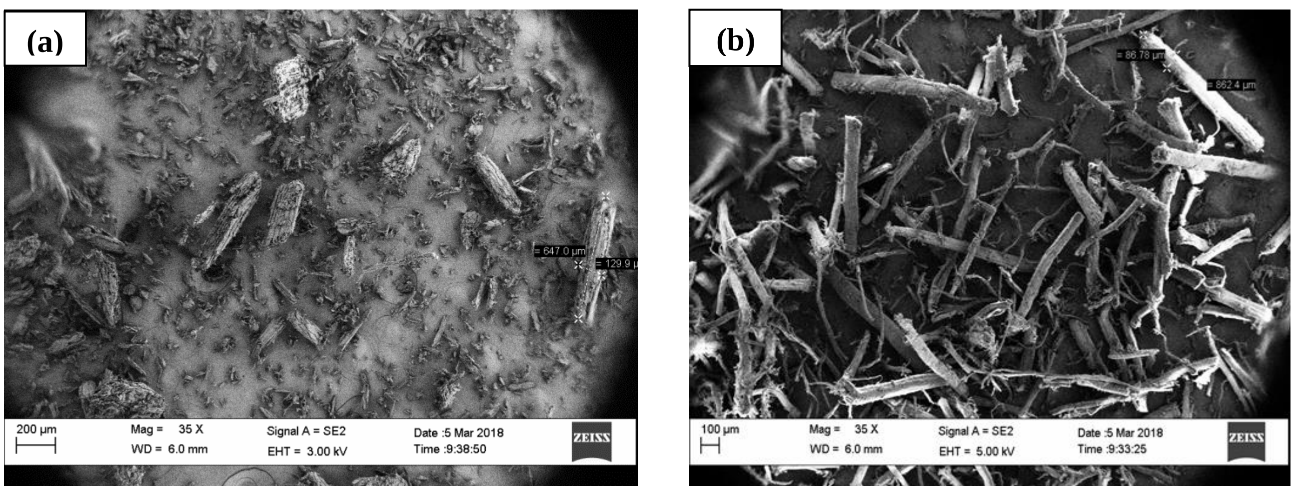

Fig. 4. (a) Micrograph of kenaf core powder at magnification of 35X, (b) Micrograph of kenaf bast powder at magnification of 35X

Figure 5 shows the open cell structure for control sample, 1 phr, and 7 phr. Figure 5(a) shows SEM images of the control sample of NRLF at 50X magnification. The pores were less interconnected, which led to relatively higher tensile strength and elongation at break compared to the kenaf core and bast filled compounds. The SEM results of 1 phr kenaf core and bast filled NRLF are shown in Figs. 5(b) and (c). Figure 5(c) shows that kenaf bast-filled NRLF had a more homogenous pore distribution compared to the kenaf core-filled NRLF (shown in Fig. 5(b)). Furthermore, it is evident in Fig. 5(d) that the kenaf core showed a lot of agglomeration, suggesting a weak interaction between rubber and filler.

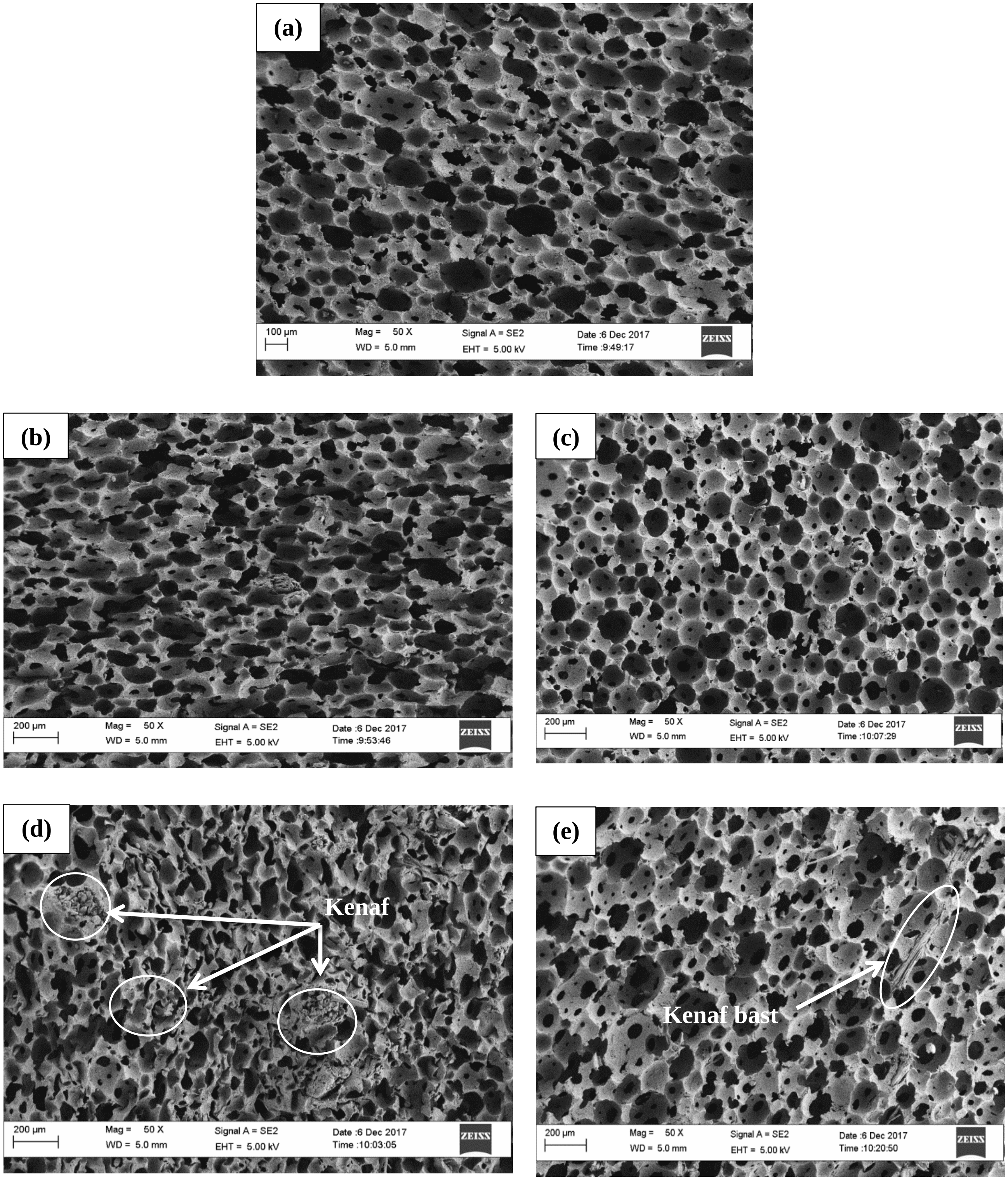

Fig. 5. (a) Micrograph of surface control sample NRLF at 50X, (b) Micrograph of surface at 1 phr kenaf core filled NRLF at magnification of 50X, (c) Micrograph of surface at 1 phr kenaf bast filled NRLF at magnification of 50X, (d) Micrograph of surface at 7 phr kenaf core filled NRLF at magnification of 50X, (e) Micrograph of surface at 7 phr kenaf bast filled NRLF at magnification of 50X

As higher kenaf loadings for both types of kenaf were used, the pores became increasingly interconnected, which led to the reduction in tensile strength and elongation at break. The cell walls of the pores also were ruptured at 7phr of kenaf core and bast. In Fig. 5(e), kenaf bast-filled NRLF in a 7 phr of kenaf loading showed more fibrous shape with higher lengths. The fibrous shapes tended to create a strong adhesion between filler and rubber. The pores were also less interconnected. Thus, the tensile strength and elongation at break of bast were higher compared to the core.

Density Test

Figure 6 presents the effect of kenaf core and bast loading on the density of NRLF. It clearly shows that the foam density increased as the kenaf loading was increased. This was due to the hindered of the foaming process caused by the additional increments of kenaf loadings. Figure 4 also shows that kenaf bast filled NRLF had a higher density compared to the kenaf core filled NRLF with the same filler loading. This can be explained by the walled structure of pores in kenaf core filled NRLF. It would rupture and two or more pores would coalescence, as shown in Fig. 4(b). Higher amounts of coalescences will decrease the density, as the resulting NRLF foam will have fewer matrices.

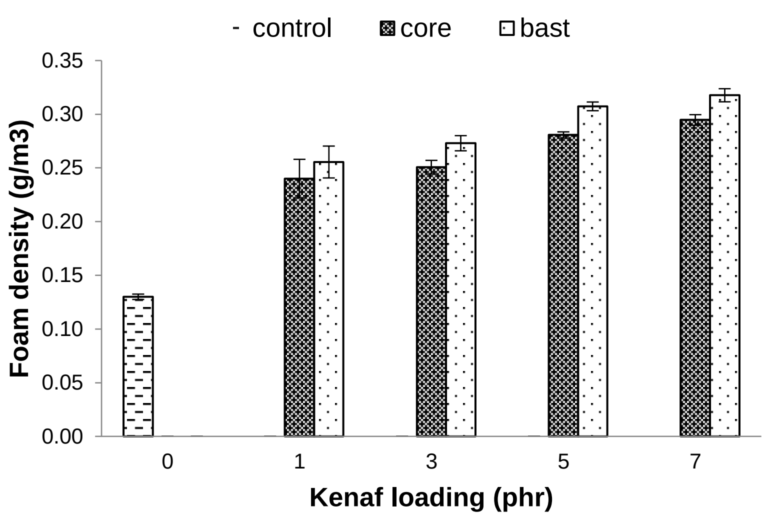

Fig. 6. The effect of filler loading on density of kenaf core or bast filled NRLF

Compression Test

Figure 7 shows the effect of kenaf core or bast loadings on the compression test at 50% strain values. The values of stress increased as the filler loadings increased. At higher kenaf loadings, a higher amount of force was needed to compress the kenaf filled NRLF at 50% strain. Increments in filler loading contributed to the higher stiffness of the foams. At the same filler loadings, kenaf bast filled NRLF showed higher values of compression compared to the kenaf core filled NRLF values. Less coalescence in the kenaf bast filled NRLF led to a greater formation of matrices, which were able to more effectively distribute the load over the foam network when it was applied.

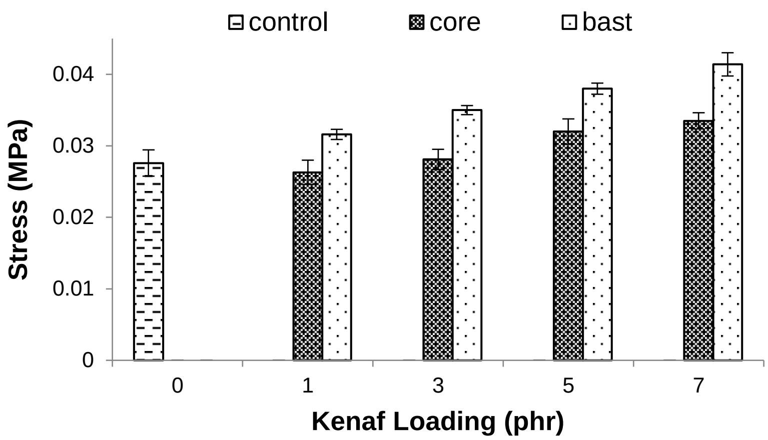

Fig. 7. The effect of filler loading on compression test at 50% strain of kenaf core or bast filled NRLF

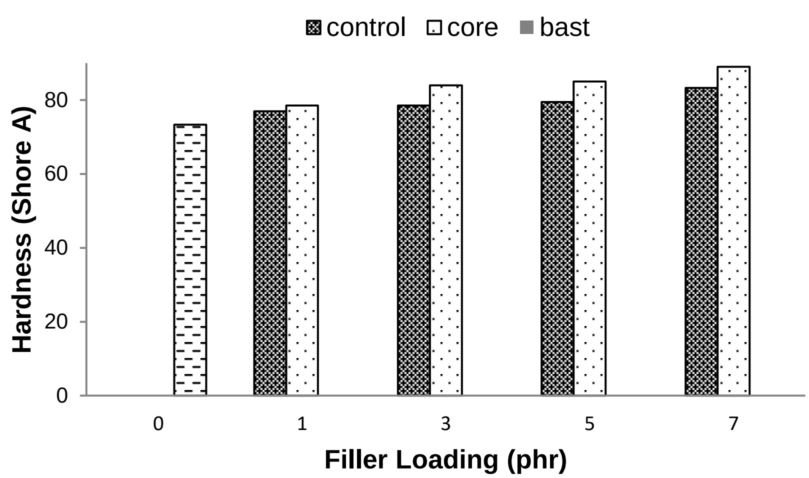

Fig. 8. The effect of filler loading on hardness of kenaf core or bast filled NRLF

Hardness

Figure 8 shows the hardness of the kenaf core or bast filled NRLF at different kenaf loadings. Figure 8 exhibits how increases of loadings resulted in increases of hardness. Addition of filler constrained the NRLF and, therefore, increased the hardness. Higher kenaf loadings caused the filler to form aggregates, which led to the formation of larger agglomerated structures. The mobilization of the filler became a hindrance to increases in the stiffness of NRLF. The flexibility and elasticity of rubber chain decreased as more kenaf was added into NRLF. Figure 8 also shows that the hardness values of the kenaf bast NRLF samples were higher compared to the kenaf core samples; this was likely due to the differences in cellulose content within kenaf core and kenaf bast. The percentage of cellulose in kenaf bast is higher compared to that of kenaf core, as was shown in Table 3. Cellulose is a linear polymer with both crystalline and amorphous regions (Jonoobi *et al*. 2009). Cellulose fibres have a high degree of crystallinity, due to the regular arrangement of its chains, which contributes to the hardness of the compound. Hence, kenaf bast filled NRLF had higher hardness values as the cellulose contents became higher.

Swelling Test

Figure 9 displays the trends of swelling percentage of kenaf-filled NRLF. The swelling percentage of pure NRLF was higher compared to the kenaf core and bast filled NRLF. This might be attributed to the high volume-fraction of polymer that acts as a swelling agent. Thus, the change in swelling of core and bast filled NRLF was a consequence of the presence of filler between the rubber networks. As kenaf loading increased, the swelling percentage decreased. The filler particles can act as obstacles to the ability of toluene to diffuse in rubber. The higher the filler loading in the rubber matrix, the more obstacles there were created and greater reductions of toluene penetration were seen. Figure 9 also shows that kenaf bast filled NRLF exhibited less swelling than did kenaf core filled samples due to the higher network elasticity. These chains restricted the extensibility of the rubber chains (normally induced by swelling), thereby minimizing the diffusivity of toluene between the gaps of rubber molecules and lowering the swelling percentages.

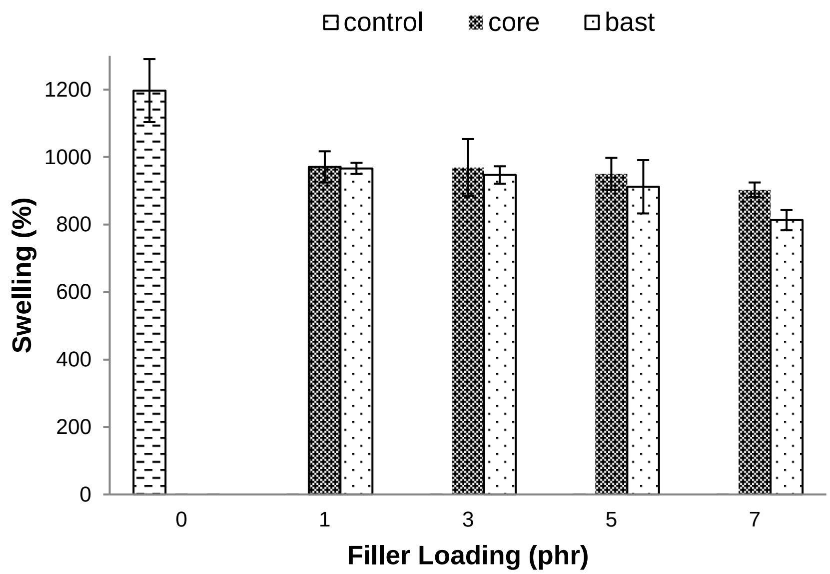

Fig. 9. The swelling percentage of kenaf core or bast filled NRLF

Compression Set Properties

Figure 10 shows the constant deflection compression set values for kenaf core or bast filled NRLF. It is clear that the compression set of pure NRLF was low. However, as kenaf loading increased, the compression set also increased. This was due to the NRLF entrapping filler agglomerates, thereby restricting molecular chains, decreasing elasticity, and increasing stiffness. These changes resulted in more molecular chains that had to be broken and there was a lower number of molecular chains responsible for strain recovery, as is shown in recovery percentage that will be discussed later. As a result, as kenaf loading increased, the compression test values increased but the percentage of recovery values were low. Figure 10 also shows that kenaf bast filled NRLF exhibited higher deflection values compared to the kenaf core values. As discussed previously, kenaf bast has a higher cellulose content, which contains highly regular chains and results in higher stiffness. Figure 11 shows the recovery percentage of kenaf core and bast filled NRLF. As the kenaf loading increased, the percentage of recovery was low. Again, more molecular chains must be broken, resulting in high compression set values but, also, in low recovery percentages.

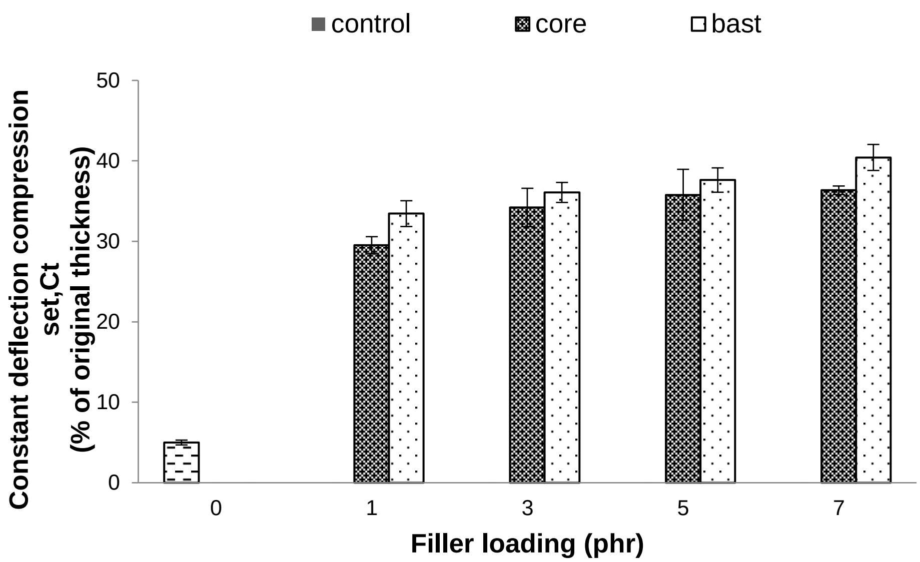

Fig. 10. Constant deflection compression set, Ct of kenaf core or bast filled NRLF

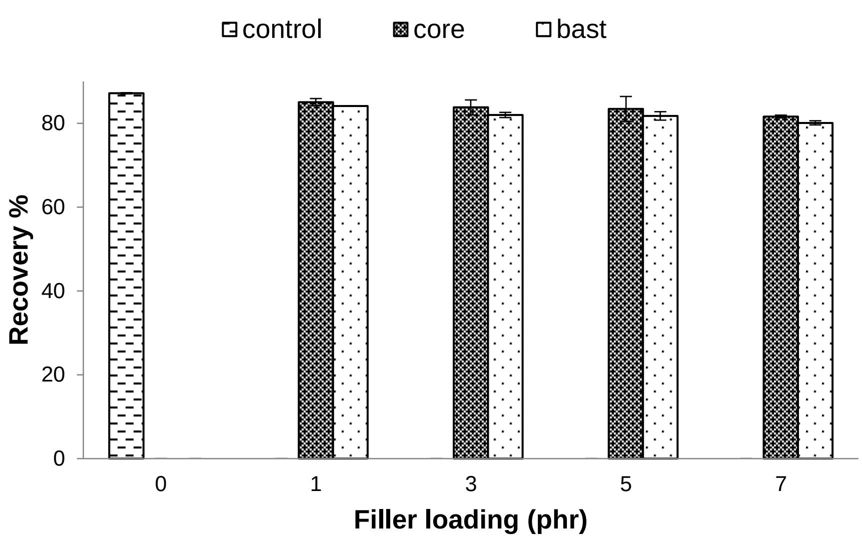

Fig. 11. Recovery percentage of kenaf core or bast filled NRLF

Accelerated Aging

The effect of filler loading and type of kenaf on tensile properties such as tensile strength, elongation at break, and modulus at 100% elongation (M100) of kenaf filled NRLF after aging are shown in Figs. 12 to 14.

Figures 12 and 13 show that the tensile strength and elongation at break of the aged NRLF samples were lower compared to the unaged samples, as all the samples had the same filler loading. This was due to the applied heat, which can break the rubber-filler, filler-filler, and rubber-rubber bonds. Figure 14 presents the results of the modulus at 100% elongation after aging tests. The results show the opposite trend that the modulus at 100% test results showed. The sample presented a higher modulus at 100% after aging for 2 days. This might be due to chain scission of the polymers; the resulting, shorter molecules entangle with one another, causing a reduction of rubber chain mobility. Ooi *et al*. (2013) also reported that M100 values for a rubber compound with oil palm ash, silica, and carbon black increased after aging.

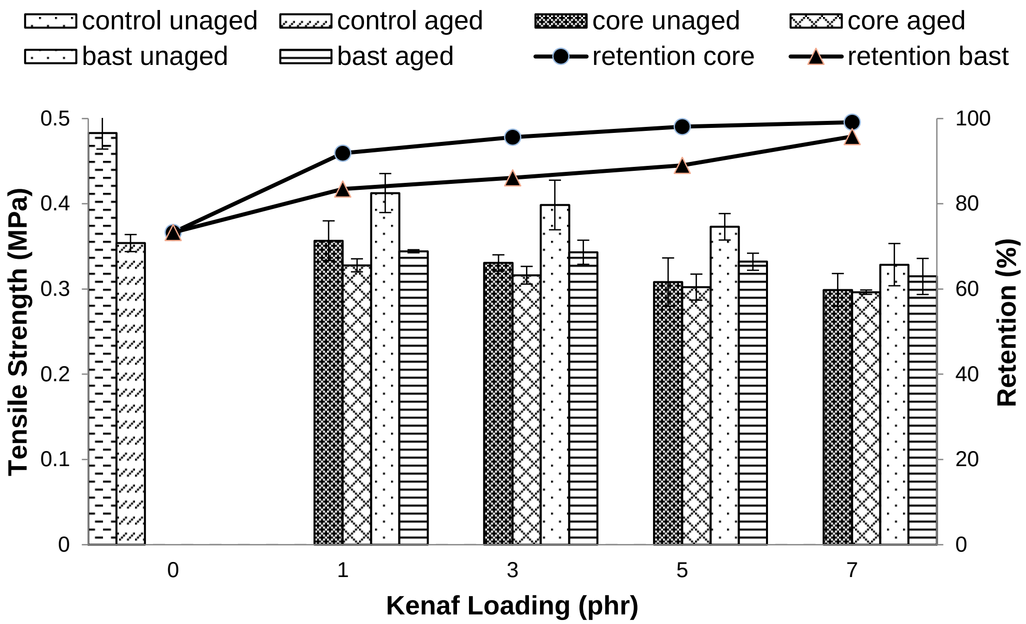

Fig. 12. Effect of filler loading on tensile strength of kenaf core or bast filled NRLF after the aging process

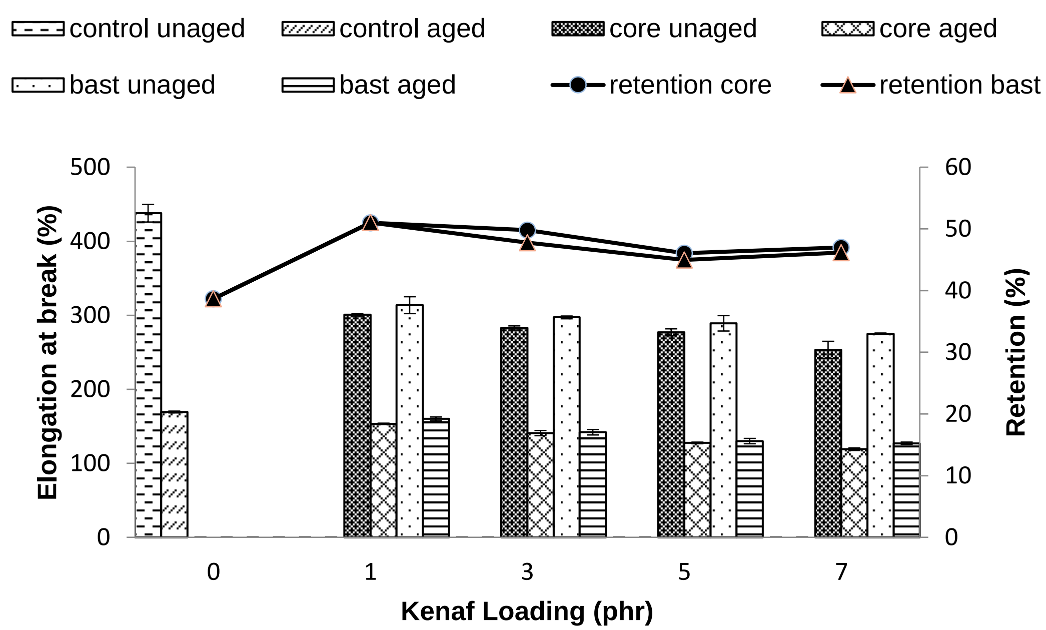

Fig. 13. Effect of filler loading on elongation at break of kenaf core or bast filled NRLF after the aging process

Figures 12, 13, and 14 reveal that the kenaf core filled samples had higher retention percentage compared to the kenaf bast filled samples. The kenaf cores contained a higher percentage of lignin–19.2%–compared to 14.7% lignin content of kenaf bast (Nayeri *et al*. 2013). The structure of lignin is that of three-dimensional polymer possessing structures based on phenyl propane. These phenolic groups prevented the compound from undergoing chain propagation of rubber oxidation.

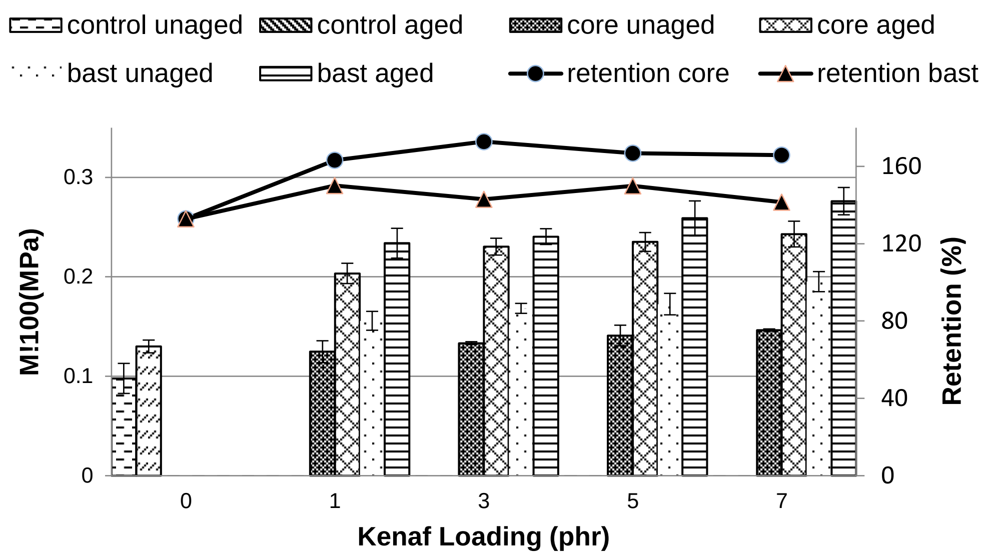

Fig. 14. Effect of filler loading on M100 of kenaf core or bast filled NRLF after the aging process

CONCLUSIONS

1. The compressive properties, hardness, and foam density of the natural rubber latex foam (NRLF) samples increased as the kenaf core or bast loadings inside them were increased. However, doing so decreased the tensile strength, elongation at break, and swelling properties.
2. Kenaf bast had higher values for tensile strength, elongation at break, M_{100}, compressive properties, and hardness compared to the kenaf core, but lower values for foam density and swelling percentage.
3. There were notable differences in morphology of kenaf core and kenaf bast, which affected tensile strength properties, compressive properties, hardness, and foam density. The kenaf core particles tended to agglomerate as filler loading increased. This was this due to the irregular structure possessed by kenaf core. However, kenaf bast filled NRLF exhibited good interactions between filler and rubber due to the fibrous structure of kenaf bast.
4. After thermal aging, both kenaf core or bast filled NRLF samples had lower values of tensile strength and elongation at break compared to the unaged samples. The retention percentage of kenaf core filled NRLF was higher than the kenaf bast filled NRLF.

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