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Effects of Cambodian Clay on the Physical and Mechanical Properties of Natural Rubber Latex Foams

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Abstract: *Natural rubber (NR) is often compounded with inorganic fillers such as clay to broaden its applications, reduce the production cost, and also to impart good mechanical properties. In this study, the effect of local clay on physical and mechanical properties of Cambodian clay-filled natural rubber latex foams was investigated. The natural rubber latex foam was produced via the Dunlop method through the conventional vulcanization system with sulfur as the crosslinking agent. The clay loading was varied at 5, 10, 15, and 20 parts per hundred rubber (phr). The preliminary tests of raw natural rubber latex for pH, alkalinity, total solids content (TSC), dry rubber content (DRC), and the volatile fatty acid number (VFA no.) were carried out according to ASTM standard with all the results comply with the accepted value according to the standard. Clay was characterized for mineral compositions using X-ray fluorescence. The particle size distribution analysis showed that clay has diameter ranging from 0.262 to 19 micrometers, suggesting a classification of semi-reinforcing filler. The results of the physical properties showed a considerable decrease in porosity and swelling while the bulk density, true density, compression set, and hardness were significantly increased. Tensile strength exhibited significant increase at high clay loading similar to the elongation at break, whereas tear strength showed significant improvement at the maximum of 15 phr and start to decrease at 20 phr loading. Thermal aging also demonstrated a significant deteriorating effect on the latex foam due to chain scissions, which reduces mechanical strengths but increases in the hardness value. The results, therefore, validate that clay filler incorporation with NR latex provides some reinforcement on the properties of NR latex foam mostly at high clay loading.*

Keywords: Cambodian clay; Natural rubber; microscomposition; Latex foam

1. INTRODUCTION

Natural rubber (NR) has been globally used for decades to develop a various forms of products application ranging from transportation and construction materials to latex foam and cushions. NR is well recognized for good green strength which enables the NR based products to withhold a high level of deformation during applications [1]. Raw NR can be used for production either in latex form or as dry rubber sheets. One of the common applications derived from latex is foam for cushion applications, which by definition is cellular materials consist of different sizes of interconnecting cells [2]. The process involved in foam production is the Dunlop method which is also known as vulcanization process. During this step, incorporation of fillers in latex is also significant to produce the products that meet the required properties at a cheaper price. Clay, in particular, has displayed outstanding performance as a reinforcing filler in terms of hardness, tensile strength, tear strength, and abrasion resistance [3].

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NR latex derived from the Hevea Brasiliensis, is an elastomeric hydrocarbon polymer consists of chains having a uniform structure called cis-1, 4-polyisoprene that gives NR a strain induced crystallization characteristic associated with excellent mechanical properties of the final products [4]. Commercial raw NR latex undergoes a concentration process to increase the rubber content from 33% to 60% [5] and is preserved with ammonia to stabilize against microorganisms and coagulation [6]. Latex stability is highly significant towards the processing behavior in terms of tolerating the mechanical effects and the storage life during transportation [7]. It is measured by characterization of the chemical properties by aligning with the specifications of the international standard ASTM. Latex is transformed into latex foam and other useful products by vulcanization, a process that converts independent chain molecules of elastomeric materials into a network of crosslinks [8]. In turn, crosslinks give the final products the ability to retract to their original shape after being subjected to mechanical deformation [9]. Crosslinks are accomplished by compounding latex with Sulfur and other chemicals and finished off with heating for vulcanization to proceed. Some examples of the applications of composite foams are noise and vibration absorbing materials, thermal insulation for building construction, and packaging materials. In order to design a rubber foam for a particular improvement in the performance, rubber foam compounding is usually coupled with clay filler. Due to the presence of alumina particles, the addition of clay is excellent for producing products that have good thermal properties, creep resistance, high melting temperature, and high strength [10]. Within the conventional reinforcement system, clay that is added at micrometer size can create an interfacial bonding with rubber chains. At a relatively small particle size, and by well distributed and dispersed in latex matrix [3,11], the bonding creates a polymer-filler interaction that in turn increases crosslink density of the materials hence improving stiffness and modulus. Moreover, clay is an inert material occupying space in the matrix as rigid filler particles, which further attributes to toughening foam materials and overcoming poor processability, thus reducing production cost [12,13].

In Cambodia, NR and clay are two of the abundant natural resources that have limited applications. NR generates revenue only from exporting as raw dry rubber while clay, which is most available in Kampong Chhnang province is used to make pottery products. The current study, therefore, potentially contributes to technical capacity building and attracting future industrial investment. This study aims to understand the characteristics of clay filled latex foam by producing samples according to Dunlop method then conducting series of physical and mechanical tests.

2. METHODOLOGY

2.1 Materials and formulation

Concentrated latex was purchased from Rubber Authority Thailand. Clay was taken from Prey Khmer Village, Kampong Chhnang Province, Cambodia. Sulfur (97%) was supplied by Fisher Chemical, UK, phenolic type antioxidant (IONOL^R LC) was supplied by Rasching GmbH, Germany. Zinc 2-mercaptobenzhiozolate (ZMBT) was purchased from OSHA, Japan. Zinc oxide (ZnO, 99%) was supplied by Merch KGaA, Netherlands, 1,3- diphenylguanidine (DPG, 97%) was purchased from Sigma, China and Sodium Silicofluoride (SSF), was bought from HIMEDIA^R, India. Oleic acid 65% was purchased from Applichen, Germany. Potassium Hydroxide 90% was supplied by UNID, Korea. All reagents and formulation for compounding is presented in Table 1.

All reagents were made into solutions of specific concentrations by mixing with water and ball mill (ASH, AV-2) for 24h. The potassium oleate soap (PO) was prepared by mixing Oleic acid 100g with distilled water (402ml) to make a solution A. Then Potassium Hydroxide 23g was dissolved into 43ml of distilled water to produce a solution B. Solution A was put on a hot plate until it reached the temperature 75 ºC, then the solution B was poured in while stirring was set at high speed and the mixture was stirred until clear brown gel is obtained [2]. Clay powder was prepared according to the ASTM D 2216 by drying in oven at 105 ºC for 24h then grinded and sieved for diameter under 75 microns. The powder was dried for 24h and subjected to dry milling for 24h to further reduce the particle size for better dispersion in latex.

Table 1 Compounding formulation for NRL/clay foam

Reagents	Concentration (%)	Formulation (phr)
NR latex	60	100
P.O	20	2
Sulfur	35	2.5
Antioxidant	22	1
Clay powder	-	*
ZMBT	35	1
ZDEC	50	1
DPG	40	0.3
ZnO	50	3
SSF	25	1

*Clay powder was added by variation of 0, 5, 10, 15, 20 phr for properties comparison.

2.2 Foam compounding process

The compounding process is shown in Fig. 1, following the Dunlop method, NR latex was stirred for 30 mins at 200 rpm (IKA Eurostar 20), to drive off some ammonia. Sulfur, P.O, and antioxidant were then added and stirred for 30 mins at 250 rpm while clay powder was added and ensured to well disperse in the mixture. Next, ZMBT and ZDEC were added while stirring for another 30 mins. The compound was beaten with hand mixer (Starlux SL-135) until its initial volume increased 3 times, where DPG was added, continued beating for 90s and finished off with addition of ZnO dispersion and SSF for 90s. The compound was immediately transferred into metal molds, allowed to gel for 3 mins then put in the oven (Advantec DRM620DB) at 105 °C for 2h. The cured latex foam was stripped of the mold, washed with tap water and continue to dry for 2h at 105 °C.

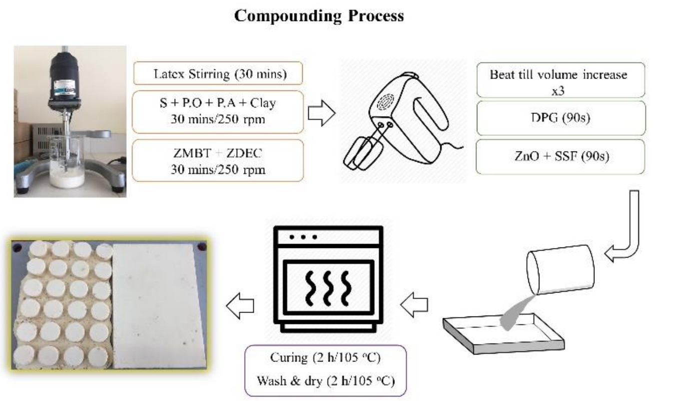

Fig. 1. Latex foam compounding process

2.3 Latex chemical analysis

The chemical properties, pH, total solids content (TSC), dry rubber content (DRC), total alkalinity, and volatile fatty acids (VFA) were analyzed on the concentrated latex according to the ASTM D 1076.

2.4 Characterization of clay sample

The chemical composition of clay was analyzed using X-ray Fluorescene Spectrometer (Rigaku ZSX PrimusIII). About 5 g of clay powder was pressed following the method of flat type dies as described by Gakuto [14]. The particle size distribution was analyzed using Particle Analyzer (SALD 2300). The pretreatment was done following Kroetch & Wang [15].

2.5 Porosity and density measurement

By following the ASTM C 830, cylindrical shaped samples were weighted for dry weight (D) and placed in a vacuum-pressure vessel of about -0.1 MPa while submerged in absolute ethanol (density = 0.789 g/cm^3) overnight. The pressure was released and the saturated samples were taken out, weighted for suspended weight (S) and then saturated weight (W). Porosity can be calculated by Eq. 1. Bulk density and true density are calculated using the equation Eq. 2 and Eq. 3, respectively.

$$P, \% = (V_{void}/V_{total})] \times 100 \qquad \text{(Eq. 1)}$$

where:

P = porosity (%)

V_{void} = the volume of open pores presented in foam sample calculated by $V_{void} = (W - D)/0.789$ (cm^3)

V_{total} = the total volume obtained by $V_{total} = (W - S)/0.789$ (cm^3)

$$\text{Bulk density} = D/ V_{total}\ (\text{g/cm}^3) \qquad \text{(Eq. 2)}$$

where:

D= the dry weight (g)

V_{total} = the total volume obtained by $V_{total} = (W - S)/0.789$ (cm^3)

$$\text{True density} = D/V_{solid}\ (\text{g/cm}^3) \qquad \text{(Eq. 3)}$$

where:

D= the dry weight (g)

V_{solid} = volume of solid component in sample calculated by $V_{solid} = (D - S)/0.789$ (cm^3)

2.6 Swelling test

Test specimen was cut from foam sample in dimension of 30 × 9 × 5 mm and weighted for M_1, then immersed in 99.99% toluene for 72 hours. The swollen specimen was taken out, gently wiped with tissue, and weighted for M_2 [16]. Percentage of swelling by change in mass is calculated following the ASTM D 471 as shown in Eq. 4.

$$\text{Swelling (\%)} = (M_2 - M_1)/M_1 \times 100 \qquad \text{(Eq. 4)}$$

where:

M_1 = the initial weight of the sample (g)

M_2 = the weight after immersion (g)

2.7 Aging test

Aging test was carried out based on the ASTM D 573. Foam samples were placed in the air oven (Advantec DRM620BD) at 105 °C for 24 h after which they were subjected to properties tests including hardness, tensile properties, and tear strength. The obtained data were then compared with the unaged samples.

2.8 Hardness

Durometer Shore A was used to measure the sample hardness according to ASTM D 2240. The presser foot of the durometer was vertically pressed to each sample at five different positions and the unit value shown on the screen was recorded and the average was calculated.

2.9 Compression set

Cylindrical shaped samples were measured of the original thickness and placed between the plates of the compressive device detailed in ASTM D 395-Test Method B, with spacer bars on each side. The plates were bolts tightened, put in air oven at temperature 105 °C for 24 h. The test samples were removed, allowed to cool off for 30 mins and the final thickness was measured. The compression set is calculated as defined in Eq. 5.

$$C_B = [(t_0 - t_i)/(t_0 - t_n)] \times 100 \qquad \text{(Eq. 5)}$$

where:

C_B = compression set expressed as percentage of the original deflection (%)
t_0 = original thickness of specimen (mm)
t_i = final thickness of specimen (mm)
t_n = thickness of the spacer bar, 9.5 mm

2.10 Tensile properties

Tensometer ETS Interlaken technology was used to perform tensile properties including tensile strength and elongation at break. Dumbbell (die C) samples were cut from the foam sheet and measured at crosshead rate of 500 mm/min according to ASTM D 412.

2.11 Tear test

Tear test was performed according to ASTM D 624, on tensometer ETS Interlaken technology. Samples were cut into trouser-shaped pieces and tested at crosshead rate 100 mm/min.

2.12 Statistical analysis

Analytical results were compiled using Microsoft EXCEL 2016 program to express standard deviation and single-factor ANOVA. The significance threshold was set at .05. All graphs were plot in OriginPro 2018.

3 RESULTS AND DISCUSSION

3.1 Chemical properties of rubber latex

Table 2 Chemical properties value of NR latex

Chemical Properties	Common samples	Studied samples
pH	10.8-11.8	10.06
Total Alkalinity	0.60 min	0.68 (as NH_3) %
Total Solids	61.5	62%
DRC	60.0	62%
VFA N°	0.06	0.016

The data in Table 2 illustrates that the NR latex used has chemical properties value range within the standard with pH value of 10.06, total alkalinity of 0.68 as percentage of ammonia, and the volatile fatty acids number of 0.016. The total solid content and dry rubber content both have the value of 62% indicating that the latex was of high purity and can be stored for a long period of time.

3.2 Clay sample characteristics

The chemical compositions of clay are shown in Table 3. The dominant compounds are silicon dioxide (SiO_2) or silica and aluminum oxide (Al_2O_3) at 54% and 26.92% w/w, respectively.

Table 3 Chemical composition of clay sample

Compositions	Weight (%)
SiO_2	54.3178
Al_2O_3	26.9291
Fe_2O_3	7.0012
K_2O	1.5737
TiO_2	0.6244
CaO	0.5697
MgO	0.4397
Na_2O	0.0941
ZrO_2	0.0408
Loss on ignition	8.2600

Particle size distribution analysis suggested that clay diameter ranged between 0.269 to 19 microns. Based on previous reviews on the particle size of clay filler for natural rubber composite [17,18], this clay sample falls between the category of a diluent, which fill pores of the latex foam resulted in decrease in porosity and a semi-reinforcing filler, which could partially improve the strength and modulus of the foam.

3.3 Porosity and density

Fig. 2 shows the value of porosity and bulk density of NR latex foam in function of clay filler loading from 0 to 20 phr. The porosity of samples dramatically dropped in contrast to the bulk density, which continuously went up in respect to the clay loading.

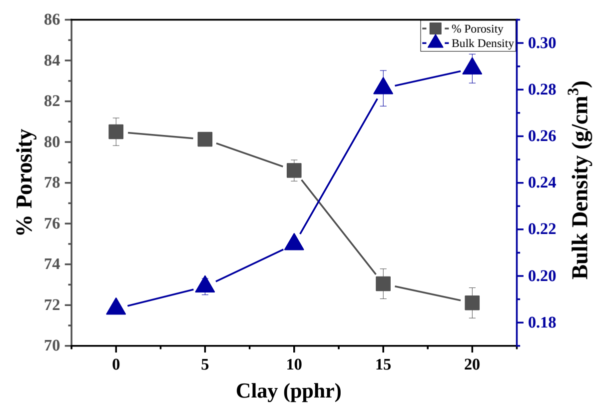

Fig. 2. Porosity and bulk density of clay-filled latex foam.

The statistical analysis suggested a significant change at clay loading of 10 to 15 phr for porosity and at every 5 phr increment for bulk density except for the highest concentration. Similar to the other studies [19,20], a high amount of filler content could reduce pore formation in foam samples, therefore reduces the porosity and increases significantly the bulk density.

The true density illustrated in Fig. 3 exhibits an upward trend with a significant improvement at clay loading of 15 and 20 phr. The value aligns with the bulk density and corresponds to the significant decrease of porosity at high clay concentration, altogether indicating that the composite foams could be stiffer by filler addition.

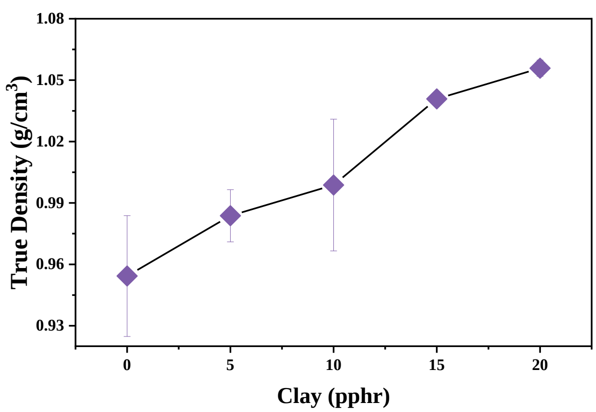

Fig. 3. True density of clay-filled latex foam

3.4 Swelling

Fig. 4 shows a steady decline in swelling percentage as clay loading increases. Each clay increments significantly affect the swelling behavior possibly because of the filler-polymer interaction. This interaction could further enhance the crosslink density of the latex foam in addition to the chemical crosslinks between rubber chains by vulcanization. Similar to the studies reported by [21-23], this enhancement could restrict the diffusion of solvent molecules through foam pores thus reduces the swelling percentage.

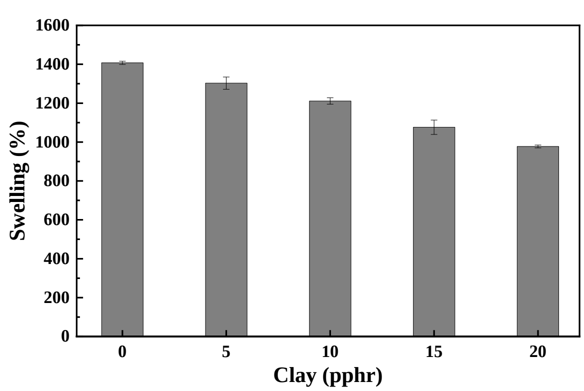

Fig. 4. Swelling percentage of clay-filled latex foam

3.5 Hardness

The effect of clay addition on hardness of NR latex foam before and after aging is presented in Fig. 5. The graph demonstrates that the hardness of unaged foams increased almost double with a significant difference for each 5 phr addition of clay except for 15 to 20 phr, similar to the results of porosity and true density. In line with the improved density and lower swelling percentage, the effect of clay incorporation favors the hardness properties of composite foams. This supports that the presence of polymer-filler network could increase the degree of crosslink density, resulting in materials having a more rigid characteristic [24]. This effect was found in similar studies on clay filler interaction with natural rubber such as those reported by Kader & Deeb [24] and Ewulonu et al. [25].

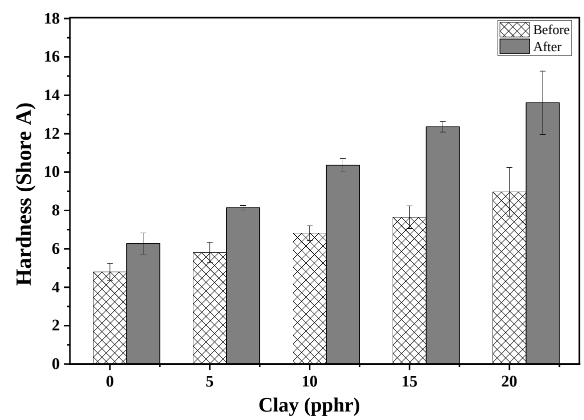

Fig. 5. Hardness of clay-filled latex foam

The hardness of NR latex foam sample after aging can be observed to have greater value than the unaged and also increased in respect to clay addition. The high value could be influenced by thermal aging that resulted in the term ‘post curing’ effect causing chain scission in rubber chains, breakage of polysulfidic crosslinks from conventional sulfur vulcanization system into mono and di-sulfidic types, responsible for reduction of modulus and elasticity yet favor the stiffness properties of foam [26].

3.6 Compression set

Fig. 6 illustrates a gradual rise in compression set with the clay filler loading. Clay addition at 10 to 20 phr was found to have a significant effect on the NR latex foam indicating a reduction of recovery rate after being subjected to constant deflection over a specified time. The crosslink density enhanced by filler interaction could decrease rubber chain mobility, inducing stiffness, and lessen elastic contribution to recover back to its original thickness which indicated the NR latex foam has been set permanently by the compression force [25,27].

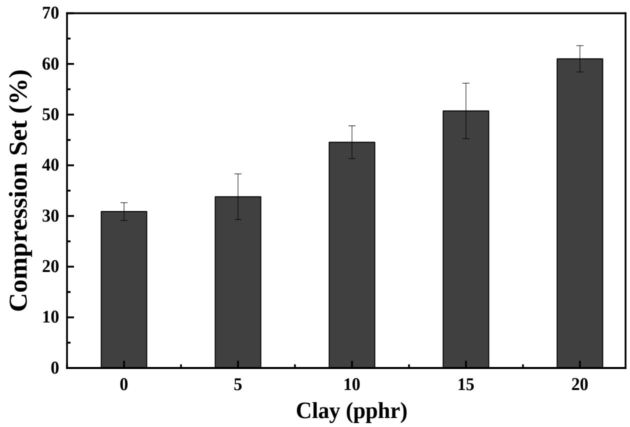

Fig. 6. Compression set of clay-filled latex foam

3.7 Tensile properties

From Fig. 7, the overall tensile strength of unaged foam shows an improvement with the addition of clay loading. The statistical analysis confirms a significant effect at the first 5 phr addition and when clay increased up to 20 phr, indicating a similar trend as other properties at high clay loading. The improvement suggests an interfacial bonding enhancement between latex and clay filler, attained by well dispersion and distribution of clay in the compound due to its relatively small particle size [25]. Moreover, the reinforcing effect of clay filler on such mechanical properties has been remarked as a co-reinforcement system. The conventional sulfur vulcanization method applied on natural rubber in addition to its unique nature has already provided the NR latex with a primary mechanical reinforcing effect i.e the ability to crystallize upon straining [3]. With the incorporation of clay, this ability received another reinforcement thus an extensive increase in tensile strength is observed [28]. Thermal aging shows a significant effect on the tensile strength of NR latex foams, breaking the crosslinks and lowering the value by almost half compared to the properties before aging.

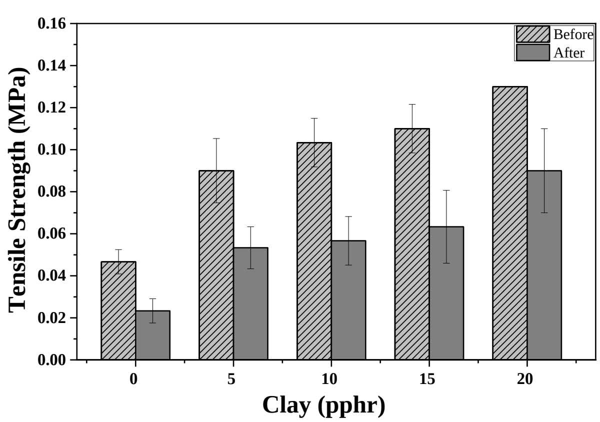

Fig. 7. Tensile strength of clay-filled latex foam

Fig. 8 demonstrates a significant increase on elongation at break at the highest clay concentration. This is due to the dual reinforcement as described previously in which the influence of rubber-filler interactions from the effect of high clay filler content [29] was possibly reinforced in terms of its aligning and crystallizing characteristics upon straining and perhaps promote the stretching ability of the NR latex foam. An increase in elongation at break from about 10 pphr of clay loading in rubber compound was also reported by [24]. Thermal aging also had a great deteriorating effect on elongation at break.

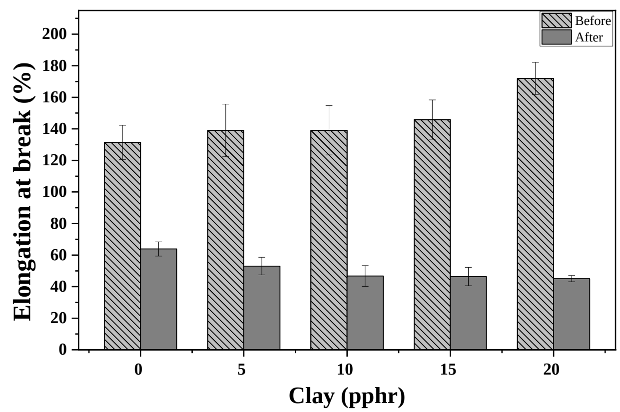

Fig. 8. Elongation at break of clay-filled latex foam

3.8 Tear strength

The graph of tear strength from Fig. 9 initially showed an increase with the addition of clay but dropped significantly at the highest clay loading. Upon applied force, the double reinforcement effects from the filler and latex crystallization could produce local reinforcement of strain-induced at the crack tip resulted in increased crack growth resistance hence higher tear strength.

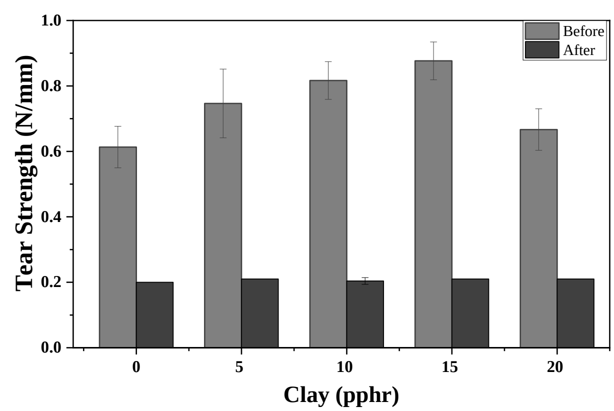

Fig. 9. Tear strength of clay-filled latex foam

On the other hand, the decline at clay 20 phr could indicate the formation of filler-filler networks, which only interferes with the mechanical performance of composites [21,30]. As a result of crosslink breakage and loss of elasticity, the composites of all loading showed no crack resistance to tearing force after aging, which indicates loss of the ability of strain induced crystallization of NR latex [31].

4 CONCLUSIONS

The compounding process of natural rubber latex/clay foam was carried out by incorporating micro-size clay filler via the Dunlop method. Chemical properties of raw latex and clay sample characterization were conducted. The presence of clay filler decreased porosity and swelling percentage but increased compression set at high clay loading up to 20 phr. It increases bulk and true density thus, significantly improved hardness except at the highest concentration. On the other hand, the tensile strength and elongation at break were considerably increased at high clay loadings, while tear strength peaked at 15 phr. The data indicates that clay filler indeed influenced the properties of clay-filled NR latex foams as a result of the polymer-filler interaction that induced a co-reinforcement system with natural rubber.

For future research, the obtimization of clay loadings in latex foam for a specific application should be carried out. Microscopy analysis such as Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) image analysis should also be conducted in order to understand in-depth the dispersion of filler in composite foam. Also, the incorporation of antibacterial and flame-retardant agents is encouraged.

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