

## Cure and Physical Characterizations of Natural Rubber Blended with Recycled Latex-Foam-Waste

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**ABSTRACT:** Recycling of an industrial latex scrap, latex-foam-waste (LFW) into a natural rubber compound (RLF-0) used to produce nonblack rubber mat in industry was investigated. A masterbatch (LFW-MB), comprised the LFW and styrene butadiene rubber (SBR) blend was pre-prepared through a prolonged mixing before it was blended in different levels of mixes. Characterizations on cure characteristics, specific gravity, hardness, crosslink density, mechanical properties and scanning electron microscope investigations were performed. The values for the onset of cure ( $T_2$ ), the optimal cure time ( $T_{90}$ ) and specific gravity (s.g.) were reduced when the added dosages of LFW-MB into RLF-0 were increased. These can provide an economic advantage to industry. Although the modulus and the hardness of rubbers were decreased when the loadings of LFW-MB were increased due to the reduction of crosslink density, the mechanical properties measured on elongation at break (EB) and tensile strength (TS) were enhanced particularly at the loading level of 65 part per hundred rubber (phr). The elastomeric LFW-MB was able to distribute and interact well across the rubber matrix. The multiple-stage-prolonged mixing through the preparation of masterbatch is an effective and practical method to recycle LFW.

**Keywords:** natural rubber, styrene butadiene rubber, latex-foam-waste, rubber masterbatch, prolonged mixing

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### I. INTRODUCTION

Recycling of rubber wastes has constantly created serious attentions worldwide as the increases of these wastes have posed negative impacts to environment. Rubber wastes are usually land-filled, and the efforts on partial or full recycling are able to reduce the depletions of natural resources and save the landfill space [1]. Numerous researches have been investigated on the approaches of rubber wastes recycling mainly focused on the end-of-life tires (ELTs) including the risk of emissions of hazardous chemicals [2], incorporations of recycled rubber crumbs as composites into concrete material [3, 4], asphalt mixture [5, 6] and insight on pyrolytic carbon black [7]. The recycling and incorporation of rubber wastes into composites usually require the preparations of rubber crumbs prior to the final mixing process. Rubber crumb is effective in dissipating impact energy when it is used in composite preparation [3]. The downsized and crosslinked rubber crumb aggregates can undergo large elastic deformation and effectively dissipate the energy whilst maintaining excellent resistances to fracture and abrasion.

Although there were many researchers studied the recycling of ELTs, the recycling of latex-foam-waste (LFW) has not been examined thoroughly. Rubber foams have wide range of applications particularly to serve as acoustic absorbing and vibration damping materials [8]. The present research investigated the recycling of an industrially generated LFW into an industrial compound using an industrially practical prolonged mixing method. The prolonged mixing through the technique of masterbatch preparation on the targeted ingredient is a convincing method to effectively disperse and distribute the ingredient in rubber compound as reported by many researchers [9, 10, 11, 12] without profoundly reducing the overall rubber molecular weight.

## II. MATERIALS AND METHODS

### 2.1 Materials

Natural rubber (NR) L-grade was supplied by Hockson Rubber Trading. Styrene butadiene rubber (SBR) (Intol 1502) with Mooney viscosity 52 and s.g.  $0.94 \text{ g/cm}^3$  was supplied by Polimeri Europa UK Ltd. Latex foam waste (LFW) with s.g.  $1.25 \text{ g/cm}^3$  was obtained from an industrial production scrap. Its breakdown ingredients in dry weight part per hundred rubber is depicted in Table 1.

**Table 1:** Dry weight ingredient breakdown of LFW

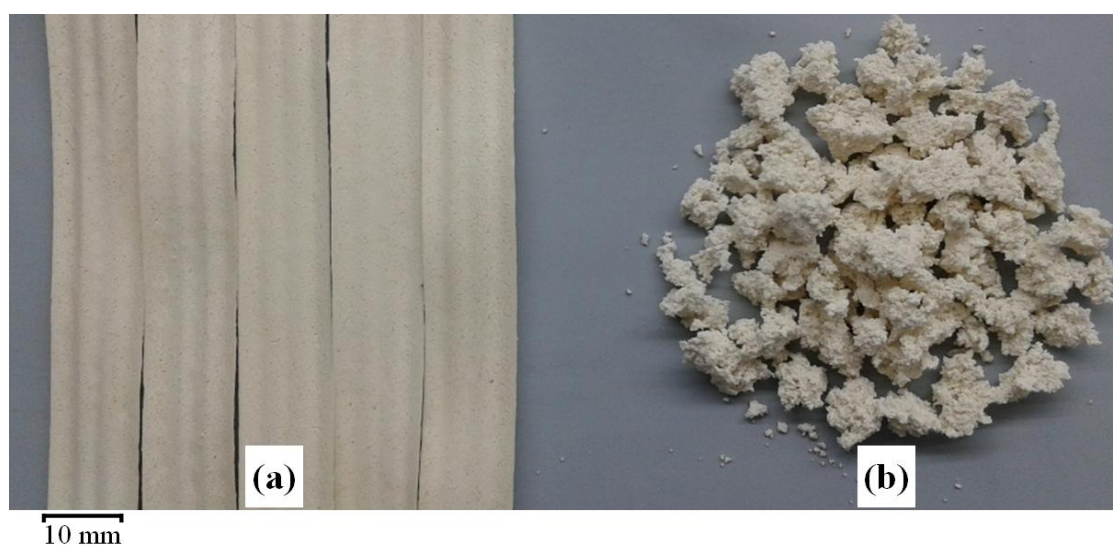
Ingredient	phr*
NR	100.00
Potassium hydroxide	0.28
Aluminium hydroxide	1.54
Oleic acid	1.75
Dispersing agent	0.16
Sodium montmorillonite	0.79
Phenolic antioxidant	1.10
UV absorber	0.63
Calcium carbonate	18.06
Titanium dioxide	8.94
Polyvinyl alcohol	11.42
Zinc oxide white	2.35
Accelerator – ZDBP	0.47
Accelerator – ZMBT	0.94
Accelerator – ZDBC	0.31
Sulphur	2.04

\* part per hundred rubber

Other rubber ingredients of zinc oxide (ZnO), calcium carbonate ( $\text{CaCO}_3$ ), stearic acid, polyethylene glycol (PEG), phenolic antioxidant, paraffinic oil, mercaptobenzothiazole disulfide (MBTS), diphenyl guanidine (DPG) and sulphur were the grades of customarily used in rubber industry.

### 2.2 Formulations and mixing

A recycling rubber masterbatch (LFW-MB) of blended SBR and LFW at a ratio of SBR:LFW = 1:3 was prepared through prolonged melt mixing at 45 minutes by using a laboratory two-roll-mill (Radicon) of friction ratio at 1.25 and temperature at  $40\text{-}50^\circ\text{C}$ . A crumbly and foamy LFW-MB was produced. It is different from the conventional tire recycling crumbs in distinctive aggregates and its specific size was unquantifiable. Fig. 1 shows the physical LFW and the downsized LFW-MB.



**Fig. 1:** Latex-foam-waste (LFW) [a] and mixed LFW masterbatch (LFW-MB) [b]

A main compound (RLF-0), referred to an industrial nonblack rubber mat formulation as shown in Table 2 was prepared by using a two wings tangential 110 litre internal mixer (X(S)N-113/30) from Dalian ChengXin Machinery Co. Ltd., China with a friction ratio of 1.22 at temperature 90 °C and 10 minutes mixing time. Subsequently, five different ratios of RLF-0 and LFW-MB were blended by using the two-roll-mill at temperature 40 °C and 10 minutes mixing time. The RLF-0 was prepared as a control sample. The final formulations are listed in Table 2.

**Table 2:** Formulations of blended LFW-MB

Ingredient	RLF-0 (phr)	RLF-5 (phr)	RLF-25 (phr)	RLF-45 (phr)	RLF-65 (phr)	RLF-85 (phr)
Natural rubber	100.00	100.00	100.00	100.00	100.00	100.00
Calcium carbonate	75.00	75.00	75.00	75.00	75.00	75.00
Phenolic antioxidant	2.50	2.50	2.50	2.50	2.50	2.50
Paraffinic oil	18.00	18.00	18.00	18.00	18.00	18.00
Zinc oxide white	6.00	6.00	6.00	6.00	6.00	6.00
Stearic acid	3.00	3.00	3.00	3.00	3.00	3.00
Polyethylene glycol	1.50	1.50	1.50	1.50	1.50	1.50
Accelerator – DPG	0.60	0.60	0.60	0.60	0.60	0.60
Accelerator – MBTS	6.00	6.00	6.00	6.00	6.00	6.00
LFW-MB	-	5.00	25.00	45.00	65.00	85.00

Three replications for each sample were then added with 2 phr of sulphur after they were rested for 24 hours at ambient temperature (24-28 °C). The multiple-stage-mixing method was aimed to effectively break the LFW into fine fragments.

### 2.3 Characterizations of compounds and vulcanizates

Cure characteristics of compounds were determined by using an oscillating rotorless rheometer (UR2010) from U-CAN Dynatex Inc., Taiwan at temperature 155 °C, pressure at 4.5 kg/cm<sup>2</sup> and test time at 240 sec. in compliance with ASTM D5289. The cure characteristics on scorch time (T<sub>2</sub>), 90 % cure time (T<sub>90</sub>), minimum torque (M<sub>1</sub>), maximum torque (M<sub>H</sub>) and cure rate (C<sub>R</sub>) were recorded. C<sub>R</sub>, is given as Equation 1 [13]:

$$C_R = \frac{M_{90} - M_2}{T_{90} - T_2} \quad (1)$$

where M<sub>90</sub> and M<sub>2</sub> is the torques at T<sub>90</sub> and T<sub>2</sub> respectively. All compounds were cured by using a 100-tonnage hydraulic compression press at 155 °C and cure times referred to the values of T<sub>90</sub> obtained from the rheometry. Hardness test was performed by using a hardness meter (Bareiss HPE II) on disc specimens measured at diameter 20 mm and height 10 mm according to ASTM D2240. Tensile test was performed by using an universal testing machine (Shimadzu EZ-500NLX) on 0.55 mm thick samples according to ASTM D412. Density measurement was determined by weighing the samples in air and water referring to ASTM D792. Solvent penetration method was used to determine crosslink density. The samples of dimension 30mm×20mm×0.55mm were immersed in 100 ml toluene (density 0.865 gml<sup>-1</sup>, boiling point 110 °C) for 5 consecutive days at room temperature to reach rubber-solvent equilibrium. The molecular weight of polymer between crosslinks was determined by using the Flory-Rehner Equation as Equation 2 [13]:

$$M_c = \frac{-\rho_r V_s (\phi_r^{\frac{1}{3}} - \frac{\phi_r}{2})}{\ln(1 - \phi_r) + \phi_r + \chi \phi_r^2} \quad (2)$$

where  $\rho_r$  is the density of rubber,  $V_s$  is the molar volume of toluene determined at 106.52 cm<sup>3</sup> mol<sup>-1</sup>,  $\Phi_r$  is the volume fraction of rubber in the swollen gel and  $\chi$  is the Huggins polymer-solvent interaction parameter fixed at 0.3795. The crosslink density was then determined by  $1/(2M_c)$ . Scanning electron microscope (SEM) was performed on the tensile fracture surfaces of RLF-0 and RLF-65 by using a Hitachi SUI510 SEM machine at accelerating voltage of 15 kV and at 1000x magnification. The samples were gold sputter coated to prevent electrostatic charging.

### III. RESULTS AND DISCUSSIONS

#### 3.1 Cure characteristics

The values of  $T_2$  or scorch time, which represent the time lapse between the initial heating and onset of crosslinking for rubber compounds continuously decreased from the control sample at 96 sec. until 63 sec. for the 85 phr LFW-MB-added sample or represented a 34.4 % reduction as depicted in Table 3.

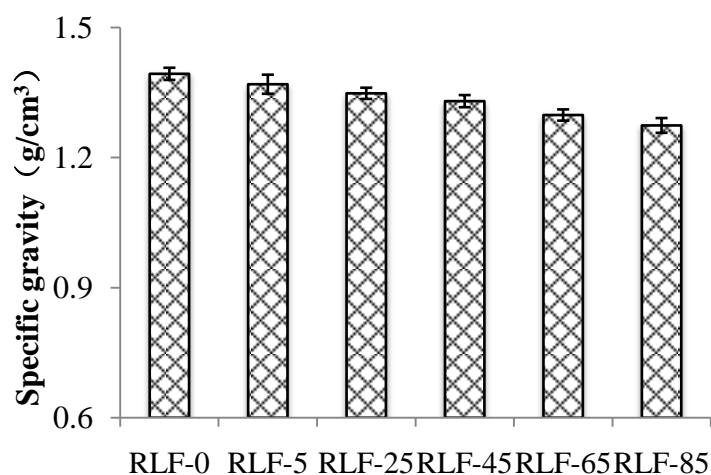
**Table 3:** Cure characteristics of compounds

Sample	$T_2$ (sec.)	$T_{90}$ (sec.)	$M_L$ (dNm)	$M_H$ (dNm)	$C_R$ (dNmsec. <sup>-1</sup> )
RLF-0	96 ± 5.57	137 ± 8.51	3.48 ± 0.58	22.11 ± 1.19	0.3502 ± 0.02
RLF-5	91 ± 3.61	136 ± 2.52	3.84 ± 0.19	24.78 ± 2.78	0.3682 ± 0.04
RLF-25	80 ± 2.00	120 ± 3.79	3.85 ± 0.41	25.99 ± 2.89	0.4335 ± 0.03
RLF-45	73 ± 0.58	110 ± 2.89	4.86 ± 0.32	28.02 ± 1.25	0.4894 ± 0.05
RLF-65	67 ± 1.16	101 ± 0.58	4.89 ± 0.38	25.56 ± 0.73	0.4651 ± 0.03
RLF-85	63 ± 2.00	98 ± 3.51	4.95 ± 0.49	25.94 ± 0.63	0.4670 ± 0.05

A similar reduction trend was recorded for optimal cure time ( $T_{90}$ ) when increasing in LFW-MB loadings. The observations supported the migration of unreacted curatives belonged to the recycled latex foam into rubber chains which induced sulphidic curing. The similar cure acceleration due to the migration of curatives especially the cure accelerators from recycled rubber was also revealed by other researchers [14, 15, 16]. Moreover, the viscosity of compounds was significantly increased supported by higher values of  $M_L$  when increasing in LFW-MB due to the dispersed LFW-MB fragments that restricted the mobility of rubber chains. The cured rubber waste acted like filler which can increase the compound viscosity and transfer it to higher  $M_L$  as reported in other works [15, 16, 17]. Therefore, the increase in rubber viscosity after adding of LFW-MB has paved to higher mixing shear and it reduced both  $T_2$  and  $T_{90}$ . Cure rate ( $C_R$ ) values proportionally increased with the loadings of LFW-MB until 45 phr and marginally reduced by ~5 % at higher levels which may be attributed to the cure dilution of additional rubber from the blended SBR in LFW-MB. Apparently, adding of LFW-MB can reduce the cure time of rubber, which is advantageous in improving the production throughput.

#### 3.2 Specific gravity

Specific gravity values of samples were apparently decreased when the loadings of LFW-MB were increased as depicted in Fig. 2.

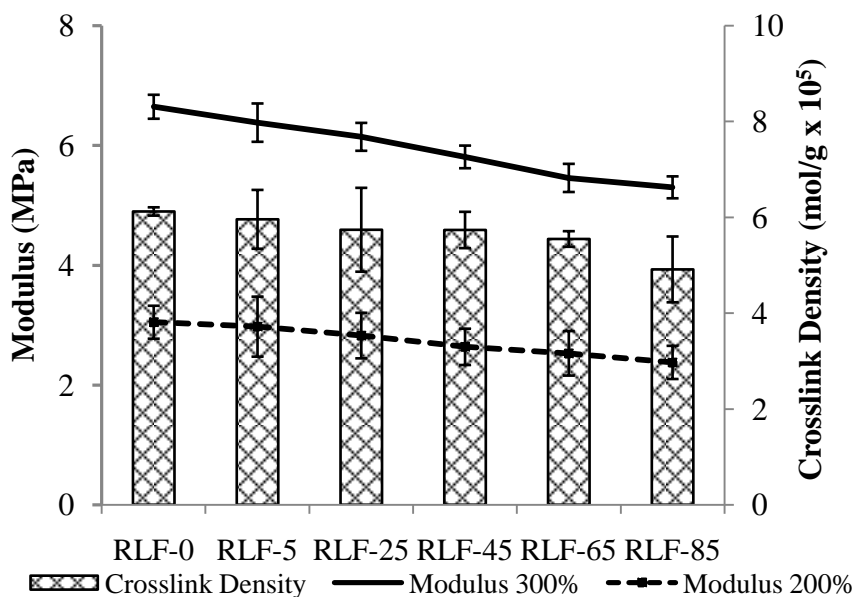


**Fig.2:** Specific gravity of vulcanizates

The inclusion of LFW-MB is dissimilar from the adding of rigid fillers in which the latter can increase the specific gravity of vulcanized rubber. The cost reduction of rubber compounds is usually realized by adding cheap fillers particularly  $\text{CaCO}_3$  [18, 19]. However, the usual practices by adding large specific gravity of inorganic fillers reduce the volume of rubber compounds. Nevertheless, when loading LFW-MB into a similar compound at an equal dosage, it is able to achieve lesser material usage in occupying a fixed volume of mould and this can directly reduce the material cost per product.

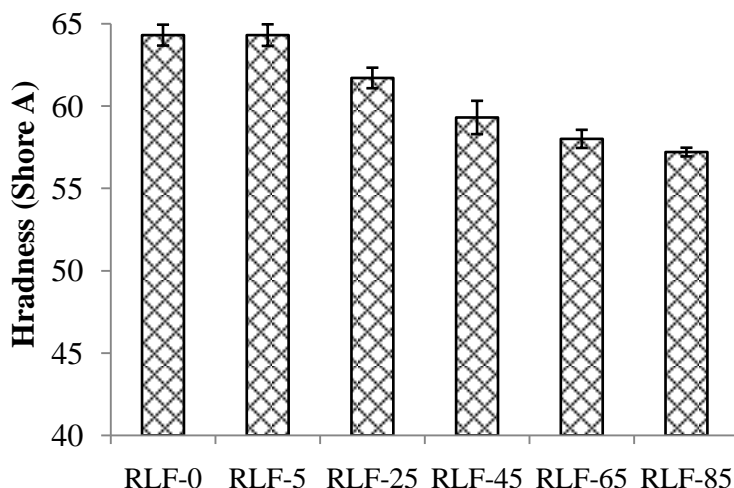
**3.3 Modulus, crosslink density and hardness**

The modulus and crosslink density of vulcanizates decreased when increasing in the loadings of LFW-MB as presented in Fig. 3.



**Fig. 3:** Results of modulus 200%, modulus 300% and crosslink density

Due to the modulus of rubber is always interrelated with its crosslink density [20, 21, 22], the decreases of modulus herein can be explained by the reductions of crosslink density when the contents of LFW-MB were increased. It is attributed to the fact that the rubber of lower crosslink density is more flexible and easier in chains mobility. The LFW-MB consists of 25 % fresh SBR and any increase of its content can lead to the increase of rubber system relatively to a level of curatives and this reduced the density of sulphidic crosslinks across the rubber chains. Particularly for RLF-85, adding of 85 phr LFW-MB appreciably reduced the crosslink density by 19.8 % against RLF-0. Hardness values of vulcanizates diminished in corresponding with the increased contents of LFW-MB by referring to Fig. 4. This observation is anticipated as the degree of rubber hardness changes in-line with the level of crosslink density [12, 21, 23, 24] and modulus [24, 25].



**Fig. 4:** Hardness of vulcanizates

**3.4 Mechanical Properties**

Mechanical properties of samples on elongation at break (EB) and tensile strength (TS) were enhanced in a proportion directly to the loadings of LFW-MB as depicted in Fig. 5 particularly when the loading of LFW-MB at 65 phr recorded at 22.5 % and 19.1 % of enhancements respectively compared to RFL-0.

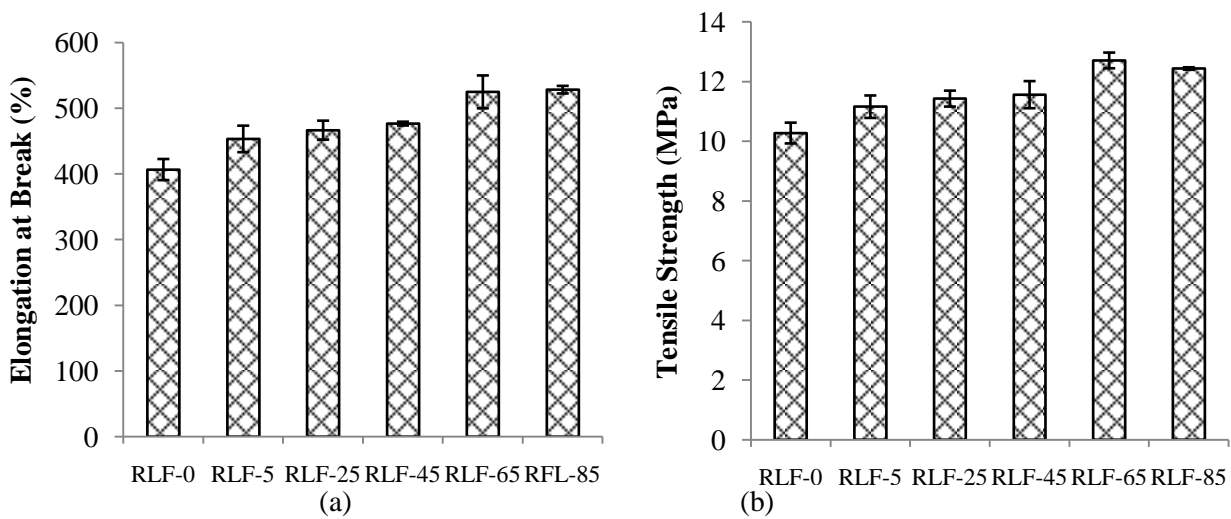
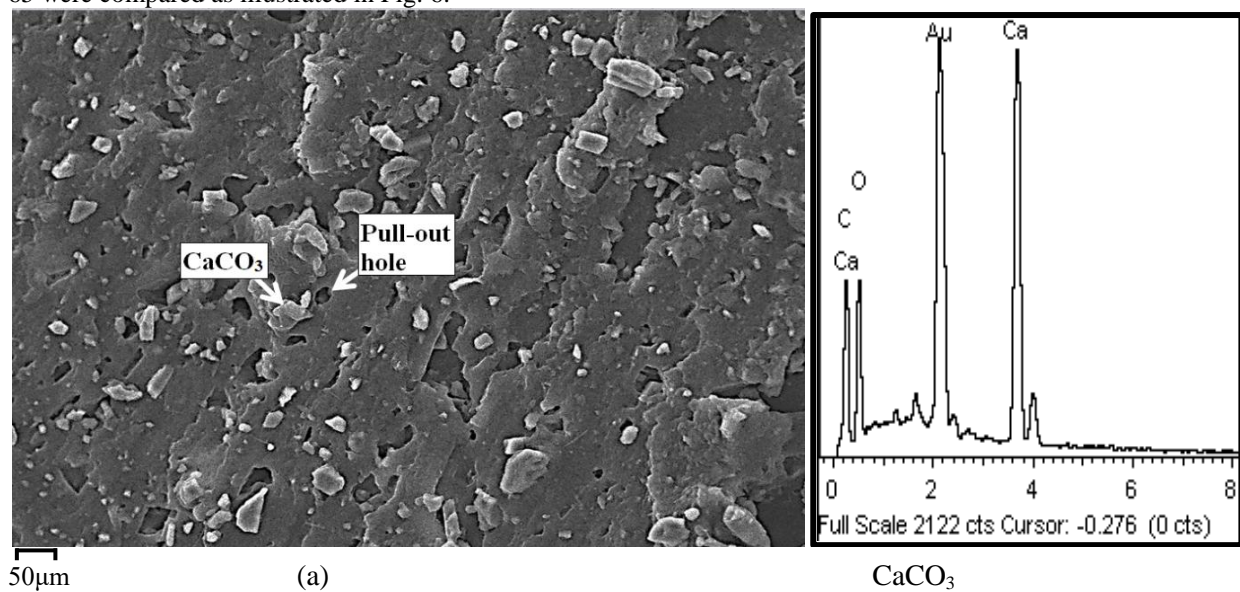


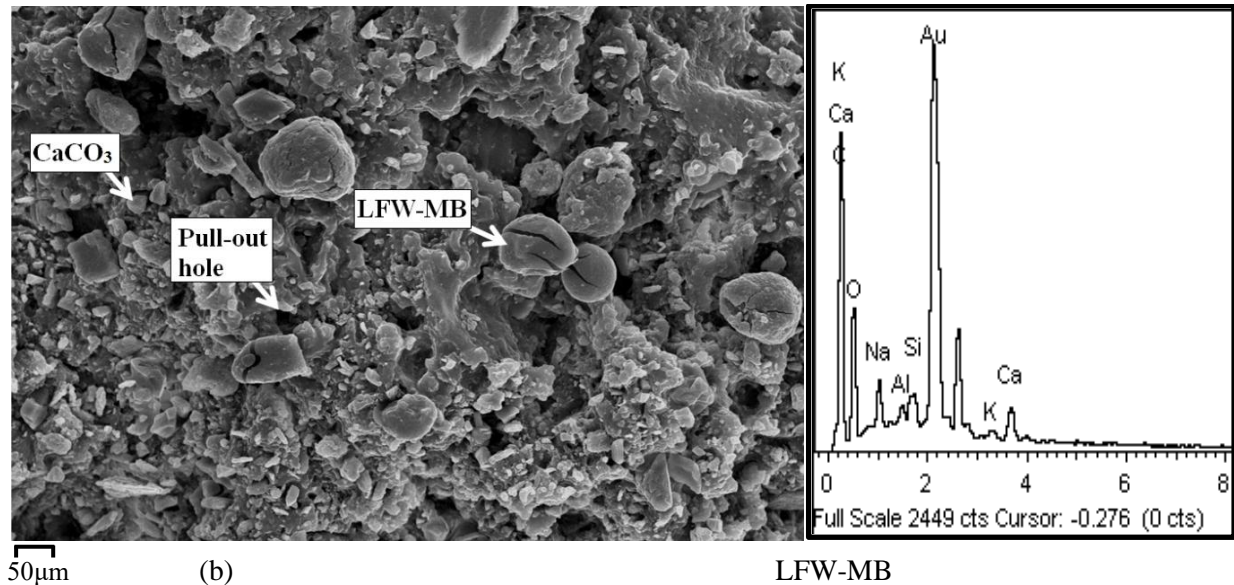
Fig. 5: Elongation at break (a) and tensile strength (b) of vulcanizates

Present research used SBR to shear against LFW which can effectively break the latex foam during prolonged shear mixing. SBR is harder to breakdown compared to NR during mixing and this makes the rubber suitable to be used in the incorporation and dispersion of other ingredients [26]. In this context, the multiple-stage-prolonged mixing by using the masterbatch method has not been excessively masticated the rubber chains while ensuring homogenous dispersion and distribution of LFW-MB aggregates across the rubber matrix. As good dispersion and distribution of rubber ingredients is a primary condition to enhance the mechanical properties [25, 27, 28, 29, 30], the elastomeric LFW-MB and other rubber ingredients are inferred able to disperse, distribute and adhere well across the rubber matrix and therefore increase the rubber mechanical properties especially at the optimal loading of 65 phr. Above this level at 85 phr, a slight reduction on TS was observed probably due to the lower crosslink density and modulus of the vulcanizate as revealed in Fig. 3. Several researchers [20, 31, 32] supported a sufficient level of modulus is important to sustain the tensile strength.

### 3.5 SEM Micrographs

SEM micrographs on the fractured surfaces of tensile samples and EDX analysis of RLF-0 and RLF-65 were compared as illustrated in Fig. 6.





**Fig. 6:** SEM fractured surface micrographs of RLF-0 (a) and RLF-65 (b) with magnification 1000x and EDX analysis on CaCO<sub>3</sub> and LFW-MB

The fractured surface of RLF-0 revealed different sizes of CaCO<sub>3</sub> aggregates of approximately 10 μm to 80 μm were distributed across the rubber matrix with profuse pull-out holes which indicated a poor rubber-CaCO<sub>3</sub> interaction and deteriorated its mechanical properties. CaCO<sub>3</sub> is added for cost reduction in rubber compounding but it impairs the mechanical properties of rubber due to its low surface area and surface activity [19]. In terms of RLF-65, smaller CaCO<sub>3</sub> of approximately 10 μm to 50 μm were coexisted with the larger LFW-MB aggregates. The filler was dispersed into smaller aggregates with lesser amount of pull-out holes ascribed to higher mix shearing effect when blending the elastomeric LFW-MB in the rubber. It is evidenced by the higher  $M_L$  observed previously that represents higher viscosity of the compound in the present of LFW-MB. Although most of the LFW-MB aggregates were above 50 μm, their cured rubbery nature inherited from the NR/SBR blend could interact well with the base compounds originated from the NR system and thus instigated effective transferring of load from the rubber chains to the elastomeric aggregates under straining. The enhanced mechanical properties of the latex-foam-waste incorporated rubbers were well-supported by the SEM micrographs.

#### IV. CONCLUSION

The adding of LFW-MB into the natural rubber compounds by using an industrially practical mixing method can achieve several advantages. LFW-MB can reduce the cure time of rubber compounds which is advantageous in increasing production throughput. Moreover, it can reduce the s.g. of rubber compounds and allow lesser material usage in occupying a fixed volume of mould to save the material cost per product. Apart from these economically viable advantages, LFW-MB can enhance the mechanical properties on both EB and TS attributed to the improved dispersion and distribution of filler and the well-interacted elastomeric LFW-MB with rubber matrix which enable better energy dissipation under the stress-strain characterizations. However, LFW-MB reduces the crosslink density of rubber vulcanizates and thus diminishes their modulus and hardness values. Since LFW-MB can reduce the cure time of rubber compound, future work may focus on its suitability in substituting the expensive rubber cure accelerators which can appreciably reduce the compound cost.

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