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Juvenile growth, rubber yield and putative tolerance to foliar diseases in interspecific hybrids and half-sibs of Para rubber tree (*Hevea* spp.)

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Abstract

Introgressive hybridization was initiated using different Hevea spp. and wild germplasm accessions. High yielding H. brasiliensis clones but susceptible to foliar diseases (RRII 105, RRII 414 and RRII 430) were hybridized with other Hevea spp. (H. spruceana and H. camargoana) and wild accessions (RO 380 and RO 2871) which are putatively tolerant to important foliar diseases. In addition, half-sibs were collected from a disease tolerant interspecific hybrid clone, Fx 516. The F1 hybrids and half-sibs were evaluated for growth and yield at the age of three years. Regarding family-wise growth in terms of girth, RRII 430 x RO 380 (mean, 28.6 cm) and RRII 430 x Hevea spruceana (mean, 23.0 cm) had more girth. In contrast, RRII 105 x H. camargoana and RRII 430 x H. spruceana had progenies with low girth. With reference to family mean yields, RRII 414 x RO 380 gave a mean yield of 64.8 g/t/10t followed by RRII 430 x RO 380 (mean yield, 15.3 g/t/10t) and RRII 430 x H. camargoana (mean yield, 15.2 g/t/10t). Remaining cross-combinations produced progenies with low yield ranging from 7.1-8.5 g/t/10t. The highest yielding individual of the family RRII 414 x RO 380 gave a yield of 190.8 g/t/10t. Half-sibs of Fx 516 had a mean yield of 17 g/t/10t with maximum individual yield of 56 g/t/10t. Progenies were variably affected by powdery mildew disease caused by Oidium. After confirming segregation of disease resistance trait among progenies through detailed field evaluation in hot spots, the F1 population will be continuously used in back crossing programme to ensure introgression of disease resistance traits.

Key words: disease tolerance, Hevea spp., introgressive hybridization, juvenile growth, juvenile yield, wild germplasm

Introduction

In genetic improvement programme of *Hevea*, the Para rubber tree, rubber yield is generally considered as the prime breeding objective (Simmonds, 1989; Mydin, 2014). Since breeding programmes were mainly yield-based, most of the high-yielding rubber clones showed variable levels of susceptibility to major fungal diseases (Liyanage & Jacob, 1992; Jacob, 1997; Jayasinghe, 1999). Leaf diseases due to *Phytophthora*, *Corynespora* and *Oidium* and stem disease due to Corticium cause up to 40% economic loss in rubber yield (Jacob, 1997; Narayanan & Mydin, 2012; Narayanan & Reju, 2020). Recently, disease epidemics have been reported from rubber plantations in Cameroon, Indonesia, Malaysia, and Sri Lanka (Ngobisa, 2018). South American Leaf Blight (SALB) caused by *Pseudocercospora ulei* (= *Microcyclus ulei*) is a devastating fungal disease which almost wiped out rubber plantations in Brazil epidemic outbreaks (Hora Júnior *et al*., 2014). SALB is a looming threat to the global rubber cultivation (Chee, 1990).

In the event of more such unexpected epidemics in future, availability of clones with enhanced tolerance to diseases will be a critical factor for sustainable natural rubber production. Development and use of disease tolerant clones will considerably reduce dependence on fungicides in disease management thereby lowering costs of rubber production (Fernando, 1969). Hence, an introgressive hybridization programme was initiated for evolving high yielding clones with tolerance to different diseases. Introgression (or "introgressive hybridization") involves integration of genes usually *via* hybridization and repeated backcrossing of alleles from one species into another (Anderson & Hubricht, 1938; Anderson, 1949). In the present breeding programme, the aim was to transfer genes for disease resistance from other *Hevea* species into the high yielding clones belonging to *H. brasiliensis* through hybridization and subsequent back crossing. In the first step of introgressive breeding, hybridization was carried out between high yielding but disease susceptible *Hevea brasiliensis* clones (RRII 105, RRII 414 and RRII 430) as female parents and two *Hevea* spp. (*H. spruceana* and *H. camargoana*) and two wild germplasm accessions from Rondônia (RO 380 and RO 2871) as male parents. In addition to the above, half-sibs were collected from an interspecific hybrid Brazilian clone, Fx 516. In the first phase, the full-sib and half-sib populations recovered through the above breeding programme were assessed for juvenile growth and yield and superior selections were identified. This paper reports the findings from the first phase of the study on variability in early growth, juvenile yield, and disease resistance of the progenies and their selections.

Materials and Method

The parental clones of *H. brasiliensis* and other *Hevea* spp. were located at Central Experimental Station (CES) of Rubber Research Institute of India, situated at Ranni Dt. (Kerala State, India). Details of parents are given in Table 1. Distinctive species variation of the parents was evident from their leaf, flower, fruit and seed characters (Figs. 1 and 2). Identity of the species was further confirmed based on their unique and distinguishing reproductive characters already described (Schultes, 1990). Regarding floral characters, *H. spruceana* produced long-pedicellate flowers which are dark yellowish above and brownish to reddish purple below. The wild accession RO 380 possessed smaller and comparatively short-pedicellate flowers which are also slightly rose-coloured at the base. In contrast, *H. camargoana* produced very unique, strongly elongated, rose-coloured flower buds with inflated globose base. While the flowers of *H. brasiliensis* are creamy-yellowish and extremely pungent-aromatic, the flowers of *H. benthamiana* are comparatively smaller and yellowish. The interspecific hybrid clone Fx 516 possessed yellowish flowers and other characteristic vegetative and reproductive traits which are intermediary between *H. brasiliensis* and *H. benthamiana*. Hybridization was carried out at CES using various cross combinations during months of January-March during 2014 to 2016. Details of cross-combinations are given in Tables 2. Half-sibs were collected during 2015 (91 nos.) from clone Fx 516. The full-sib and half-sib progenies of the different years were evaluated (spacing 60 x 60 cm) in nursery trials in a contiguous area in the experimental farm of Rubber Research Institute of India, Kottayam (Kerala State, India). Progenies were assessed for growth and juvenile yield after 36 months of planting following standard protocols (Mydin & Saraswathyamma, 2005). Briefly, girth (cm) was recorded at 20 cm height and rubber yield was evaluated through test-tapping at same height. Progenies were tapped for latex by the modified Hammaker-Morris-Mann method where thin layer (1-3 mm thick) of bark was excised from outer stem portion following a downward angle using a modified tapping tool. A tin spout was attached to the end of tapping wound or channel to collect the exuding latex in a collection cup. Test-tapping was carried out following S/2.d3.6d/7 system (1/2S, half-spiral incision; d/3, collection every third day). After discarding latex from first five tappings, latex from subsequent ten tappings were accumulated, air-dried and weighed for recording individual yield performance. Since some of the rubber lumps were larger for air-drying, they were dried in a smoke house. Dry rubber yield (g/t/10t) was recorded during the months of November and December which is the period when latex yield is high in *Hevea*.

Table 1. *Details of parental clones and other Hevea spp.*

Parental clone	Clone origin/distribution of species/Habitat	Remarks
RRII 105 (Tjir 1* x Gl 1#); RRII 414, RRII 430 (RRII 105 x RRIC 100)	Hybrid of *H. brasiliensis* (Willd. ex A. Juss.) Mueller-Argoviensis evolved in India. The species naturally occurs in South of Amazon river (Brazil, Bolivia, Ecuador and Peru). Large trees	High-yielding clone classified under Category-I of India Rubber Board's Clone recommendation
Fx 516** (F 4542 x AVROS 363$)	Hybrid developed in Par ́a (Brazil). F 4542 is a selection of *H. benthamiana* Mueller-Argoviensis originating from upper Rio Negro in South America. Medium to large sized trees	Highly tolerant to leaf diseases caused by *Phytophthora* and *Corynespora*

Parental clone	Clone origin/distribution of species/Habitat	Remarks
RO 2871	Wild germplasm accession. Collected from Rondônia province of Brazil in 1981. Large trees	Highly tolerant to *Powdery mildew*
RO 380[@]	Wild germplasm accession from Rondônia. Medium to large sized trees	Variably tolerant to major diseases
H. camargoana Murca Pires	Naturally occurs in Marajo island of Amazon river delta (Brazil). Small trees of 2-2.5 m ht.	Variable tolerance to SALB
H. spruceana (Benth.) Mueller-Argoviensis	Naturally occurs in banks of Amazon, Rio Negro and lower Madeira (Brazil). Medium sized tree	Variable tolerance to SALB

[*]Primary clone of *H. brasiliensis* from Indonesia; [#]Primary clone of *H. brasiliensis* from Malaysia; ** Fx, Ford Cross; [$]AVROS, Al-gemene Verneiging Rubber planters Oostkust Sumatra, Indonesia; [@]possibly a putative interspecific hybrid of *H. brasiliensis* with *H. pauciflora* (Jayashree *et al*., 1997)

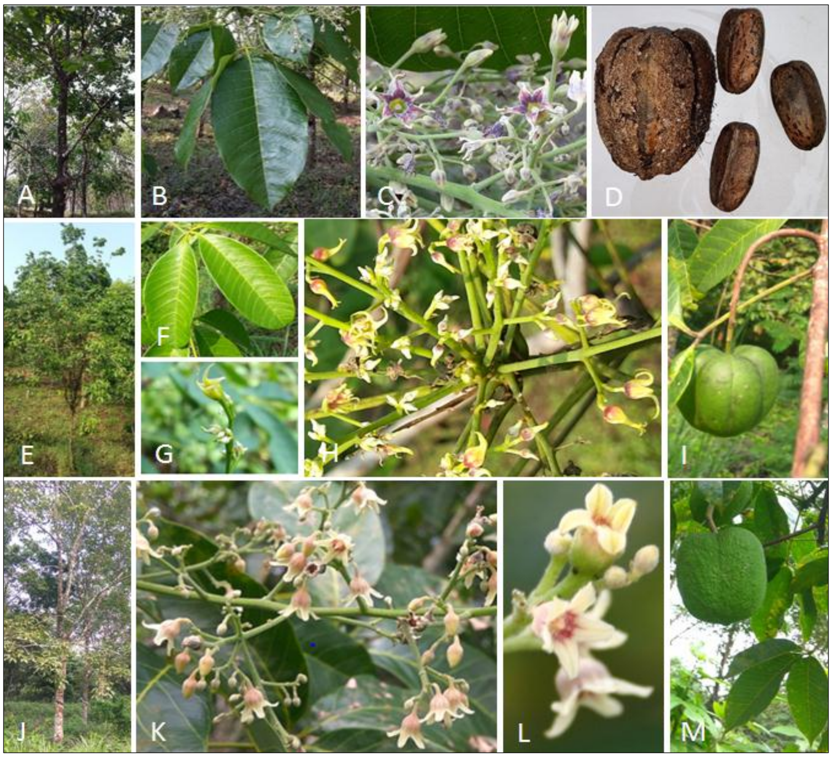

Fig. 1. A-M. Habit, leaf, floral, fruit and seed characteristics of *Hevea* spp. and wild germplasm accession used in introgressive hybridization. A-D. *H. spruceana* E-I. *H. camargoana* J-M. Rondônia accession, RO 380

Fig. 2. A-N. Habit, leaf, floral, fruit and seed characteristics of *Hevea* spp. used in introgressive hybridization. A-E. Clone RRII 430 (*H. brasiliensis*) F-I. Clone F 4542 (*H. benthamiana*) J-N. Clone Fx 516 (*H. benthamiana* x *H. brasiliensis*)

Results and Discussion

Success rate of hybridization and progenies recovered

The success of hybridization over three years ranged from 0.9 per cent to 11.4 per cent (Table 2). The average success rate over the years was estimated as 5.6% which is in agreement to earlier reports in *Hevea* (Simmonds, 1989). Family-wise, RRII 430 x RO 380 had maximum progenies while RRII 414 x RO 380 and RRII 430 x *H. camargoana* had minimum progenies.

Table 2. *Details of year-wise cross-combinations with success rate*

Year	Cross combination	Number of crosses	Hybrids recovered	Success rate (%)
2014	RRII 414 x RO 380	446	6	1.3
	RRII 105 x RO 380	371	19	5.1
	RRII 105 x *Hevea spruceana*	147	6	4.1
2015	RRII 430 x RO 380	1605	95	5.9
	RRII 430 x *Hevea camargoana*	58	6	10.3
	RRII 430 x *Hevea spruceana*	429	49	11.4
2016	RRII 430 x RO 2871	1259	11	0.9
	Total	4315	192	
	Overall success rate			5.6

Growth and yield performance of progenies

The F1 hybrids and half-sib progenies showed considerable variation for juvenile growth and yield (Table 3). Individual girth of full-sibs ranged from 10.0 to 39.0 cm. Family RRII 414 x RO 380 (mean girth, 28.6 cm) followed by RRII 105 x RO 380 (mean girth, 23.0 cm) and RRII 430 x *Hevea spruceana* (mean girth, 22.7 cm) displayed good growth vigour. Family RRII 430 x RO 2871 attained comparatively lesser girth (mean girth, 13.9 cm). In terms of individual growth performance, a progeny of RRII 414 x RO 380 achieved maximum girth (39.0 cm). Overall, progenies from cross-combination involving RO 380 as male parent possessed high girth. This indicated that RO 380 could be a good candidate for improving growth vigor with potential adoption in future breeding of vigorous latex-timber clones. Half-sibs of Fx 516 exhibited a mean girth of 20.1 cm with girth ranging from 5.0 to 40.0 cm.

There was high variation in test-tapping yield of the progenies. While highest test-tapping yield (190.8 g/t/10t) was recorded in a progeny of RRII 414 x RO 380, lowest yield (0.5 g/t/10t) was recorded in a progeny of RRII 105 x RO 380. Regarding family mean yields, RRII 414 x RO 380 gave high yield (mean, 64.8 g/t/10t) followed by RRII 430 x RO 380 (mean, 15.3 g/t/10t) and RRII 430 x *H. camargoana* (mean, 15.2 g/t/10t). Remaining cross-combinations produced progenies with low mean yield (7.1 - 8.5 g/t/10t). One progeny of RRII 430 x RO 380 recorded high yield within that family (54.4 g/t/10t). Half-sibs of Fx 516 gave a mean yield of 16.9 g/t/10t with maximum individual yield of 56.1 g/t/10t.

Among different cross combinations, RRII 430 x RO 380 produced more selections followed by RRII 430 x *Hevea spruceana* (Table 3). Considerable number of half-sib selections could also be recovered from the disease tolerant clone Fx 516.

Table 3. *Family-wise growth and yield performance of F1 hybrids and half-sibs of Fx 516*

Family	Girth (cm)		Yield (g/t/10t)		Number of selections
	Range	Mean	Range	Mean	
Year 2014					
RRII 414 x RO 380	18.5-39.0	28.6	1.0-190.8	64.8	4
RRII 105 x RO 380	10.0-35.0	23.0	0.5-33.5	7.1	7
RRII 105 x *Hevea spruceana*	10.0-24.0	17.0	1.0-16.9	8.5	3
Year 2015					
RRII 430 x RO 380	10.0-30.5	19.4	0.7-54.4	15.3	36
RRII 430 x *Hevea camargoana*	15.5-19.0	17.6	6.8-25.3	15.2	4
RRII 430 x *Hevea spruceana*	14.0-35.5	22.7	1.0-23.9	7.9	19
Year 2016					
RRII 430 x RO 2871	10.0-19.5	13.9	1.0-23.9	7.9	3
Year 2015					
Half-sibs of Fx 516	5.0-40.0	20.1	1.1-56.1	16.9	34
Total number of selections					110

Putative resistance to powdery mildew disease among progenies

Based on disease assessment during first three years of the nursery evaluation, progenies did not exhibit any visible symptoms of leaf diseases caused by *Phytophthora*, *Colletotrichum* and *Corynespora*. However, progenies were variably affected by powdery mildew disease caused by *Oidium* sp. (Table 4). Percent disease incidence (PDI) of powdery mildew ranged from 12% in RRII 105 x RO 380 to 22% in RRII 430 x RO 380 and disease severity (*DI*) ranged from 1.3 in RRII 105 x RO 380 to 1.8 in RRII 430 x *H. camargoana*. Progenies of crosses which involved *H. spruceana* and RO 2871 as male parents had progenies without the disease indicating high disease tolerance of above two parents. Half-sibs of Fx 516 were not affected by any of the above diseases indicating high disease tolerance of this clone.

Table 4. *Variation in incidence of powdery mildew*

Family	***Powdery mildew***	
	PDI (%)	***DI***
RRII 414 x RO 380	18	1.7
RRII 105 x RO 380	12	1.3
RRII 105 x *H. spruceana*	-	-
RRII 430 x RO 380	22	1.4
RRII 430 x *H. camargoana*	16	1.8
RRII 430 x *H. spruceana*	-	-
RRII 430 x RO 2871	-	-
Half-sibs of Fx 516	3	0.7

The wild accession RO 380 used in above hybridization programme is a putative interspecific hybrid of *H. brasiliensis* and *H. pauciflora* (Fig. 1; Jayashree *et al*., 1997). Since *H. pauciflora* was found to be tolerant to SALB, it was used in breeding for tolerance to the disease in Brazil (Chee & Wastie, 1980; Goncalves *et al*., 1980, 1982). Similarly, the female parent of clone Fx 516, F 4542 (*H. benthamiana*), was identified as highly resistant to SALB and was used in breeding for resistance to SALB and other major fungal diseases in Sri Lanka (Senanayake & Wijewantha, 1968; Fernando & Liyanage, 1975, 1980). Similarly Fx 516 showed high tolerance to many fungal diseases including those caused by *Phytophthora* and *Oidium* (Narayanan & Mydin, 2012). Besides, Fx 516 is possibly a potential source of genes for resistance to SALB, as it originated from the breeding programme which was aimed to combine SALB-resistance (of original SALB-resistant seedling selections from the Ford plantations, Fordlandia) with yield of high-yielding clones of Southeast Asia (Subramanian, 1970). Hence, it is hypothesized that some of the progenies might have inherited few resistance genes, which was the primary aim of the present introgressive breeding. While introgression implies transfer of a small sequence the genome from one parental (or species) to another through hybridization and repeated backcrossing, adaptive introgression refers to introgression of genomic regions with potential positive fitness (in the present case, disease resistance) in the recipient species (Bechsgaard *et al*., 2017; Suarez-Gonzalez *et al*. 2018). In order to determine whether the present breeding population is introgressive *per se* or due to adaptive introgression, there is need for more detailed investigations without which results from present study is inconclusive.

Conclusion

In the present study, progenies with vigorous growth, very high juvenile yield and putative resistance to diseases have been recovered through introgressive hybridization. In order to further confirm their disease resistance, the selected population will be screened for major diseases in hot-spots across multiple locations. Once the segregation of disease resistance trait in the progenies could be confirmed through laboratory or field screening, the F1 generation will be used in back crossing to ensure introgression of specific disease resistance traits. Simultaneously, the best selections will also be subjected to clonal evaluation for testing their viability for large-scale commercial planting.

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Effect of blowing agent type and blowing agent loading levels on mechanical and cell morphological properties of dry natural rubber cellular compounds

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Abstract
Cellular rubber is important in various industrial applications, due to its unique structural properties, such as its low density, cushioning performance, thermal and acoustic insulator properties and moderate energy absorption. In this study, the influence of blowing agent type and blowing agent loading levels on mechanical and morphological properties of cellular natural rubber were investigated. Three types of blowing agents namely $NaHCO_3$, DNPT and isopropanol were used. The cellular rubber compounds were prepared using a conventional compression molding technique via a heat transfer foaming process. The mechanical properties were evaluated according to ASTM standards and the results yielded were observed to systematically correlate with the morphology of cellular compounds. Uneven morphology was observed in the compounds with 10 phr for all three blowing agents owing to high gas content in the rubber matrix. Overall, most of the properties of the cellular rubber compounds gradually decreased upon increasing the blowing agent loading levels in terms of mechanical properties, relative density, water absorption and crosslink density. In addition, isopropanol incorporated compound had the lowest compression set properties.

***Key words**:* blowing agent, cellular rubber, mechanical properties, morphology

Introduction
Cellular materials are currently used extensively in a wide variety of applications such as packaging and comfort materials like bedding or seating and as well as in energy absorption and heat transfer applications (Gibson *et al.*, 2010). Properties of cellular structures are dependent on various factors such as cell morphology, cell density, geometric parameters, cell size, material characteristics and manufacturing processes as well as structural boundary and loading conditions for lower weight applications (Mazur *et al.*, 2016). Depending on their cellular structure, cellular materials can be classified in to two groups as closed and open cell structures (Kabir *et al.*, 2006) and they could be selectively

prepared by the proper selection of blowing agents (BAs), their loading and curatives to achieve the correct balance between the volume of gas generated and the degree of curing (Zakaria *et al.*, 2017). BA is the most important ingredient to make the cellular compound due to its gas generating ability within the polymeric materials as they are thermally decomposed into gases causing the cellular structure formation as temperature increases. However, the correct BA is selected for a polymer based on decomposition temperature, decomposition rate, particle size (surface area) and the gas generation capacity of the BA (Wagner, *et al*., 2014). Typical gases released from blowing process are nitrogen, carbon monoxide and carbon dioxide (Sombatsompop *et al*., 2000). In this study, three BAs namely sodium bicarbonate ($NaHCO_3$), Dinitroso-pentamethylene tetramine (DNPT) and isopropanol were used. DNPT which is an organic BA with decomposition temperature of 190 °C was used as it is a BA widely used in the world for most of the commercial applications (Choi, *et al*., 2006). Isopropanol is also an organic BA with the lower decomposition temperature (83 °C). Sodium bicarbonate is an inorganic BA which decomposes at 50°C producing CO_2 and H_2O and sodium carbonate (Tangboriboon *et al*., 2015 and Kmetty *et al*., 2018). Natural rubber (NR) was selected as the polymeric material in this study due its natural availability and its renewable properties, in order to promote its usage replacing synthetic polymers, such as polyurethane.

It is evident from literature that a majority of studies focuses on rubber foam derived from latex and synthetic polymers lattices (Sombatsompop *et al*., 2000). However, studies concerning dry rubber foams with the comparison of organic and inorganic BAs have not received much attention and dry rubber based cellular materials are considered to have rigid cellular structures compared to latex-based foams which can be used for wide variety of applications such as compressive bushes, automotive items, *etc.* Therefore, in this study, natural dry rubber based cellular material was prepared by varying the loadings of the above mentioned three types of BAs and their properties were investigated.

Experimental

Materials and Methods

The natural rubber used in this study was crepe rubber (1X), obtained from Rubber Research Institute of Sri Lanka. $NaHCO_3$ (w/w 99.9%), DNPT (w/w 99%) and isopropanol (w/w 99.9%) were purchased from Organic Trade (Pvt.) Ltd, Sri Lanka. All other rubber ingredients, such as sulphur (w/w 99.5%), zinc oxide (w/w 98%), stearic acid, tetramethyl thiuram disulphide (TMTD), white oil and diphenyl guanidine (DPG), were purchased from Glorchem Enterprise, Sri Lanka.

Preparation of dry rubber based cellular compounds

Three series of dry rubber based cellular compounds were prepared with three different types of BAs and by varying the BA loading from 2 to 10 phr at 2 phr intervals. The compound prepared with 6 phr loading of DNPT was considered as the Control. The formulations of the compounds are given in Table 1. Compounding was carried out using a laboratory two-roll mill (6” x 13”) (David Bridge & Co. Ltd. Castleton Rochdale, England). Total mixing time of the two roll mill was kept constant at 10 minutes. The mixing cycle used in the preparation of the dry rubber based cellular compounds is given in Table 2.

Table 1. *Formulations of cellular compounds*

Ingredient	Function	Part per hundred of rubber (phr)
Crepe rubber	Polymer	100
ZnO	Inorganic activator	5
Stearic acid	Organic activator	2
TMTD	Primary accelerator	1
DPG	Secondary accelerator	0.5
White oil	Paraffinic Processing oil	15
WSP	Antioxidant	1
Sulphur	Vulcanizing agent	1.5
Sodium bicarbonate	Inorganic BA	2/4/6/8/10
DNPT	Organic BA	2/4/6/8/10
Isopropanol	Organic BA	2/4/6/8/10

Table 2. *Mixing cycle used in preparation of cellular compounds*

Added ingredients/chemicals	Time (minutes)
Crepe rubber (NR)	0
ZnO + Stearic Acid + WSP	2
½ Blowing Agent + ½ White Oil	3
½ Blowing Agent + ½ White Oil	5
DPG	6
TMTD	7
Sulphur	8
Dump	10

Vulcanization and foaming process

The compounds were moulded with a hydraulic press (KAO-Tech compression moulding machine) for 20 min at a temperature of 150˚C.

Characterization of cellular compounds

Mechanical properties

Tensile test

Tensile properties of cellular compounds were determined using the Instron tensile testing machine according to ASTM D3574. Dumbbell shaped tensile test specimens were used. Crosshead speed was maintained at 50 mm/min.

Compression set

The compression set of cellular compounds was evaluated according to ASTM D395, using following Equation 1.

$$compression\ set\% = \left(\frac{h\circ - h1}{h\circ - hn}\right) \times 100\% \ ....1.$$

Where, h_0 and h_1 are initial thickness and thickness after test of cellular compound respectively and h_n is the spacer thickness.

Relative foam density

The relative density of cellular compounds was measured according to ASTM D3575, using Equation 2 as given below:

$$Relative\ density = \frac{Foam\ Density}{Solid\ Density} \ \ldots 2.$$

Crosslink density

The crosslink density was determined as per ASTM D471 standards. Three test specimens having dimensions of 30 mm × 1 mm × 2 mm were immersed in p-xylene for 24 hours in closed-lid bottles. The samples were then removed from the solvent, wiped thoroughly to remove excess solvent and weighed again; this value was taken as the swollen weight. The crosslink density of the sample was calculated using the Flory-Rehner equation given in Equation 3.

$$-\{\ln(1 - \mathcal{V}_r) + \mathcal{V}_r + \mathcal{X}\mathcal{V}_r{}^2\} = \rho\mathcal{V}_0 \quad \mathcal{M}_c^{-1}\,\mathcal{V}_r^{1/3} \ldots\ldots\ldots\ldots\ldots\ldots\ldots 3.$$

Where,

$\mathcal{X}$ = Interaction constant characteristic between rubber and xylene, 0.34

ρ = Rubber density

$\mathcal{V}_0$ = Molar volume of xylene

$\mathcal{V}_r$ = Volume fraction of rubber in swollen sample

$\mathcal{M}_c$ = Average molecular weight between crosslinks

The volume fraction of rubber in the swollen sample, $\mathcal{V}_r$ is given by Equation 4.

$$\mathcal{V}_r = \frac{\left(\mathcal{X}_r/\rho_r\right)}{\left(\mathcal{X}_r/\rho_r\right) + \left(\mathcal{X}_S/\rho_S\right)} \ \ldots\ldots\ldots 4.$$

Where,

ρ_S = Density of xylene

ρ_r = Density of the raw rubber

$\mathcal{X}_S$ = Mass fraction of xylene, which can be obtain from Equation 5

$\mathcal{X}_r$ = weight of the rubber, given by equation 6

$$\mathcal{X}_S = \frac{(weight\ of\ swollen\ sample - original\ weight)}{weight\ of\ swollen\ sample} \quad ..5.$$

$$\mathcal{X}_r = 1 - \mathcal{X}_S \quad ....6.$$

Therefore, the value of $\mathcal{M}_c$ can be used to calculate the physical crosslink density, using Equation 7.

$$[\mathcal{X}]_{phys} = \frac{1}{2\mathcal{M}_c} \quad ....7.$$

Cell morphology

Surface morphology of cellular compounds was examined by scanning electron microscopy (SEM) using a ZEISS EVO LS 15 microscope. The specimens were cut and mounted on aluminum stubs. The specimens were then sputter coated with a thin layer of gold to avoid electrostatic charging during examination.

Water absorption

The water absorption was evaluated according to ASTM D570, using Equation 8 where initially the weight of the test specimens was measured and recorded (Dry weight). Then the specimens were emerged in distilled water for three days and the weight was measured (Wet weight). The test specimens were patted dry with a tissue paper before measuring the final weight.

$$Water\ Absorption\ \% = \frac{(Wet\ Weight - Dry\ Weight)}{(Dry\ Weight)} \times 100\% \quad .....8.$$

Data analysis

All three series of cellular compounds were subjected to the above physio – mechanical tests and obtained data were analyzed by using the Minitab 17 statistical software with the use of General Liner Model (GLM) with Dunnett comparison tests at 95% confidence interval. All the tests were replicated for three times at least and error bars have been calculated for the replicated results.

Results and Discussion

Mechanical properties

Figure 1 shows a decreasing trend in tensile strength with the increase BA loading for all three blowing agents investigated. This could be attributed to the increase in the free volume or air space generated at high BA loading resulting the cells to collapse in the final morphological structure (Fig. 7). Decrease in crosslink density as evident from the results presented in Figure 7 may have also contributed to this trend. The compounds prepared with 2 phr loading of BA showed a significantly high tensile strength when compared to that of other loadings. Further, compound prepared with 2 phr loading of isopropanol shows remarkable tensile strength compared to other cellular rubber types. In addition, as the BA loadings increased the gas phase of the compound increases resulting the cells to collapse in the final morphological structure (Fig. 7).

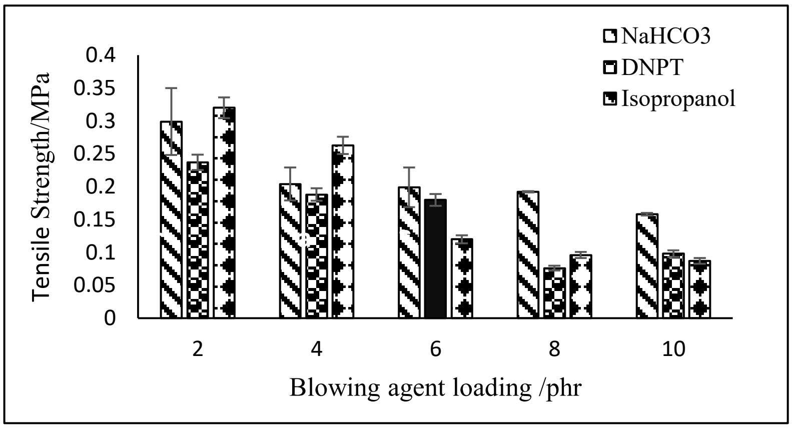

Fig. 1. Tensile strength of cellular rubber compounds

Figure 2 shows variation of elongation at break with different types of BAs and loadings. Elongation at break is an important physical parameter to explain the elastic behavior of the amorphous material. There is a decrease in trend on elongation at the break of $NaHCO_3$ and DNPT with the increase of BA loadings. In contrast, the results of the isopropanol incorporated compound indicate a fluctuating nature. The compound prepared with 2 phr loadings of $NaHCO_3$ shows better results compared to other BA loadings. This could be due to the better adhesion among the NR molecules. Figure 2 shows the surface morphology of 2 phr loading of the former three types of Bas and it shows that $NaHCO_3$ has large cells compared to isopropanol and DNPT. Similar morphological observation can be seen in the studies conducted by Najib *et al*., (2009). It could be a possible reason for having high elasticity for the compound with $NaHCO_3$ BA. In contrast, the surface morphology of DNPT incorporated compound has high cell density at 2 phr loading. As a result, the elasticity of this compound is lower than $NaHCO_3$ and isopropanol.

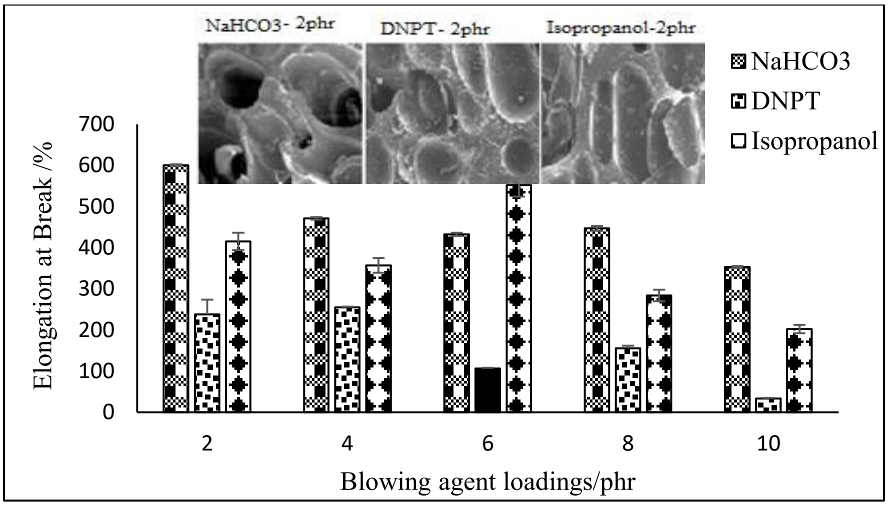

Fig. 2. Elongation at break of cellular rubber compounds

Modulus at 100% elongation is an important parameter to explain the hardness of a polymeric material (Gauthier, 1995). The compounds prepared with 4 phr loadings indicate the highest results for modulus at 100% elongation. Therefore, it might be the optimum loading to enhance the proper adhesion between NR and compounding ingredients. When the overall trend of modulus at 100% elongation is considered, an increment up to 4 phr loadings of BAs followed by a decline from 4 phr to 10 phr loadings were observed. At higher loadings of BAs, compounds indicate lower results of modulus at 100% elongation. There are number of possible reasons for this observation. The increase in the gas phase within the compound reduces the rubber phase by creating micro voids which then reduces the strength of the compound compared to a compound without BAs. Additionally, with the increase in BA loading some of the cell walls can be fragmented leading to low strength structures within the compound. Another reason might be the possible agglomerates of the BAs at higher loadings which lowers the interfacial interactions between NR and BA. This is evident from the morphological studies carried out at 6 phr and 10 phr loading of the $NaHCO_3$ BA (Fig. 7). This observation enables ease of fractions to occur leading to a reduction in hardness. Further, similar results have been obtained in the study of property evaluation of cassava starch incorporated sponge based on NR (Temna *et al.*, 2014).

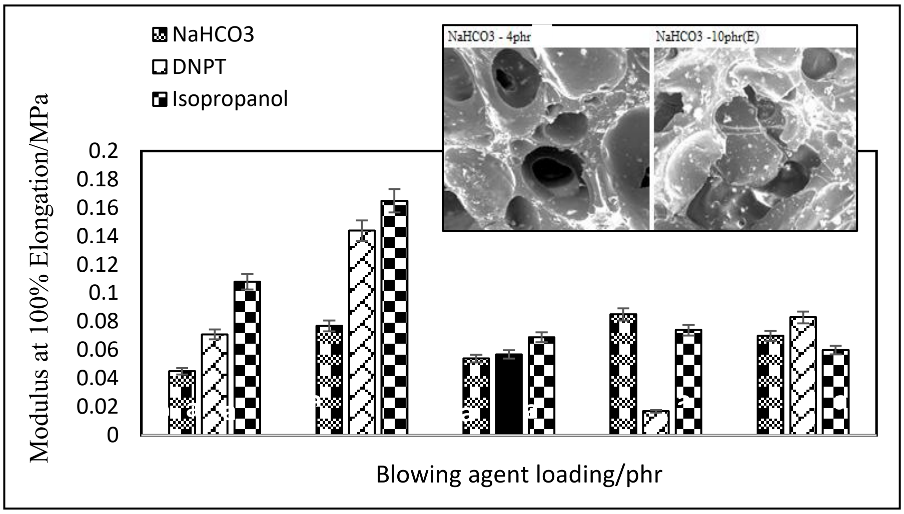

Fig. 3. Modulus at 100% elongation of cellular rubber compounds

According to Figure 4, a decreasing trend is observed in the compression set with increasing BA loading. This can be attributed to the number of cells generated when increasing the BA (Najib *et al*., 2009). When the BA loading increases the number of cells within the structure is increased (Fig. 7). Therefore, a high amount of micro voids enables the compound to store more gas within the structure and release it when it is compressed. This helps to retain the elastic properties resulting in a decreasing trend in the compression set values for each BA. When comparing the results of different BAs $NaHCO_3$ has the highest compression set values followed by DNPT and isopropanol compounds. The main reason for this observation can be the open/closed cell structures present in the compound. The air trapped within the closed cell structures helps it to bear the force to retain until it is released. However there is no such support in the case of open cell structure which then leads to possible breaking of the cell walls providing higher compression values when compared to closed cell structures. The study conducted by Tangboriboon, *et al*., (2015) also reveals this trend with $NaHCO_3$ and DNPTBA.

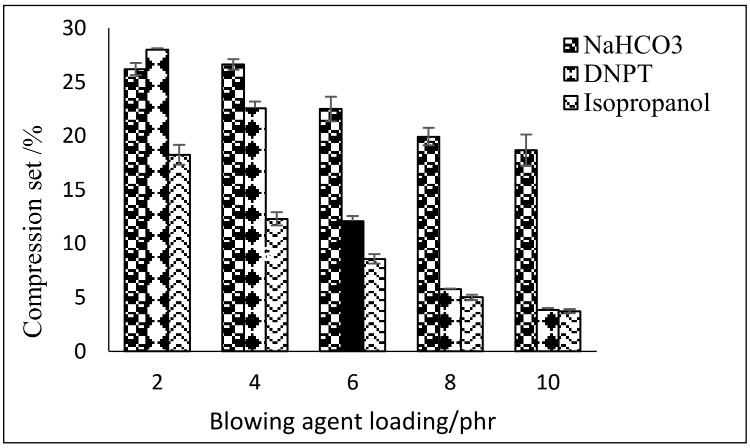

Fig. 4. Compression set of cellular rubber compounds

Relative density

Figure 5 illustrates the effect of the BA and BA loading on the relative density of the cellular rubber compound. It shows the relative density value decreased with increase in BA loading. As greater loadings of BAs were used, more gas was subsequently generated, reducing the relative density. Zakaria, (2007) reported that higher BA loadings shorten the growth time of the foam, thus restricting the gas from escaping through the foam surface, allowing the foam to expand more, and consequently, producing foam with a lower relative density. Further, $NaHCO_3$ has low relative density when compared to DNPT and isopropanol. In closed-cell structure, the gases are trapped in the cells and the weight of the compound can be higher than the open cell structure. Additionally, the free volume in the compound with the $NaHCO_3$ BA is higher compared to the other two BAs due to its open cellular structure. When considering the types of gases evolved throughout the decomposition process the CO_2 gas evolved by $NaHCO_3$ and isopropanol is considered to be more soluble in the polymer material when compared to N_2 gas generated from DNPT, and as a result the DNPT containing compound can generate higher number of closed cells (Di Maio *et al*., 2005). This leads to having lower densities with the cellular structures which are having CO_2 as the decomposition gas than N_2.

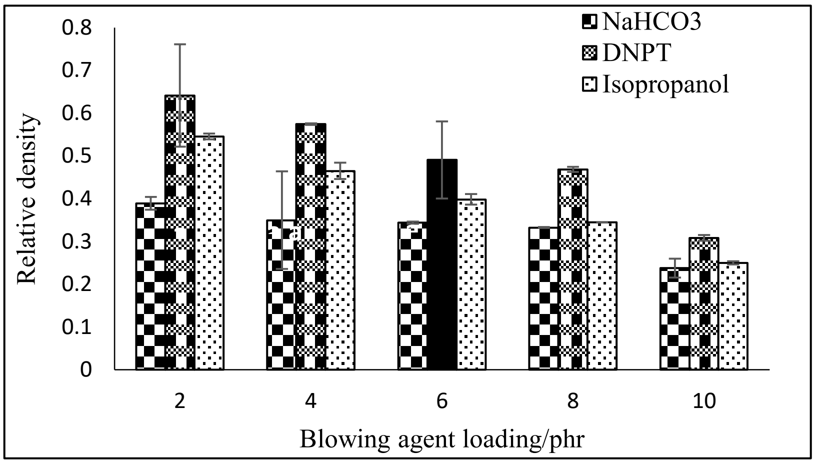

Fig. 5. Relative density of cellular rubber compounds

Crosslinking density

Crosslink density has decreased with increasing BA loading as indicated in Figure 6. DNPT incorporated cellular compound has a higher crosslinking density than the $NaHCO_3$ and isopropanol. This trend can be observed due to the fact that crosslinking and decomposition occur simultaneously; at high BA loadings; thus, the gas phase will be more prominent than the solid phase. As a result of that, thinner cell walls are formed, and consequently, less crosslinking occurs. It would be expected that similar crosslink densities would be obtained for all the samples because the same amount of sulphur (crosslinking agent) was used.

However, the $NaHCO_3$ used in this study decomposed endothermically (Wason *et al*, 1992); this may result in crosslinking deficiency than DNPT as the BA loading increases. DNPT decomposed exothermically (Nitzsche, 2003) and as a result it has higher crosslink density than $NaHCO_3$. At high loadings of $NaHCO_3$, more heat was absorbed from the system for the decomposition process hence, interrupting the crosslinking process. When considering DNPT, when the BA concentration is increased, more heat has been generated from the system making the crosslink formation process easier and as a result more crosslinks have been generated than $NaHCO_3$. Najib, *et al*. (2009) and Ariff, *et al*. (2008) has reported similar observations. Additionally, since the number of cells of N_2 generating BA is high (Di Maio *et al*., 2005), the amount of rubber phase present to form the crosslinks is also high, indicating high results for cross link density for the compounds with DNPTBA.

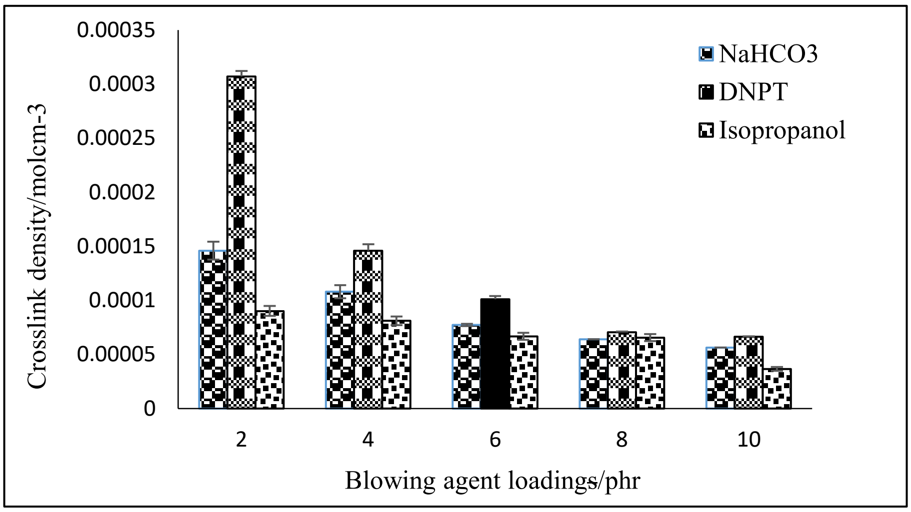

Fig. 6. Crosslinking density of cellular rubber compounds

Morphology analysis

Figures 7 (A), (C) and (E) which are produced using $NaHCO_3$ show open cell structure distinctly and other images that are produced using DNPT and isopropanol show closed-cell structures. When considering the organic BAs (DNPT and isopropanol) the morphology clearly indicates that it not only contains closed cells but also open cells are present in the structure to a certain extent. This indicates that even though DNPT and isopropanol BAs are known as closed-cell BAs, it generates open cells to a certain extent. Cellular rubber compounds at 10 phr of BA loading exhibit the fractures between bubbles due to high amount of gas produced from the thermal decomposition of $NaHCO_3$, DNPT and isopropanol (inserted circle in Fig. 7). There is no uniformity in cell size when increasing the BA loading. Tangboriboon *et al*. (2015) and Charoeythornkhajhornchai, *et al*. (2016) have reported that cell size fluctuates when BA loading is increased. Ramesh, *et al*. (1991) stated that the size of the cell depends on the particle size of the BA and other chemical substances within the vulcanization process, temperature, pressure, and shear rate. Therefore, this can be the reason for the ununiformed cell structure in Fig.7. Furthermore, when considering Fig 7. (C), (E) it can be seen that as the BA loading increased certain level of agglomeration of particles are present (Wu & Shih, 1993).

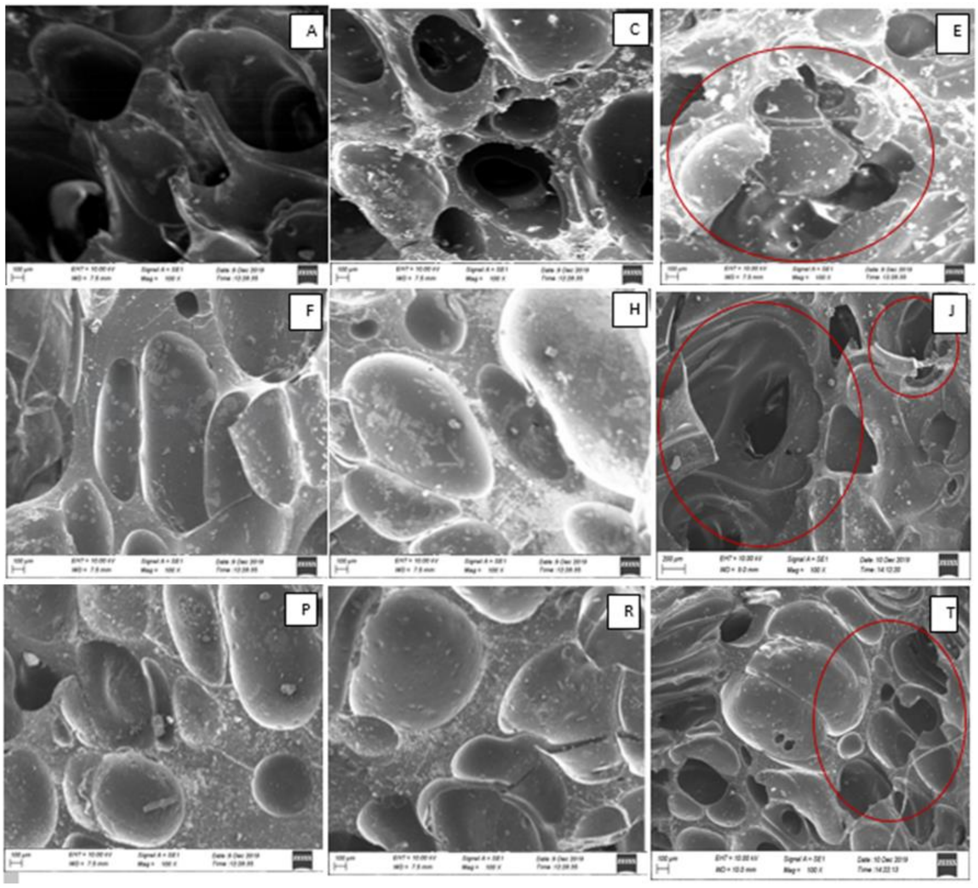

Fig. 7. SEM images of cellular rubber compounds **A**. $NaHCO_3$-2 phr; **C**. $NaHCO_3$-6phr; **E.** $NaHCO_3$-10 phr; **F**. Isopropanol-2 phr; **H.** Isopropanol-6 phr; **J**. Isopropanol-10 phr; **P.** DNPT -2 phr; **R**. DNPT -6 phr; **T.** DNPT -10 phr

Water absorption

Fig. 8 illustrates an increasing trend of water absorption towards increasing BA loadings. This could be related to the relative density results. Najib, *et al*. (2009) reported that a decrease in relative density increases the number of cells per unit volume. As per Fig 5, the relative density is decreasing with the increase of BA loadings for each type of BA. Therefore the number of cells per unit volume and free volume increases with the increase of BA loading (Najib *et al*., 2009). As a result, water absorption is increasing with increasing BA loading. When considering the type of BA, $NaHCO_3$ shows a significantly high water absorption value than the DNPT and isopropanol. This can be attributed to the open/closed cellular nature of the resultant foam.

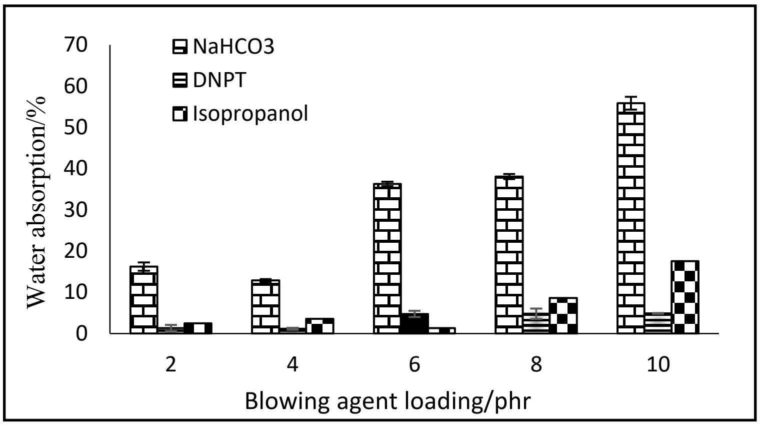

Fig. 8. Water absorption of cellular rubber compounds

Open-cell structures can absorb more water than closed-cell structures. Therefore, $NaHCO_3$ incorporated cellular compound has higher water absorption values than DNPT and isopropanol. When comparing the DNPT and isopropanol results DNPT has a higher relative density than isopropanol. Therefore, the number of cells per unit volume is higher in DNPT than in isopropanol. As a result of that, although both have closed cell structures, DNPT has the lowest water absorption value.

Conclusions

The increase in BA from 2 phr to 10 phr has led the microstructure of the cellular natural rubber changed from dense to porous phase. The highest porosity at 10 phr loading showed the lowest relative density. Further, the water absorption results at 10 phr shows the different abilities to absorb water for open and closed cell structures generated by $NaHCO_3$ and DNPT respectively whereas the trend of DNPT levels from 6 phr have not generated a significant increment of water uptake. Furthermore, the mechanical properties of compounds were decreased when increasing the BA loadings. Therefore, lower loadings of BAs would generate better performance of cellular compounds in terms of tensile and elongation properties. Additionally, the selected control sample provided moderate results within the physical properties when compared to other compounds.

In addition, Isopropanol incorporated compound has the lowest compression set properties and hence, it could be used to produce soft sponge for makeup activities and cushion applications. Water absorption was increased when increasing $NaHCO_3$ loadings. Therefore, it can be recommended to produce rubber sponges for cleaning and washing proposes with the use of $NaHCO_3$. Compound prepared with DNPT showed superior mechanical properties hence, it would be better to produce soft sponge rubber for shoe inner sole and shoe pads for high heels using DNPT.

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Study of raw rubber and dynamic properties of RRISL 203 genotype using rubber process analyzer

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Abstract

Quality raw materials are essential for a sustainable rubber product industry. RRISL has introduced RRISL 203 clones as a solution for demand in high-yielding, and disease-resistant clones with vigorous growth. The objective of the present study is to study the raw rubber and rheological properties to find the suitability to produce advanced rubber products. Un-fractioned-unbleached crepe rubber manufactured using latex from the clone was collected from three crepe rubber factories in three different areas in Sri Lanka. RRIC 121, which is the most popular clone in Sri Lanka was selected as the control. Studies carried out on the raw rubber properties of these rubbers revealed that RRISL 203 clone recorded the highest Plasticity number as well as the highest Mooney viscosity among clones available in Sri Lanka. Further, this clone possesses relatively higher Plasticity Retention Index, nitrogen, and gel content with a significantly higher Mooney relaxation rate which leads to high elasticity. A frequency sweep test carried out using RPA (Rubber Process Analyzer) revealed that RRISL 203 possesses linear polymer with a low degree of branching while having a high molecular weight and broader molecular weight distribution. The overall results revealed that rubber obtained from the new clone leads could be used to produce rubber products with high strength and elastic properties. RRISL 203 clone showed moderate raw rubber properties with the highest Mooney viscosity value. Hence, the associated mastication energy requirement would be higher than that of RRIC 121.

Key words: dynamic properties, elasticity, molecular weight, raw rubber properties, rubber genotype, viscosity

Introduction

Development of new clones is a major task in research and development activities related to the rubber industry to develop clones with various intended features such as vigorous growth (Kudaligama, 2010). Therefore, during the initial hybridization stage above factors are generally considered. RRISL 203 clone is such a clone introduced recently to the plantation sector by crossing of two promising clones, RRIC 100 and RRIC 101. This clone has a vigorous growth in both pre and post-tapping periods and has a significantly higher yield compared to other clones and resistance to some threatening diseases (Liyanage, 2016). The commercial average latex yield of this clone is around 2,500 $kgha^{-1}yr^{-1}$ and it has a potential yield of 3,000-3,500 kg ha^{-1} yr^{-1} as a high yielding clone. This clone has recorded relatively higher polyphenol content (Karunaratne *et al*., 2018). This is the reason for the formation of blackish colour layer in the coagulum. According to the Sakdapipanich, 2022 the polyphenol react with oxygen in the presence of polyphenol oxidase. When these quinines react with amino acids and proteins, colourants will form (Attanayake *et al*, 2015). It has been shown that the fractionation process of the latexm obtained from the RRISL 203 clone yields white colour crepe rubber compared to the widely used clones in Sri Lanka. Similar to colour, raw rubber, and dynamic properties are also of great importance during the conversion process of rubber into value-added advanced products.

As the rubber properties derived from latex from RRISL 203 were not ascertained during the clone development, the properties of dry rubber produced out of this clone are not available. Quality and property of natural rubber depends on the non rubber elements in the latex (Yip, 1990: Haque *et al*., 1995; Le Roux *et al*., 2000).Technological properties of natural rubber vary according to the time of shelf life and clonal origin (Le Roux *et al.*, 2000; Ferreira *et al.*, 2002; Moreno *et al.*, 2003). Physical and technological properties of rubber could be uniform within a clone, but clone wise differences do occur (Attanayake *et al*., 2014). Therefore, this study focused on the raw rubber and dynamic properties of fractioned unbleached crepe rubber of this genotype. Un-fractioned unbleached crepe rubber (UFUB) was selected for this study. UFUB has the simplest manufacturing process among the other possible types of crepe rubber. Other types of crepe rubber such as fractioned-bleached (FB), Fraction-Unbleached (FUB), Un-fraction bleached (UFB) crepe rubber are subjected to various chemical modifications by the addition of certain chemicals which can be influenced for the raw rubber and dynamic properties. Properties of Crepe rubber derived from RRIC 121 clone were also evaluated for the purpose of comparison.

Materials and Methods

Sample preparation

Un-fractioned and unbleached crepe rubber samples were prepared with unpreserved latex RRISL 203 clone and RRIC 121 clone by following standard crepe rubber manufacturing procedures, were used for the study. RRIC 121 clone which is the most abundant clone in Sri Lanka was used as the control genotype. Samples prepared by latex collected from three locations in Wet Zone and Intermediate Zone in Sri Lanka.

Determination of raw rubber properties

All raw rubber properties were tested according to ISO standard procedures. The initial plasticity value was tested according to the ISO 2007:2007 while the plasticity retention index (PRI) was tested according to the ISO 2930:2009) by using Wallace rapid plastimeter. Gel Content was tested according to the ISO/DIS17278:2012 standard. Mooney Viscosity was tested by the (EKT 2001) EKTRON TEK Mooney viscometer according to ISO 289-4:2003 standard. The Mooney stress relaxation test was done simultaneously with the Mooney viscosity measurement. ASTM D1646-07 was used to calculate the elasticity of natural rubber. Relaxation data was expressed in a log-log plot. Slope and the intercept of the graph (trend line of the graph) were used to quantify the stress relaxation co-efficient and rubber elasticity (Malac, 2009).

$M = k\,(t)^a$ ………………… Eq 1

Where, M = the torque value in Mooney units;

k = the torque value one second after the rotor has stopped;

a = an exponent that measures the rate of stress relaxation

t = time in seconds

Power low model was converted into log-log expression as follows,

$\text{Log } M = a\,(\log t) + \log k$ ……………. Eq 2

Relaxation data was expressed in a log-log plot. Slope and the intercept of the graph (trend line of the graph) were used to quantify the stress relaxation co-efficient and rubber elasticity.

Determination of dynamic properties RPA analysis

The rubber process analyzer (RPA) is a dynamic mechanical rheological tester and it is universally used for characterizing raw elastomers and unvulcanized compounds (Henry, 1992). Dynamic measurements of raw rubber were tested by D-MDR 3000 (Mon-Tech, Germany) Rubber Process analyzer (RPA) according to the ASTM D6204 standard at 100°C. This method describes a test method for determining the dynamic properties of raw rubber and uncured rubber compounds.

Frequency sweep is used to characterize the polymeric materials. The samples were cut from a pneumatic cutter and placed into a molding chamber. The frequency sweep was carried out at a strain of 99.9% and at 100°C while the frequency was increased from 0.5Hz - 2.0Hz. The following parameters were measured by the frequency sweep test. Storage modulus (G') and Loss modulus (G'') represents the elastic nature of the sample and the viscous nature of the sample respectively. Tan δ, which is known as the dissipation factor *i.e.* ratio between the loss modulus (G'') to storage modulus (G'), was also measured.

In rubber rheology, the strain sweep is used to characterize the raw polymeric material as well as rubber compounds after the mixing. Strain sweep can reflect the rubber compound behavior during the processing such as injection molding, extrusion, *etc*. ACS 1 rubber gum compound was prepared for two rubber clones according to the standard rubber formulation and a strain sweep test was carried out at a constant frequency of 1.67Hz. Stain value increased from 7-99.9% Storage modulus and Tan δ values were measured by the above test.

Results and Discussion

Raw rubber properties

Table 1. *Summary of raw rubber properties (with the coefficient of variation in parentheses)*

Property	RRIC 121	RRISL 203
Initial Plasticity Number (P^o)	49.20 (3.94)	66.82 (9.30)
Plasticity Retention Index (PRI)	64.25 (6.44)	52.02 (9.92)
Mooney Viscosity [MU] ML(1+4) @100°C	87.70 (4.12)	110.61 (13.14)
Exponent a [log MU/log (s)]	-0.272	-0.224
Elastic energy retention exponent (a+1)	0.728	0.776
Stress Relaxation time (seconds)	18.56 (0.72)	26.18 (1.72)
Gel Content % (w/w)	10.54 (3.45)	34.62 (19.57)

Initial Wallace Plasticity number (P_o) is a measure of the plasticity of the rubber, which indirectly gives the estimation of the polymer molecular chain length (or molecular mass). In general, rubbers with high P_o values would possess a high molecular mass (Bonfils, 1997). The plasticity value of the RRISL 203 clone showed a significantly higher P_o value when compared to the RRIC 121 clone (Table 1), therefore, we can expect a higher molar mass in RRISL 203 clone. This result is on par with the Mooney viscosity values yielded for clones studied. Mooney viscosity is a measure of the processability of raw natural rubber or compounded rubber. However, Mooney viscosity cannot be considered the direct indication of the molar mass of rubber (Malac, 2011). This is because Mooney viscosity is not sensitive enough to the average molecular mass of natural rubber as characterization at a speed lower than 2 rpm (Malac, 2011). This value could also increase due to the presence of non-rubbers at significant levels in the rubber. Raw rubber produced from RRISL 203 clone showed a comparatively higher Mooney viscosity value than the raw rubbers produced from most of the natural rubber clones present in Sri Lanka (Attanayake, 2014). This would increase the cost of compounding due to the additional energy and time required to reduce the high Mooney viscosity value to the desired level.

The plasticity Retention Index (PRI) measures the oxidation resistance of the rubber. PRI value indicates the resistance of the material toward thermal aging (Bateman, 1966). RRIC 121 clone showed a relatively higher PRI value than RRISL 203 clone. This suggests that latex from 203 clones has a lower content of antioxidants than that of 121 clone, even though the non-rubber content of the former is higher than the latter. According to Karunaratne (2018), RRISL 203 clone has relatively higher content of thiol during wintering period whilst no significant changes observed during high yielding period.

Stress relaxation is the measurement of the change of stress with time under constant strain. The rate of Mooney stress relaxation can correlate with molecular structure characteristics such as molecular weight distribution, chain branching, and gel content. It can also be used to give an indication of the polydispersity index (Brown, 1997). Stress relaxation co-efficient exhibit the elasticity of the material and deformation characteristics.

According to Figure 1, RRISL 203 clone showed significantly higher stress relaxation time when compared with the RRIC 121 clone. This might be due to the molecular entanglement of higher molecular weight in the above clone. This observation is further confirmed by the high plasticity value recorded for rubber manufactured from this particular clone. If we place relaxation data in a log-log plot, the uncured rubber elasticity is given by slope “a” (Malac, 2009). The rubber with higher elasticity has a slower relaxation and *vice-versa* (Mark, 1979). RRISL 203 clone exhibit a higher value for elastic energy retention exponent (Table 1) calculated according to Eq2. Higher values of exponent (a + 1) mean higher deformation energy retention in material and therefore higher elasticity of rubber. By measuring Mooney viscosity, it is very difficult to predict the viscoelastic behavior since it does not distinguish between the elastic and viscous components. Therefore, two polymers with the same complex viscosity could have totally different elastic and viscous components (Ehabe *et al*., 2005). Montes and white (1982) reported that *Hevea* rubber possesses the slowest relaxation amongst a range of polyisoprene and it can be attributed to the presence of gel or long-chain branching. The gel content of the RRISL 203 clone is significantly higher than that of the RRISL 121 clone. Sakdapipanich *et al*., 1999 reported that virgin trees tapped for the first time contained as much as 80% gel, and this amount reduced to 3% within 6 days of tapping, while molecular weight changed from 262 to 2,530 kg/mol.

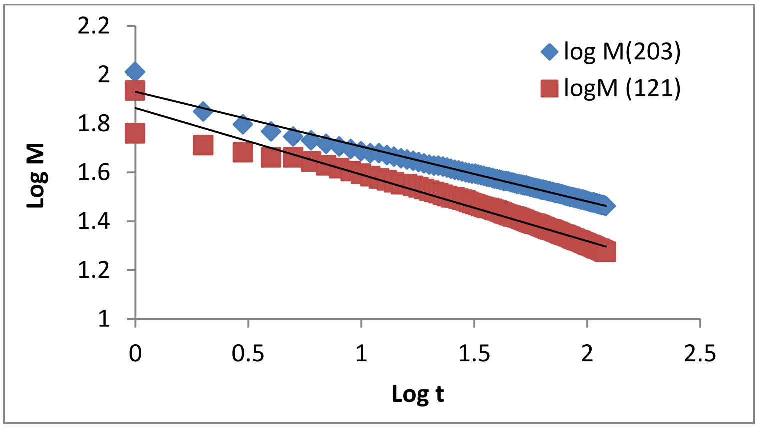

Fig. 1. Relaxation data on a log t (time) versus log M (Mooney value) values for RRIC 121 clone and RRISL 203 clones

Results of dynamic properties

Under an external force, molecular chain orientation is caused by internal friction. When the elastomer molecular weight is lower than a certain value, there is a crossover point between the curves of G' and G" which means that there is a balance in the state of internal friction and disorientation (Fig. 2). With an increase in molecular weight, the crossover point moves to a lower frequency because of restricted disorientation (Zhai, 2010). The lower frequency allows for sufficient time for molecular orientation. The higher molecular weight molecules, therefore, need more time for molecular orientation (Tianming Gao, 2015). The mechanical properties of a material depend on frequency. Isothermal frequency sweep provides information about the molecular weight distribution (crossover modulus) as well as average molecular weight (crossover frequency). Due to the higher molecular weight of RRISL 203, it is not possible to capture the crossover point as the crossover point is happening at a very low-frequency domain. However, a crossover point can be observed for RRIC 121 clone at the very low frequency range.

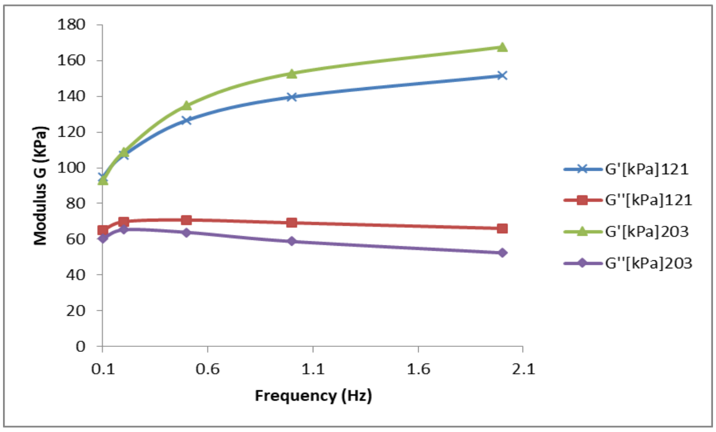

Fig. 2. Variation of storage modulus and loss modules under frequency sweep raw rubber

According to Figure 3, the RRISL 203 clone showed higher crossover modulus and crossover frequency than the RRIC 121 clone. Therefore, RRISL 203 showed lower average molecular weight (AMW) and wide molecular weight distribution (MWD). However, the raw rubber of the RRISL 203 clone showed broader MWD and higher AMW. Therefore, we can suggest that, in the process of rubber compounding, there must be significant chain breakdown to reduce MWD and AMW. Rubber compounds are extremely sensitive to processing operations such as milling. During the milling process, high molecular weight long-chain polymers may be more prone to breakdown than short-chain polymers.

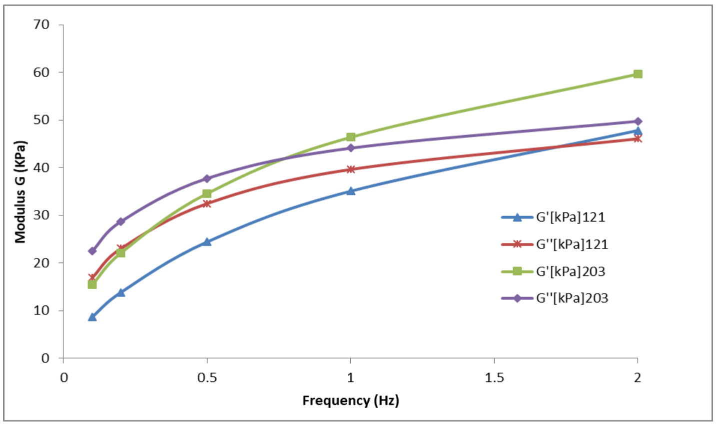

Fig. 3. Overlay plot of frequency sweep test results: Storage modulus (G‘) and Loss modulus (G‘‘) for ACS1 gum compound

Trend of storage modulus of RRISL 203 sample is relatively higher than RRIC 121 sample, which indicates that the RRISL 203 sample has a relatively higher molecular weight and thereby higher molecular entanglements than RRIC 121 sample (Fig. 4).

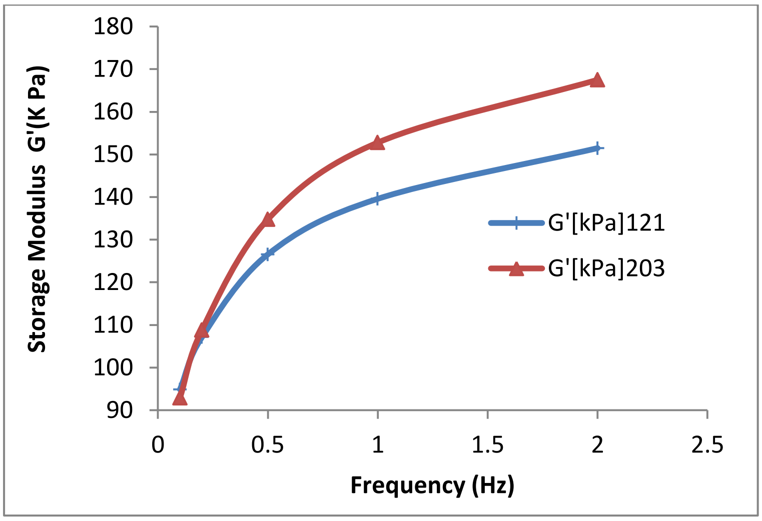

Fig. 4. Overlay plot of frequency sweep test results: overlay plot of storage modulus (G‘) only

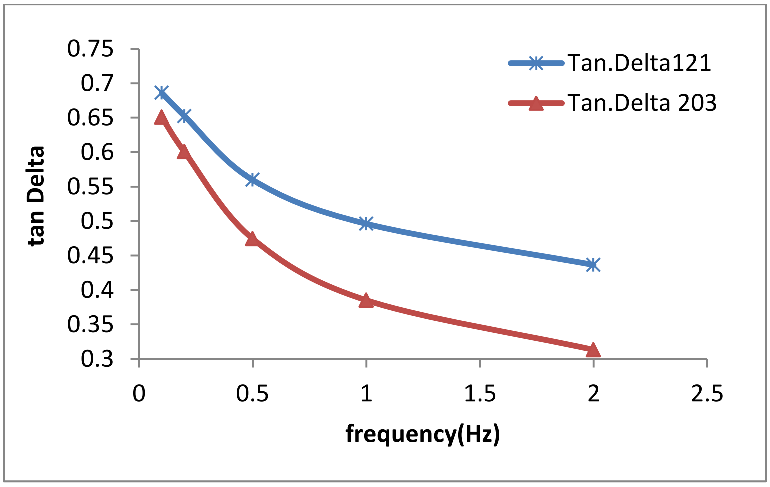

Fig. 5. Variation of Tan Delta values under frequency sweep for raw rubber

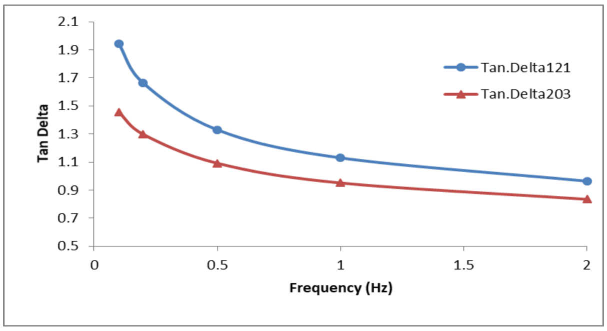

Fig. 6. Overlay plot of tan delta value comparison for ACS 1gum compound

Tan delta is a good indicator of the molecular weight and molecular weight distribution. According to the overlay plot of tan delta values (Fig. 6), the tan delta values of RRIC 121 is higher than R203. It is clearly shown that lower frequency tan delta values are slightly different. The above results showed that the lower molecular fraction of the RRISL 203 clone and RRIC 121 clones are slightly different However, higher frequency tan delta values are significantly different for the two clones studied. A large change in tan delta values indicates that the RRISL 203 clone is a linear polymer with low chain branching with respect to RRIC 121 clone.

Conclusion

RRISL 203 clone showed moderate raw rubber properties with the highest Mooney viscosity value. RRISL 203 possesses linear polymer with a low degree of branching while having a high molecular weight and broader molecular weight distribution. The overall results showed that RRISL 203 could be used for rubber products with high strength and elastic properties. However, the associated mastication energy requirement would be higher than that of RRIC 121.

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Behavioural factors affecting the adoption of manuring of smallholder mature rubber cultivations in Moneragala district

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Abstract

This study was carried out to identify the effective factors that influence the adoption of Manuring of Mature Rubber Cultivation (MMRC) in Moneragala District. The data were collected through a self-administrated survey from 397 smallholders owing mature rubber cultivations in 2020, using stratified random sampling. The conceptual model was developed based on the decomposed theory of planned behavior. The variables were measured with the use of validated items. The responses of rubber smallholders for items were captured on a Five-point Likert scale. Two-stage model-building process was applied in this study. The conceptualized model was empirically tested using partial least square structural equation modeling by bootstrapping procedure using the SMART- PLS 3.2 software. The composite six-predictor structural model was validly and reliably capable of explaining 78% the variability of the adoption of MMRC by rubber smallholders. Adoption of MMRC by rubber smallholders was positively and significantly correlated with the behavioural factors; behavioural intention, perceived behavioural control, whilst perceived behavioural control under adoption was the most significant influential factor. There were positive significant relationships between compatibility and attitude, perceived usefulness and attitude, perceived ease of use and attitude, out of which, compatibility had the most significant effect on attitude, whilst, the relationship between relative advantage and attitude was not significant. These findings can help boost the behavioral intention of rubber smallholders in Moneragala District to adopt on MMRC, especially by improving the aspects of facilitating conditions and subjective norms with the improvement of appropriate development and extension plan.

***Key words**:* Adoption, manuring of mature rubber cultivation, structural equation model

Introduction

Moneragala (6.7563° N and 81.2519° E) has wet, intermediate and dry climatic conditions and many agro-ecological regions, out of which IL1c, IL2 and IM2b are used for rubber (*Hevea brasiliensis*) cultivation which are distributed among eight Divisional Secretariat (DS) divisions (Wijesuriya *et al*., 2011). At present, the total extent of rubber smallholdings in Moneragala is about 5,087 ha (9,415 number of holdings) and Moneragala is the fifth rubber growing district according to the land extent under rubber cultivation in Sri Lanka (MPI, 2019).

The Rubber Research Institute of Sri Lanka (RRISL) is the apex body in the country, which undertakes research studies in all aspects of Rubber Farming (RF) and delivers extension service to the smallholder rubber sector. Many recommended farming practices were introduced to Moneragala area by RRISL, out of which, the Manuring of Mature Rubber Cultivation (MMRC) was introduced with the aim of increasing the rubber productivity in a sustainable manner (RRISL, 2003). The recommended fertilizer dosage was 750 g per plant and should be applied in 2 to 4 points, in the area cleared of weeds around the tree within a radius of 100 - 120 cm. The fertilizer should be forked into the top 10-20 cm of soil. From an economic as well as agronomic point of view, fertilizers for mature rubber should be applied within one month after refoliation (depending on weather conditions at the time).

Many knowledge dissemination approaches have been launched by both the public and private sector extension services to enhance the adoption level of MMRC (Gunarathne *et al*., 2020) in Moneragala. The information for Moneragala on the level of adoption of practices in MMRC developed by RRISL is very scarce and not at a satisfactory level (Wijesuriya *et al*., 2011). As mentioned in the research problem, reasonable answers are expected for the research question, in order to recognize the factors, and thus influence the adoption of MMRC in the Rubber Smallholder (RS) level. Wijesuriya *et al.*, (2008, 2010, 2011) have studied RS's expectations, potentials and constraints for RF in Moneragala. However, those previous studies do not specifically focus on MMRC in Moneragala. Therefore, this study examined the factors that influence adoption of MMRC by RSs in Moneragala area. The findings can be used in the designing of more effective policy instruments to get rid of adoption barriers of MMRC in Moneragala, and it can have a greater impact on the rubber sector, and finally on the economic development of the country.

Methodology

Sampling technique

The eight DS divisions that rubber is grown in Moneragala district was selected for this study. RSs who own mature rubber lands were selected employing stratified random sampling technique. Stratification was done on the basis of geographical distribution of RSs in all rubber growing DS divisions (8) in Moneragala district.

The minimum size of the sample was 397, which of the population with 90% confidence interval using Raosoft web-based sampling calculator. Twenty-three percent of the Grama Niladari (GN) divisions, where the highest number of RSs could be found within each DS division, were selected using the statistical sources. Subsequently, the selection of individual RSs was done and the survey sample (RSs of each GN division) was randomly selected based on the number of RSs in each GN division.

Conceptualization of the study variables and their relationships

In an effort to understand intentions of RSs to adopt to MMRC, the Decomposed Theory of Planned Behaviour (DTPB) was applied as a theoretical framework (Taylor and Todd, 1995). The DTPB focuses on the direct measures of Attitude (ATD), Subjective Norms (SN), and Perceived Behavioural Control (PBC) to predict Behavioural Intention (BI) which in turn predicts one's behaviour (Ajzen and Hartshorne, 2008). The DTPB provides increased explanatory power, with a more precise understanding of the behaviour and examination of the relationships among factors that influence the adoption and use of new technologies. The DTPB was applied by many scholars to find out the relationship between belief structures and intentions to adopt in innovations. According to the theoretical framework and literature cited, adoption on MMRC of the RSs can be conceptualized as presented in Figure 1.

Data collection

Both primary and secondary data were collected for this study in 2020. The cross-sectional pre-tested questionnaire survey was carried out to gather the information from RSs based on the objectives of the study. The questionnaire for RSs has consisted of items which measure behavioural factors. A structured direct interview schedule was used in gathering data from RSs, by the researcher himself.

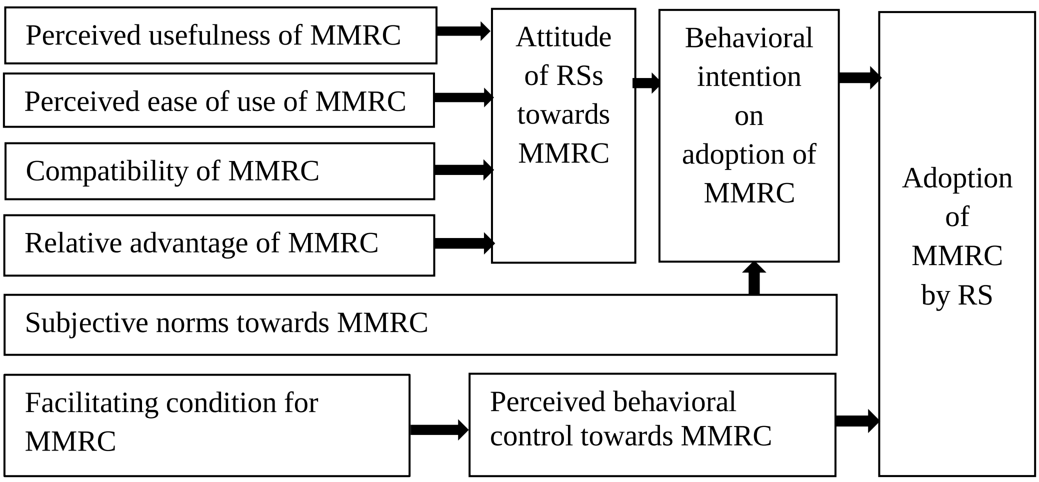

Fig. 1. The conceptual model of the study

Development of hypotheses

Relative advantage (RA)

RA refers to the degree to which an innovation provides benefits which replace those of its precursor and may incorporate factors such as economic benefits, image, enhancement, convenience and satisfaction. RA is an important factor in determining attitude of innovations (Rogers, 1983). In this study, relative advantage refers to financial advantages of MMRC. Therefore, the hypothesis was proposed as an alternate hypothesis, as “perceived RA towards the adoption of MMRC positively affects ATD of RSs” (H_{RA}).

Compatibility (CO)

According to Rogers (1983), CO is the degree to which an innovation is perceived being consistent with the existing values, past experiences and need of potential adopters. An innovation is more likely to be adopted when it is compatible with the value system of an individual. In this study, CO refers to the extent to which RSs believe that application of MMRC would be compatible with their rubber farming practices. Therefore, the hypothesis was proposed as the alternate hypothesis, as “perceived CO towards the adoption of MMRC positively affects ATD of RSs” (H_{CO}).

Perceived usefulness (PU)

Perceived Usefulness (PU) is defined as a person’s subjective evaluation of the extent of using a system which would enhance the productivity. It is likely that, the attitude towards using the technology would become positive, with a higher PU (Ajjan and Hartshorne, 2008). PU has been proven to be antecedent to attitude (Taylor and Todd, 1995) hence, PU is positively related to attitude towards using MMRC. In the context of MMRC, PU would be the degree to which an individual views that MMRC would result in more productivity than previous MMRC. Therefore, the hypothesis was proposed as the alternate hypothesis, as “PU towards the adoption of MMRC positively affects ATD of RSs” (H_{PU}).

Perceived ease of use (PEU)

Perceived Ease of Use (PEU) is defined as the expectation by an individual of the degree to which the target system will be free from effort. Studies have proven that PEU has a direct relationship towards attitude (Taylor and Todd, 1995) hence, PEU is positively related to attitude towards using MMRC. In this study, perceived ease of use refers to the level of the easiness of using MMRC introduced by the RRISL. Thus, the hypothesis was proposed as an alternate hypothesis, as “PEU towards the adoption of MMRC positively affects ATD of RSs” (H_{PEU}).

Attitude (ATD)

ATD is a positive and negative feeling of an individual towards the particular object or towards the intention of performing the particular behaviour (Ajzen and Hartshorne, 2008). According to Rogers (1983) ATD refers to general feeling of an individual on favourableness or unfavourableness towards using an innovation. ATD is linked to behavioural intention as an individual forms psychological intentions to perform behaviours toward which they have a positive feeling. Studies have proven significant direct relationship of ATD towards behavioural intention (Taylor and Todd, 1995) thus, the ATD towards using MMRC will be positively related to the intention to use it. In this study, ATD refers to the feeling of RSs about using MMRC in their rubber cultivation. Accordingly, the hypotheses was proposed as the alternate hypothesis, as "attitude toward the adoption of MMRC positively affects their behavioural intention of RSs" (H_{ATD}).

Perceived Behavioural Control (PBC)

According to (Ajzen and Hartshorne, 2008), Perceived Behavioural Control (PBC) is defined as the level of confidence of an individual about their ability to perform the behaviour based on the difficulty or ease they perceive on its performance as it relates to difficulties or facilitators. It reflects beliefs regarding access to the resources and opportunities needed to affect a behaviour (Ajzen and Hartshorne, 2008). PBC refers to the factors that may encourage the performance of the behaviour. Knowledge is one of the important components of behaviour and it plays a vital role in the adoption in improved technologies. Knowledge becomes power to a person, hence, farmers' technical knowledge determines their ability to reach and find solutions. Adoption levels of fertilizer recommendations, pest and disease management and pruning by tea smallholders show positive relationship with the knowledge level. Study of Perera (2003) on the adoption behaviour of sugarcane growers, revealed that agricultural knowledge and adoption were positively and significantly related. The type of skill has a relationship with the adoption of cultural practices. In this study, PBC refers to adequate level of knowledge and skill of MMRC. Therefore, the hypothesis proposed as the alternate hypothesis, was "PBC of RSs positively affects on adoption of MMRC" (H_{PBC}).

Facilitating conditions (FC)

Facilitating Conditions (FC) is defined, as the environmental factors that influence an individual's desire to perform a task. FC reflects the availability of resources/inputs which are needed to engage in a behaviour. Such resources may include time, money, and other specialized resources needed to perform a particular behaviour. Taylor and Todd (1995) showed that fewer obstacles can produce a feeling of greater control and in turn, positively affect intentions to use technology. Farmers did not adopt certain management practices due to resource limitations. Contact of extension officers with farmers showed a significant relationship with adoption on farming practices. One of the reasons for non-adoption of farm innovations was that the technology which is generated by researchers and disseminated to farmers by extension workers was not accompanied by adequate and timely supply of farm inputs (Gill and Sarda, 1999). In this study, availability of inputs and advisory contacts done by extension officers and availability of training programmes refers to FC for the behavioural intention of MMRC. Therefore, the following hypothesis was proposed as the alternate hypothesis: FC towards the use of MMRC positively affects PBC of RSs (H_{FC}).

Subjective Norm (SN)
According to (Ajzen and Hartshorne, 2008), SN is defined as “the person’s perception that most people who are important to him think he should, or should not, perform the behaviour in question”. The determinant of SN is the sum of normative beliefs which reflects the perceived behavioural views or expectations of important referent individuals or groups. SN denotes that the behaviour is initiated by an individual’s wish to act according to the thought or action of the important referent others. Referent people, such as family members and friends, represent the expectations of other people to perform a particular behaviour, thus they can potentially influence the behaviour (Pantano and Di Pietro, 2012). SN has been observed to be more important in the early stages of implementation, when users have limited direct experience from which to develop the ATD toward the innovation (Taylor and Todd, 1995;). SN refers to the individual’s perceptions of broad social pressure to (or not to) perform the expected behaviour. The social pressure exerted by the significant “referent” others whose beliefs may be important to the individual whom the individual perceives to, (or perceives not to), support the behaviour, conceives more (or less) the likelihood for the individual to perform it (Borotis and Poulymenakou, 2009). The adopter’s family members, friends, and colleagues are groups that will potentially influence adoption. In this study, subjective norms towards adoption of MMRC refers to the influence of Extension Officers, RSs and mass media. Therefore, the following hypothesis was proposed as the alternate hypothesis; The SNs toward the adoption of MMRC positively affect their behavioral intentions of RSs (H_{SN}).

Behavioural Intention (BI)
Ajzen and Hartshorne, 2008 defined BI as a person’s subjective probability in performing certain behaviour. Intention to use is defined as “indications of how hard people are willing to try, and of how much of an effort they are planning to exert, in order to perform the behaviour”. A number of studies have reported a significant and strong relationship of BI being the most important factor in predicting a decision to take a specific action (Ajzen and Hartshorne, 2008). Given this close relationship between intention and behaviour, the past studies have used BI to predict specific behaviour (Ajjan and Hartshorne, 2008). Thus, it is expected that there would be a positive relationship between intention and the actual behaviour of RSs. Therefore, it is hypothesized that the behavioural intention of RSs to use MMRC positively affects their behaviour leading to adoption. In this study, BI refers to the intention to apply the

MMRC in the next season, on a regular basis and to strongly recommend it to others, too. Therefore, the following hypothesis was proposed as the alternate hypothesis; the behavioural intention of RSs positively effects on adoption of MMRC (H_{BI}).

Adoption (ADN)

Rogers and Shoemaker (1971) defined adoption as a decision to make full use of a new idea as the best course of action available. Accordingly, the process of adoption or innovation decision is a psychological process in which an individual move from awareness, interest, evaluation and to trial and finally either to adopt or to reject the practice. In this context, adoption refers to utilization and application of MMRC recommended by RRISL.

Instrument development and measurements

Ajzen and Hartshorne, 2008 was reviewed to generate an initial list of items. Pre-test interviews were conducted with RSs to assess the instrument's clarity and question wordings of proposed items. PU, PEU, CO, RA, SN, PBC and ATD were measured two items while, BI, FC and ADN were measured with three items (Table 1). The responses of RSs to these measurement items were captured on a 5-point Likert scale which ranged from 'strongly disagree' for which a 1 was given to 'strongly agree' for which a 5 was given, indicating the degree to which they agreed with the set of statements. Two-stage model-building process was applied for testing both measurement and structural models (Hair *et al*., 2013). The SMART- PLS 3.2 software was used to confirm and modify the basic hypotheses in the study.

Test of measurement model

The suitable fit for the measurement model was assessed by Communality Test (CT) (Hulland and Business, 1999). The Cronbach's alpha (α), Composite Reliability (CR) and Factor Outer Loadings (FOL) were assessed to measure the reliability, validity and internal consistency of items respectively, for covering the Convergence Validity (CV) (Hulland, 1999). The Average Variance Extracted (AVE) was assessed to measure Discriminant Validity (DV) of items. The Heterotrait–Monotrait ratio of correlations was used to assess DV of questionnaire suggested by Henseler *et al*., (2015). The measurement model was estimated using Confirmatory Factor Analysis (CFA) to test reliability and validity of the measurement model.

Assessment of the structural model

After assessing the reliability and validity criteria for all reflective measurement of the research model and ensuring the integrity of the research data, SMART-PLS Algorithm was applied after determining 300 maximum iterations with stop criterion of 7 using path scheme to maximize the R^2 value for the model on endogenous latent variables (Henseler *et al*., 2015). For predictive purposes, Partial Least Square Approach for the Structural

Equation Modelling (PLS-SEM) was applied. The coefficient of determination (R^2), effect size (f^2), Stone-Geisser (Predictive relevance) index (Q^2) and the path coefficients (β) were assessed using the blindfolding procedure which necessitates the predictive capacity measurements as suggested by Hair *et al*., 2017 and Henseler *et al*., 2015. The cross-validated redundancy method was used to measure the Q^2 by using a blindfolding procedure as recommended by Sarstedt *et al*., 2014. The Variance Inflation Factor (VIF) was applied to assess the Multi-collinearity and inter-correlations among the independent constructs within the structural model (inner model) (Hair, 2016). Both the model goodness-of-fit indices; (SRMR) and Normed Fit Index (NFI) (Hair *et al*., 2016) of the structural model were assessed to examine the model fit.

Hypothesis testing

The conceptualized model was empirically tested using PLS-SEM to evaluate the set of predictive relationships. The Bootstrapping procedure was applied to examine β significance which is a variance based method used to estimate structural equation models, using Smart PLS 3.1. The advantage of using PLS-SEM lies in the fact that no assumption on the distribution of data is needed (Chin *et al*., 2010). The t-statistics were used to test the statistical significance of both the indicators (outer model) and the structural model constructs (inner model). Two-tailed t-test of significance at 5% level was carried out, with t-statistic values larger than 1.96 indicating significance of the structural path significance tests. The results were interpreted with standardized β and coefficients of determination (R^2), with the bootstrap samples set at 5000 and the standard error at a 95% confidence level as suggested by Hair *et al*., 2017.

Results and Discussion

Test of measurement model

The results of CT of items were more than 0.40 and acceptable for suitable fit for measurement model (Hulland and Business, 1999). The Cronbach α values ranged from 0.71 to 0.93, which were above the acceptable threshold 0.70 (Table 1). The FL for all items exceeds the recommended level of 0.6 (Table 1). CR values of all items exceeded recommended level of 0.7 (Table 1). According to α (>0.07), CR α (>0.7) and FL (>0.6) which were greater than standard values, indicated the questionnaire had high reliability, validity and internal consistency. The AVE of all items exceeded recommended level of 0.5 (Sarstedt *et al*., 2014) (Table 1).

According to Henseler *et al*., (2015), DV was well established across all the constructs as no item is cross-loaded higher on another construct than on its own construct. In summary, the measurement model demonstrated adequate communality, reliability, CV, DV and fitness of model.

Table 1. *The results of convergent validity assessment, average variance extracted of measurement and variance inflation factor of items*

Item	Convergent validity			Average variance extracted	Variance inflation factor
	Cronbach's alpha	Factor loading	Composite reliability		
PU1	0.820	0.701	0.766	0.508	1.133
PU2		0.717			1.333
PEU1	0.732	0.585	0.714	0.547	1.271
PEU2		0.668			1.271
CO1	0.793	0.688	0.721	0.666	1.233
CO2		0.780			1.233
RA1	0.715	0.604	0.745	0.565	1.244
RA2		0.892			1.244
SN1	0.844	0.651	0.715	0.504	1.065
SN2		0.730			1.065
FC1	0.852	0.615	0.868	0.594	1.235
FC2		0.602			1.235
FC3		0.734			1.235
PBC1	0.875	0.764	0.745	0.864	1.432
PBC2		0.711			1.432
ATD1	0.766	0.784	0.837	0.765	2.254
ATD2		0.897			2.254
BI1	0.857	0.674	0.761	0.781	1.818
BI2		0.666			1.314
BI3		0.738			1.700
ADN1	0.932	0.812	0.912	0.993	1.882
ADN2		0.782			1.934
ADN3		0.678			1.969

Assessment of the structural model

The R^2 is a measure of the predictive power of a model for the dependent variables (Sarstedt *et al*., 2014). R^2 with a value of 0.67, 0.33 and 0.19 is considered substantial, moderate and low, respectively. R^2 of ATD towards BI, BI towards to ADN and PBC towards to ADN were 0.755 (Substantial), 0.671 (Substantial) and 0.516 (Moderate), respectively.

The R^2 of the structural model of this study was 0.785 and it was considered substantial in line with the recommended value. The four exogenous variables (ATD, BI, PBC, SN) explain 78% of the ADN of SDTS by RSs. Effect size (f^2) of 0.02, 0.15, and 0.35 indicates small, medium, and large effect, respectively (Chin *et al.*, 2010). Four hypothesis (H_{PEU}, H_{ATD}, $H_{PBC,}$ $H_{FC,}$ H_{SN} and H_{BI}) were in the large effect category, whilst the rest (H_{RA}, H_{CO} and H_{PU}) were medium effect size (Table 4). Since Q^2 values for all latent variables (ADN = 0.762, ATD = 0.609, BI = 0.449 and PBC = 0.304) of the inner model were greater than zero

Sarstedt *et al.*, 2014, the path model had a favorable strong predictive relevance. All the VIF values for all the independent latent variables were less than five and fulfill the requirement of data according to the Multi-collinearity test and thus there was no collinearity problem according to Hair *et al.*, 2009. The recommended value of SRMR should not exceed 0.08, while NFI value ranges from 0 to 1, where the closer the NFI to 1 means better fit (Hair *et al.*, 2013). The SRMR value for the study model was 0.074 and NFI value was approximately 0.8. It could be concluded that both indices represent an acceptable fit for the research model and a good fit. The previous analytical and statistical outcomes gave adequate answers to test research hypotheses.

Hypotheses testing

Table 4 shows β, t-values, and p-values for all hypotheses concerning the path analysis supported by empirical data using the bootstrapping method. All nine hypotheses in the conceptual model achieved the β standard. The results showed that RA, had not effect on attitude towards adoption of MMRC and intention to use MMRC for adopters (H_{RA}). CO had positive effects on attitude towards adoption of MMRC (H_{CO}) (t-value=6.826, $p<0.01$). It reveals that RSs perceive practicing the MMRC being consistent with their existing cultural practices of RF, lifestyle and past experience. PU had positive effects on attitude toward MMRC (H_{PU}) (t-value=6.617, $p< 0.05$). PEU had positive effects on attitude towards MMRC adoption (H_{PEU}) (t-value=5.512, $p<0.01$).

Table 4. *Hypothesis test results*

Hypothesis	Path coefficient (β)	Standard Deviation	t-value	Effect size (f^2)
H_{RA}	0.111	0.054	5.678	0.135
H_{CO}	0.488	0.067	6.826**	0.278
H_{PU}	0.384	0.073	6.617*	0.257
H_{PEU}	0.242	0.067	5.512**	0.567
H_{ATD}	0.334	0.067	3.912**	0.843
H_{PBC}	0.566	0.234	3.910*	0.459
H_{FC}	0.647	0.098	4.567**	0.832
H_{SN}	0.866	0.135	6.035***	0.459
H_{BI}	0.344	0.198	5.891*	1.121

Notes: * $p<0.05$; ** $p<0.01$; *** $p<0.001$

However, effect of CO on attitude towards MMRC was comparatively high. Therefore, extension personnel should emphasize the benefits in aspects of MMRC. In addition, extension personnel should provide adequate information and clearer guidance to encourage RSs to use the MMRC in their rubber lands. Eventually, they should educate RSs the benefits of MMRC, using training and awareness programmes covering rubber farming areas in Moneragala area. ATD had significant positive relationship with BI and thus supported H_{ATD} (t-value = 3.912, $p < 0.01$). ATD was also the most influential factor that predicts intention of RSs to use MMRC in this area. This result implies that if RSs have a positive attitude, they will certainly be more attracted to use MMRC. ATD is considered as a powerful factor that motivates to develop a positive intention.

It signifies that when ATD is favourable, that the chances of RSs to use a MMRC will definitely be increased. The H_{SN} hypothesis results (t-value = 6.035, $p < 0.001$) significantly and positively showed the influence of SN on BI. This implies that opinion of specific referent groups is also important in the development of an intention towards the use of MMRC. This result can be explained by the fact that the MMRC have been just introduced to Moneragala, and hence the knowledge of RSs remains rather limited. As a result, they may consult people from their own social environment to seek for advice in the process of adoption of this new MMRC. The positive relationship also denotes that, the more a person has a favourable social influence in ADN of MMRC, that the more favourable his intention would be. The H_{PBC} hypothesis of this study that PBC affected on ADN of MMRC was positively and significantly supported (t-value = 3.910, $p < 0.05$). The H_{FC} hypothesized relationship *i.e.* that of FC and PBC too was significant. Hence, H_{FC} too was supported. The H_{BI} hypothesis of relationship of BI to ADN was significantly and positively supported too (t-value = 5.891, $p < 0.001$).

As effects of SN on BI towards MMRC were comparatively higher than that of AT, policy makers should give more attention to improve the SN, which could be helped by improvement in the advisory services. Effect of PBC on ADN was seen to be comparatively higher than that of BI. This result concludes that RSs in Moneragala are likely to engage in practicing the MMRC, when they have the required resources to perform the behavior. Therefore, the availability of necessary fertilizer requirements in the market and skills of MMRC should be improved to enhance adoption of MMRC among the RSs in Moneragala.

Conclusion

The nine-predictor conceptualized model explained 78% of the variance in adoption of MMRC of rubber smallholders in Moneragala. This study identified eight behavioral factors; perceived usefulness, perceived ease of use, compatibility, behavioral Intention, attitude, subjective norms, facilitating condition and perceived behavioral control that act as drivers for adoption of MMRC. In order to enhance the adoption of MMRC, a favorable environment contributing to these psychological factors should be improved through proper extension and development plan in rubber smallholder sector in Moneragala.

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Preparation of Liquid NR *via* microwave irradiation as performance improving agent in tyre carcass compounds

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Abstract

In this research, a new process for preparation of liquid natural rubber (LNR) by means of microwave irradiation heating was investigated. Natural rubber (NR) obtained in the form of crepe rubber was first subjected to mastication in the presence of a peptizing agent. Peptizing agent used was pepton 4040 and its optimum concentration was first established as 3 phr. Micro wave irradiation heating was carried out for 10 minutes to heat the per-masticated NR incorporated with optimum concentration of peptizing agent in the presence of different loading of the micro wave absorber, Polyethylene glycol (PEG). According to the results, optimum microwave absorber loading was 4 phr. LNR prepared was characterized by means of Fourier Transmission Infrared Spectroscopy (FTIR). LNR was also characterized for its viscosity and molecular weight. Significant reduction in molecular weight confirmed the effectiveness of the process employed. Based on the results, the combination of 3phr pepton and 4phr PEG is suggested for microwave assisted LNR preparation within 10 min heating period. LNR prepared was then incorporated in a tyre carcass rubber compound. The compound was cured and the physical properties of the tyre carcass was evaluated. Addition of LNR improved the processeability of the compounds. Results also suggest that incorporation of LNR prepared following the new method could be used to improve the rubber to metal adhesion, tear strength and tensile strength of tyre carcass compounds.

***Key words*:** Liquid NR, microwave absorber, microwave irradiation, peptizer agent, tyre carcass compounds

Introduction

Natural rubber (NR) itself is used in product industry with the incorporation of certain ingredients to achieve desired processing and physical properties when products are manufactured out of them. Various additives such as peptizing agents, processing aids, curative ingredients, protective system, reinforcing agents, cheapeners and specific compounding ingredients are incorporated to NR to make a coherent homogeneous mass referred to as rubber compound, which is subsequently processed into a desired product (Barlow, 2020). These rubber compounds are then subjected to vulcanization process in moulds where formation of crosslinking between the macromolecules takes place under heat. The vulcanization causes initially a decrease in its plasticity and the increase in elasticity while enhancing resistance to environmental stresses upon vulcanization. Vulcanized compounds referred to as rubber end products are capable of giving desired properties with improved service performance.

In dry rubber based product manufacturing industry including tyre industry, NR is initially masticated using high powered mixing mills to assure uniform and well dispersed coherent mass of additives in NR phase. A majority of these ingredients are added in dry form in preparation of rubber compounds. As the first processing aid, a rubber compounding ingredient, typically known as peptizer is added to improve the mastication efficiency of NR. Peptizers which could act as radical acceptors at low temperature, decrease the molecular weight of the rubbers by facilitation of breaking rubber macromolecular chains (Moneypenny *et al.*, 2004). A sulphide (Pepton 4040) (Fig. 1) is a common peptizing agent used in the rubber industry.

In the next stage, another class of low molecular weight organic materials such as processing oils and Plasticizes are added as processing aids to improve processing and achieve uniform dispersion of ingredients, particularly fillers in the compound. Conventional processing oils are mainly derived from

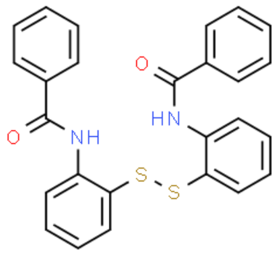

Fig. 1. Chemical structure of 2,2'-Dibenzamido Diphenyl Disulfide (Pepton 4040)

petroleum oils and coal tar fractions. This class of processing aids offers multiple benefits such as reduction of viscosity of rubber compounds, reduced power consumption, improved likability, shortening the mixing cycle, ability to incorporate higher filler loading and certain improved product properties as well as improved product appearance (Chokanandsombat *et al.*, 2013; Jayawardena *et al.*, 2009). Sometimes, these processing aids carry certain inherent disadvantages too. They generally reduce the tensile and tear strength of vulcanizates and sometimes tend to bloom to the surface. Moreover, with the present growing public concerns on the health and environment risk, most of these petroleum oil based conventional processing aids used are restricted for use as they contain toxic chemicals such as polycyclic aromatic hydrocarbons (PAHs) *etc.* (Anon 2005). Therefore, researchers in the sector have been paying attention in finding alternative non-extractable green processing aids for rubber compounding. Jatropha seed oil, natural oils and LNR are some of the environmentally friendly processing aids investigated in such attempts (Intharapat *et al.*, 2020; Chandrasekara *et al.* 2011; Nasruddin and Susanto 2018; Ren *et al.* 2020). Out of them, LNR, a depolymerized form of NR having shorter polymeric chains has few unique advantages as a processing aid for NR (Lloyd 1991). In addition to its ability to offer most of the functions of other types of processing aids, LNR could co-vulcanize with the main rubber phase. It also acts as a green processing aid providing a straight and sustainable solution for environmental concerns and restrictions associated with conventional petroleum based processing aids. LNR has also been used to achieve useful properties in a wide range of other applications such as compatibilization of natural rubber blends and toughening of plastic materials [Dahlan *et al.* 2000; BenSaleh *et al.*, 2014). It could be used as an adhesion promoter at the interface between NR layer and fabrics chiefly found in tyre carcass of radial tires too. LNR being a cost effective and more compatible processing aid would be a sustainable alternative to eliminate drawbacks of metal containing compounds and inorganic based chemicals currently used as adhesion promoters for NR compounds.

LNR is manufactured by breaking down of long chain rubber molecules into stable short chains. It is found in literature that there are various degradation processes carried out to transform larger NR molecules into useful low molecular weight materials (MW, below 50,000 g/mol). Some of these processes are Mechano-chemical breaking down, oxidation and photodegrdation of NR (Ibrahim 1993; Ibrahim *et al.* 2021; Chaikumpollert *et al.* 2011).

Microwave is a form of electromagnetic radiation and could consider as an environmentally friendly heating source suitable for bringing chemical bonds to higher energy levels (Dudley 2015). Heating methods using microwave is becoming an increasingly used source of energy in different applications particularly in organic reactions due to its simplicity in practical use alone with other inherent benefits. Formela *et al.* (2019) and Aoudia, *et al.* (2017) have reported the use of microwave radiation for recycling of NR waste. Pre-curing of NR-compounds using microwave energy at a frequency of 2.45 GHz has been reported by Makul *et al.* (2010) who also introduced a new method to pre-cure NR compounds. Recently, microwave technique has also been used to modify LNR in another study (Idris *et al.* 2019). Although there is evidence on the use of microwave radiation in different polymer associated applications as shown in above studies, there is no evidence available on utilization of micro irradiation as an energy source in preparation of LNR from conventional NR in the presence of a peptizer. This method is expected to be a simple and cost effective route for preparation of LNR. No much evidence available in literature on the effect of low molecular weight LNR on the important service performance of NR based tyre carcass compounds, when it is used as processing aids.

Therefore, objective of the present study is to investigate a new route for preparation of LNR through microwave assisted molecular breakdown of peptizer incorporated NR. The performance of prepared LNR in tyre carcass compounds with special attention to metal to rubber adhesion properties was also studied.

Material and Methods

Materials

Pepton 4040 and Polyethylene glycol (PEG 4000) were supplied by Loadstar Pvt. Ltd, Ekala, Sri Lanka. Crepe rubber (TPC-1X) was supplied by Rubber Research Institute of Sri Lanka (RRISL), Dartonfield, Agalawatta. N-hexane (99.5%) and Toluene (99.5%) were supplied by the Department of Chemistry University of Sri Jayewardenepura Sri Lanka.

Methodology

This research work consists of two stages, *i.e.* Preparation of LNR and LNR incorporated rubber carcass compounds. The schematic flow diagram of the experiment is presented below (Fig. 2).

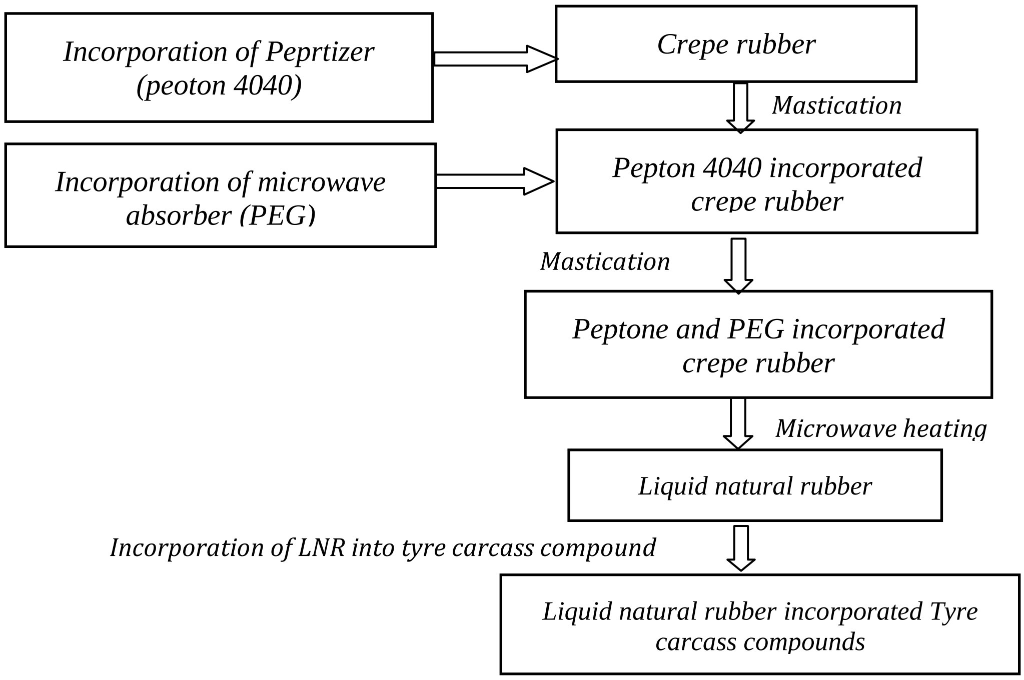

Fig. 2. Preparation of LNR

Preparation of masticated rubber

In this work, a disulphide based peptizer, namely dibenzamide diphenyl disulphide (Pepton 4040) was used as a mastication promoter. Different concentrations of Pepton 4040 were used (Table 1). Subsequently, crepe rubber-peptizer mix was masticated using a lab two roll mill (David Bridge & Co. Ltd., England, size 150 - 300 mm) at 28 ^{0}C ± 2 with a zero nip gap for four minutes. The masticated samples were allowed to cool, and Mooney viscosity was measured. Optimum peptizer concentration was selected for further studies.

Table 1. *Composition of natural rubber and different peptizer doses used*

Crepe/rubber (g)	PEPTON4040/phr
200	0.5
200	1.0
200	2.0
200	3.0
200	4.0

Study of microwave absorber concentration

Polyethylene Glycol (PEG4000) was used as the microwave radiation absorber. Varying doses of PEG 4000 [0.5phr-6 phr] were added with 200 g of crepe rubber incorporated with Pepton-4040 at the optimum concentration found in the previous study. They were mixed for 2 minutes in the Brabender plasti-corder maintained at 40 rpm and 100 ^{0}C. Then each product was further masticated for 3 minutes using the same lab two roll mills with zero nip gap. Masticated product was kept in the domestic microwave oven (Samsung 28L ME6104ST-1) for 10 minutes at 850W microwave power level to allow further molecular breakdown (Table 2).

Each of the resultant products were immersed in hexane for 6 hours and then in water to purify the sample. The purified LNR was then removed and dried in a heating oven at 70 ^{0}C for one hour. Optimum dosage of PEG determined from this study was used to prepare LNR for further studies.

Table 2. *Recipes used to study the effect of PEG concentration*

Sample No	PEG4000/Phr	Microwave power, 850W (ON/OFF)
Control	0	ON
A	0.5	ON
B	2.0	ON
C	4.0	ON
D	6.0	ON

Characterization of LNR

Viscosity average molecular weight of LNR was determined by using viscometric method following ASTM D2857 standard. Fourier Transmission Infra-red Spectroscopy (FTIR) analysis was carried out using an FTIR spectrophotometer (Nicolet IS50, Madison, WI, America) to identify functional groups on LNR chain.

Preparation of LNR incorporated tyre carcass compounds

A general tyre carcass formula was used to study the performance of developed material as an adhesion promotor. Different proportions varying from 5phr to 20 phr of prepared LNR were added to the selected formula. All recipes used were given in Table 3.

Table 3. *LNR incorporated tyre carcass compounds*

	Control	**F5 (5 pphr)**	**F10 (10 pphr)**	**F15 (15 pphr)**	**F20 (20 pphr)**
TSR 20	70	70	70	70	70
Reclaimed rubber	20	20	20	20	20
LNR	-	5	10	15	20
Styrene butadiene rubber BR 1502	25	25	25	25	25
Butadiene rubber	10	10	10	10	10
Powdered Rubber Crumbs	30	30	30	30	30
N 330 Carbon black	32	32	32	32	32
N 550 Carbon black	8	8	8	8	8
Powdered Silica	5	5	5	5	5
Zinc Oxide 99.5%	3	3	3	3	3
Stearic acid	2	2	2	2	2
6PPD [a]	0.8	0.8	0.8	0.8	0.8
Vulkanox HS-TMQ[b]	0.8	0.8	0.8	0.8	0.8
Penacolite B 19S	1	1	1	1	1
Sulphur	1.40	1.40	1.47	1.50	1.54
Insoluble Sulphur 80	1.56	1.56	1.64	1.67	1.72
Vulkacit H 30	0.11	0.11	0.12	0.12	0.12
MBTS[c]	0.26	0.26	0.27	0.28	0.29
CBS[d]	1.00	1.00	1.05	1.07	1.10
TMTM[e]	0.13	0.13	0.14	0.14	0.14
PVI[f]	0.35	0.35	0.37	0.37	0.39
Total PPHR	212.41	217.570	222.6505	227.7467	232.891

[a] p - phenylenediamine type antiozonant, [b]2,2,4 - Trimethyl-1,2-Dihydroquinoline,
[c]2-2'-Dithiobis(benzothiazole); [d]N - Cyclohexyl-2-benzothiazole Sulfenamide;
[e]Tetramethlthiurammonosulphide, [f] n - Cyclohexylthiophthalimide

Control was prepared using general formulation without any modification. Sample F5 to F20 were prepared varying the LNR level from 5 phr to 20 phr at 5 phr intervals. Total Sulphur content in LNR modified samples were adjusted to be in par with rubber: sulphur ratio of the control compound.

Preparation of rubber compounds

There are two stages at the compounding.

I. First stage mixing
II. Second stage mixing

First stage mixing

2.0 kg of Rubber and other chemical ingredients except accelerator were mixed using an internal mixer (Farrel BR1600 Banbury® Mixer) for nearly three minutes and then the mixture was taken out from the internal mixer. The compound was further milled on a two roll mill used in earlier studies with 8 rounds of milling for 2 minutes and about 3 mm thick sheets were obtained. These sheets were allowed to cool for 2-3 hours, before the second stage mixing was commenced.

Second stage mixing

Compounds (Sheets) from the first stage were cut in to medium size pieces and mixing of same was carried out in the same internal mixture for 45 seconds, before the accelerator was fed and further mixed for 60s. The final compound was again rolled 8 rounds on the two roll mill. All samples were prepared following the same method as described above. Mooney viscosity, cure characteristics and other mechanical properties were then studied.

Characterization of the compound

Mooney viscosity at 1st stage

Mooney viscosity of the samples was measured following ASTM D1646-17 standards. Mooney viscosity at 1st stage was studied as it indicates the plasticity of the mix that gives an idea how easy to mix chemicals to the rubber compounds.

Cure characteristics

The cure characteristics of samples were tested at 150 ^{o}C using an oscillating disk rheometer (ALPHA ODR 200) following ASTM D6601 standards.

Tensile and tear properties

Testing for Tensile and tear properties were done using a tensometer (Gotech/Loyd) by using dumb bell test specimens following ASTM D412 standards. Cross-head speeds used were 500 mm/min and 50 mm/min for tensile and tear strength tests respectively.

Adhesion strength properties

Unaged adhesion strength tests were done following according to ASTM D429 standards and aged adhesion/peel tests were done following ASTM D572-04 standards. Test species were kept in an air circulated ageing oven at 70 ^{0}C for 3 days for ageing tests.

Results and Discussion

Effect of pepton (4040) concentrations on Mooney viscosity of masticated rubber

Mooney viscosity values of crepe

rubber masticated after addition of different concentrations of Pepton 4040 are represented in Figure 3. Mooney viscosity of untreated crepe rubber was 56.67 MU. During mastication, long chain rubber molecules were broken down to short chains with reactive terminal ends due to mechanical stress applied on NR (Dimier 2004). These broken short chains with reactive ends could react with each other and recombined. When Peptizers are added, its free radicals formed during mastication can react with reactive ends of short chain rubber molecules and form stable short NR chains preventing the recombination. Therefore, in the presence of peptizers, flow properties of masticated samples improved as rubber molecules with lower molecular weights are stabilized restricting their recombination.

According to Figure 4, the lowest Mooney value is 14.56 MU which was registered at 3 phr of Pepton 4040, beyond which, there is no significant reduction in the Mooney viscosity with further increase in the peptizer level. Maximum efficiency of prevention of recombination may have been attained when the above concentration of pepton 4040 was used. Therefore, masticated crepe rubber with 3 phr was found to be the best among the candidate phr levels of pepton 4040.

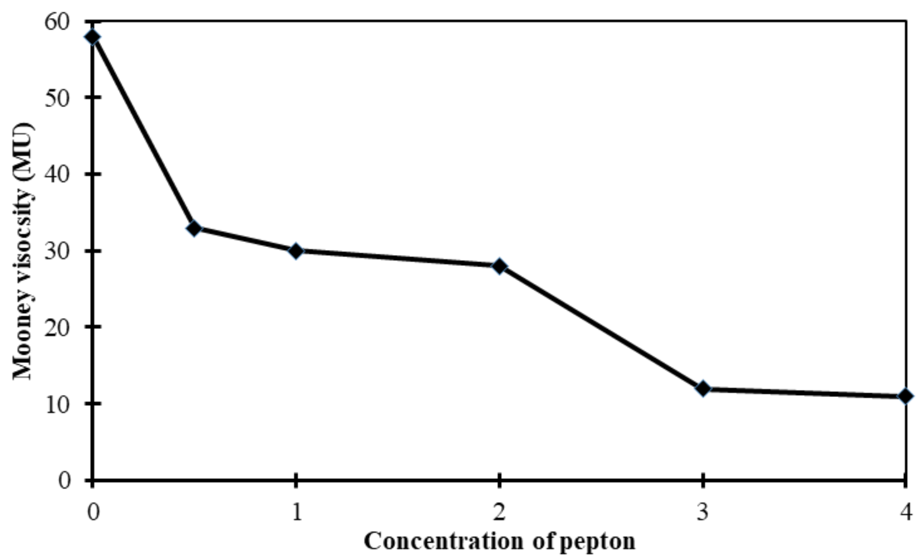

Fig. 3. Effect of Pepton loading on molecular weight

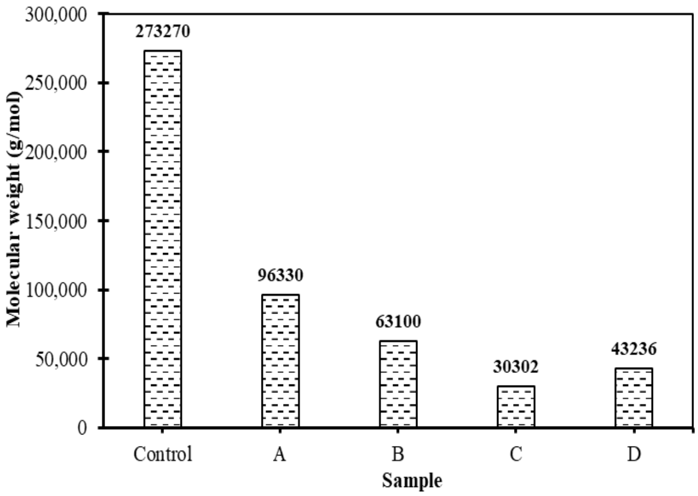

Fig. 4. Effect of PEG loading on molecular weight

Effect of concentration of PEG 4000

The effect of concentration of PEG 4000 on the viscosity average molecular weight of the masticated samples with 3 phr pepton concentration after 10 minutes microwave exposure is shown in Figure 4. Molecular weight of the masticated crepe rubber without incorporation of either the peptizer (pepton 4040) or microwave absorber (PEG) labeled as (control) is also seen in the same Figure. It could be seen that the molecular weight of the control was 273,720 g/mol while all treated samples exhibit significantly lower molecular weights. The high molecular weight of masticated NR devoid of free peptizers and microwave absorbers suggests the poor mastication efficiency. The gradual reduction of molecular weight of rubber with a constant concentration of peptizer (3 phr) with increasing amount of PEG concentration clearly shows the efficiency of PEG as a microwave absorber. PEG has a high dielectric constant and therefore, it is sensitive for microwave heating (Phinyocheep and Duangthong 2000). PEG quickly absorbs microwave radiation and transfers thermal energy to the rubber compounds. As a consequence, long rubber chains could be oxidized to short ones. The reactive ends of these short chains could be stabilized with reactions between O_2 and peptizer radicals. When PEG level exceeds 4 phr, molecular weight of NR again tends to increase (sample D). It could be due to the generation of increased number of reactive ends of NR chains under higher thermal energy generated in the presence of higher level of PEG and with fixed level of peptizer content allowing recombination of already shorten molecules.

Among the peptizer and microwave absorber incorporated samples, Sample "C'' (3 phr Pepton, 4 phr of PEG) exhibited the lowest molecular weight among the candidate samples and therefore, this ratio could be considered the optimum combination of peptizer and microwave absorber.

Analysis of FTIR spectrums of NR and LNR

FTIR spectrums are used to identify functional groups of LNR.

FTIR spectra of NR and LNR show similar pattern except for few peaks (Fig. 5). Major peaks of NR are C=C stretching vibration of 1,4 unit,1667cm^{-1}, CH_2 deformation 1446 cm^{-1}, out-of-plane blending vibrations of C-H 835 cm^{-1}[22-23]. FTIR spectrum of LNR (sample "C") shows extra peaks appear around 3000-3500 cm^{-1} and 1700-1750 cm^{-1}. These peaks relate to O-H, (3400 cm^{-1}) and C=O (1720 cm^{-1}) respectively. It must note that a very narrow band is appeared in the spectrum for C=O group, though it is expected to appear a broad band. However, disappearance of peak at 887 cm^{-1} attributed to vinylidene group (-C=CH_2) is a clear evidence of oxidation C=C bond. Other major observations were reduced intensity of 1667 cm^{-1}, 1444 cm^{-1} and 836 cm^{-1} These peaks are related to the C=C bond , C-C bond and =C-H bond respectively. According to the FTIR spectrum, LNR has C=O and O-H groups as functional groups indicating the formation of reactive sites. These functional groups could be the result of thermal degradation of NR chain. CH_2-CH_2 is the weakest bond of the rubber chain. Cleavage of this bond could yield hydroxyl terminals and cleavage of C=C could result carbonyl groups in LNR (Shao *et al.* 2013). The reduction of the intensity of C=C, C-C and =C-H bonds and appearance of additional C=O and OH bonds shown in FTIR spectrum suggest conversion of NR to LNR.

LNR incorporated rubber compounds and vulcanizates

Compositions of all formulations used in this study are given in Table 3 and the summary of Mooney viscosity of rubber compounds are depicted in Figure 6.

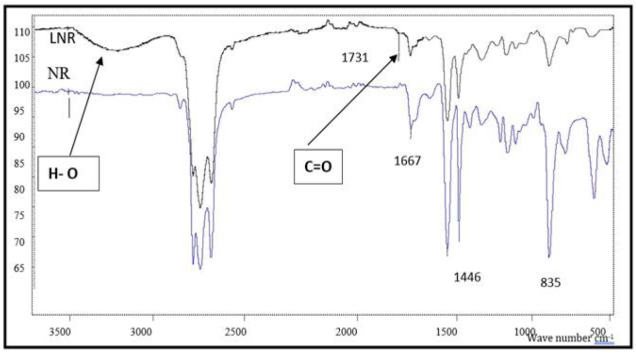

Fig. 5. FTIR spectra of NR and LNR

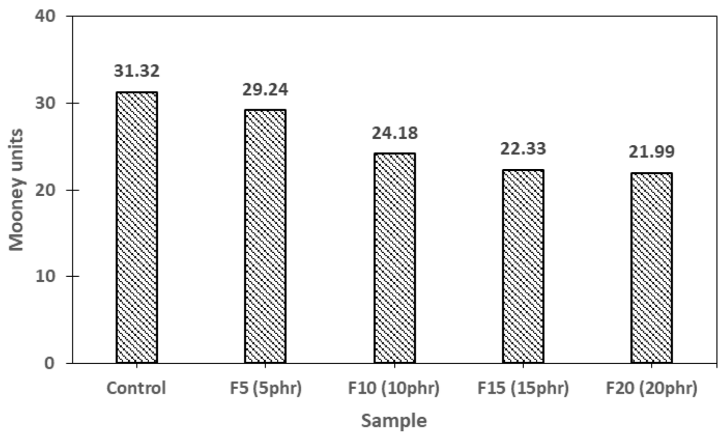

Fig. 6. Mooney viscosity of LNR modified rubber compounds

Mooney viscosity values decreased as modified LNR content in the compound was increased. However, the effect of reduction of the Mooney viscosity diminishes as the LNR content increases. As other conventional processing aids, LNR is a low viscosity material having short chains of NR and could behave as lubricant in the medium. Low viscosity enhances the flow properties of the compound which helps to increase processeability, to shorten processing cycle and to bring down process energy consumption. It also leads to enhanced product quality and consistency achieved through uniform and efficient mixing of ingredients during product processing process.

Curing characteristics of LNR modified samples

Addition of LNR into a rubber compound reduces the viscosity of rubber matrix due to increased percentage of low molecular weight LNR in the rubber compound (Table 4). It can be seen that addition of even a small portion of 5phr of LNR, a significant reduction in M_L is observed indicating the processing advantages of incorporating LNR. As mentioned earlier, this is due to the plasticizing effect offered by the shorter NR molecules. However, it shows that there is no significant effect on the reduction of M_L value in LNR incorporated rubber compounds with further addition of LNR. This is almost comparable to the trend shown for the Mooney viscosity values of the compounds (Fig. 6). The crosslink density of a rubber vulcanizate is represented by MH-ML value. The crosslink densities as represented by (MH-ML) of the samples show no significant variation with the incorporation of LNR except for F20 sample. However, as the LNR content is increased up to 20 phr, a considerable increase in crosslink density could be observed. All the compounds maintain same ratio of cross linking agent and curing agent/accelerator ratio. Improved curing ingredient dispersion in the compound along with increased percentage of curable rubber (as LNR) in the compound may have contributed to this increment in the cross link density. It is also clear from the results presented in the table that incorporation of LNR has no effect on the scorch time or cure time of the compound, as could be expected since no extra accelerating or activating chemical substances are added with LNR.

Table 4. *Curing characteristics of LNR modified rubber compounds*

Rheometer 150c/30min	Control	F5 (5phr)	F10 (10phr)	F15 (15phr)	F20 (20phr)
MH	80.14	76.88	77.68	75.67	82.25
ML	13.68	11.67	11.69	11.01	11.09
MH-ML	66.46	65.21	65.99	64.66	71.16
tS_2	2.36	2.41	2.34	2.56	2.45
t90%	6.13	6.49	6.40	6.5	6.01

Adhesion strength

As it could be seen in Figure 7, adhesion strength (unaged and aged) has increased with the addition of LNR. The rubber content in the medium is increased by addition of LNR which act as a curable adhesion promotor. Low viscosity induced by addition of low molecular weight NR will increase in the flowbility thereby introducing more bonds between rubbers to textile interface. LNR having low molecular weight (shorter length molecular chains of rubber) has higher penetration power to pass through nylon rubber interface making effective bonds between rubber and fabric. In addition, LNR used could co-vulcanize with the main rubber phase and make a strongly bonded rubber to fabrics phase. Application of other conventional processing aids has no such ability.

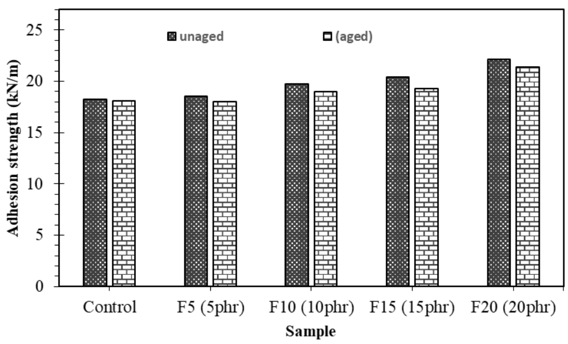

Fig. 7. Mooney viscosity of LNR modified rubber compounds

Tear strength

Tear strength increased with the increase of LNR content beyond 5 phr (Fig. 8). As the rubber phase increases in the compound, filler distribution and compatibility between the two rubber phases (NR and LNR) could be improved offering better mechanical properties. It is interesting to note that even though LNR has low molecular weight, addition of LNR to NR phase and vulcanization do not seem to result in reduction of mechanical properties, instead, it has enhanced the mechanical properties. This highlights the unique advantage of using LNR as a processing aid in NR compounds as the co-vulcanization of LNR and NR could result in better crosslinking network which, in turn, could enhance mechanical properties of the vulcanizate.

Tensile strength

It could be seen from Figure 9 that tensile strength of the LNR incorporated vulcanizates showed an initial reduction followed by a graduate increase similar to the trend shown for tear strength. Initial reduction may be due to the dilution effect of the vulcanizate with LNR. As mentioned above, as the percentage of LNR is increased, the total percentage of rubber in the mass is increased with improved dispersion of the filler materials and other ingredients added at fixed loading in all formulations. It is well reported that improved filler dispersion could increase the tensile strength (Surya *et al.* 2018). In addition, separately added LNR forms a fully miscible rubber phase which is cross linked along with higher molecular weight NR as a single phase material. This factor may explain the gradual increase in the tensile strength and showed the additional advantage of use of LNR as a processing aid in NR compounds. It is reported that when LNR is incorporated to epoxy composites where a two phase system is formed, tensile strength has shown an increment only at the low LNR loading and further addition has resulted in a continued decrement in the tensile strength which is an opposite trend to the observation made in this study (Seng *et al.* 2011). Therefore, this observation provides evidence for the advantage of ability to crosslink the processing aid used (LNR) along with the based polymer (NR) offering both processeability and improved properties.

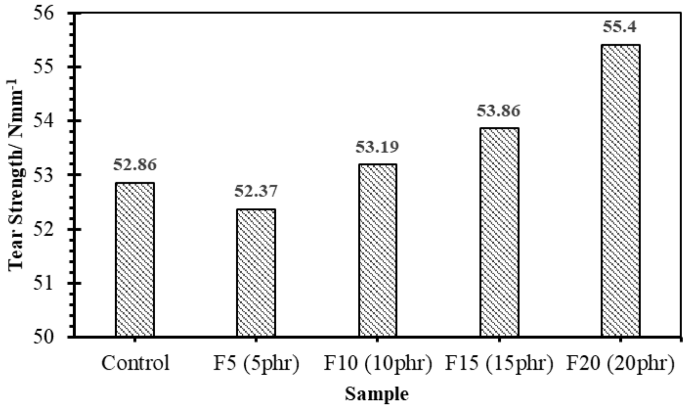

Fig. 8. Tear strength results of LNR modified Sample

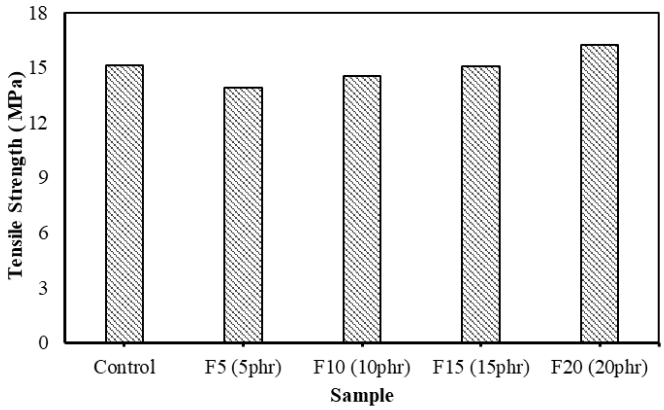

Fig. 9. Tensile strength of LNR modified Sample

Conclusion

NR was masticated in the presence of a peptizer (Peptone 4040) and also with a combination of a Pepton 4040 and a microwave absorber (PEG). It was found that efficiency of molecular breakdown of NR through mastication in the presence of peptizer (Pepton) could be enhanced by application of microwave heating in the presence of suitable microwave absorber (PEG). There are optimum dosages for both peptizer and Peptone to obtain the maximum efficiency. Optimum concentrations of Pepton 4040 and PEG were found to be 3 and 4 phr respectively. Therefore, a microwave assisted LNR manufacturing process with following conditions could be proposed to produce LNR;

(i). Formula: NR: 100; Pepton 4040: 3phr; PEG 4000: 4phr, (ii). Heating conditions: Microwave heating period: 10 min., microwave power level: 850W

Curing studies showed that incorporation of LNR has no adverse effect on the curing characteristics. Incorporation of LNR into carcass compounds improves both aged and un-aged adhesion properties, tensile and tear properties while improving the processeability of the compound.

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A study of different tapping times on latex production in smallholder rubber fields in Moneragala District in Sri Lanka

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Abstract

Harvesting latex from rubber trees at different times in the early hours of the day or so-called night tapping has led to more time to extract latex from trees and saves time for the tapper to engage in other farm activities during the daytime in non-traditional areas in Sri Lanka. A night tapping trial was conducted during the 2017-2018 period for one year in Moneragala District (IL1c) to investigate whether night tapping is advantageous for farmers to obtain higher latex yields from their fields. In this study, two smallholder rubber fields were selected in Moneragala District, and trees were tapped at S/2 d2 tapping system without rain guards. For each starting time, 10 trees were allocated for the collection of latex in each treatment, and treatments were set up at five different time intervals i.e., 1.30, 2.30, 3.30, 4.30, and 5.00 for field 1 and from 1.00 am to 5.00 am hourly intervals for field 2. Tapping time at 5.00 am was considered as the control treatment for both fields as the normal tapping time in Moneragala. A forehead-mounted torch was used by each tapper to illuminate the tapping cut. Results revealed that there was a variation in latex yield tapped from 1.00 am to 5.00 am in both fields. The present trial indicated that the latex yields are given at different tapping times before 4.30 am comparatively low in pre-dawn tapping.

Key words*:* climate, early, rainfall, weather, yield

Introduction

As far as technological developments in rubber cultivation are concerned, the improvements in harvesting practices have the longest history and also slow and uninterrupted developments over the years. Starting from ad-hock wounding made to take the latex out from Amazonian wild rubber trees up to robot-driven harvesting machines today demonstrate the interest of the scientists and growers in harvesting practices of rubber. Whatever the method of harvesting or the devices used, the basic principle is the same; the opening of microscopic latex vessels in which the latex is stored and located in the bark of the tree making the latex ooze out (Priyadarshan, 2011).

The time of tapping of rubber trees is usually the daytime mostly as the day dawn, and sometimes later in the day due to rain interferences in the morning (Priyadarshan, 2011). Due to a shortage of tappers, each tapper is given two tapping blocks and the second block is tapped in the afternoon.

One reason to start tapping in the morning hours is the higher latex yield collected. The time was decided by the amount of light available for the tapper to see the tapping cut to perform tapping without wounding the tree (Nayanakantha, 2021). Another valid reason is for the tapper to come to the field and move about without facing accidents if started in the dark. As the day goes by, a reduction of the latex yield is expected by 10-15%, but the amount of reduction depends on the weather condition of the day.

However, the biggest challenge in front of the scientists, industrialists, or engineers right from the beginning of the rubber industry was to develop a knife or a device to tap the tree without wounding the tree or the bark and also to be used without any skill on tapping. However, in the recent past, farmers and scientists thought of the advantages if tapping was done at the night. It has one main advantage if the tapper engages in another job during the daytime. Nevertheless, even if a person needs a rest, one can engage in any activity depending on the task to be undertaken. Then which job is to be done at the night should be judiciously decided. Rubber tapping in the traditional rubber growing areas is not easy even during the daytime due to uneven terrain with rock outcrops drains and ditches, covered with weeds and cover crops. Also, snakes are normally out, searching for food during dark hours. Scorpions, Centipedes, Porcupines, and even Wild bores are common in rubber lands.

Tapping quality is the most important factor for the amount of latex that can be harvested from the tree as well as for the life span of the tree. The life span of a commercial plantation is determined by the availability of tappable bark. If the tapper cannot see the panel or the tapping cut properly, correct tapping without wounding is a challenge. However, in non-traditional rubber growing areas in Sri Lanka, namely Moneragala and Ampara, the cultivable lands are flat and the land owner himself taps the rubber trees in the early hours and engages in his usual on-farm activities during the daytime. Since rubber cultivation in those areas is new and not popularized, demand for a job like a “rubber tapper” is not experienced yet. As the knowledge on proper tapping, *i.e.* getting the potential crop while making minimum damage to the tree, is poor in this area due to rubber being a new plant to the people of the region, there are many miss-concepts and miss-beliefs. One such is night tapping yields high latex yield. Therefore, the main objective of this trial was to see whether a higher crop can be obtained by tapping in the early hours than usual 5-6 am. Some stateowned large estates are available in the Moneragala area but this study focused on smallholder farmers in Moneragala District.

Methodology

Two smallholder farmers' fields were selected for this trial in Moneragala District belongs to the Agro-climatic Zone of IL 1. Details of the two fields where the trial was conducted are given in Table 1. As given in Table 1, trial periods were different for the two fields. Due to the low rubber price prevailing after about 10 months of the commencement of the trial, tapping of Field 2 was stopped and the data collection had to be discontinued after 10 months. Data collection from Field 1 was continued for 18 months. Different starting times of tapping of each field, which were the treatments are given in Table 2. Tapping time at 5.00 a.m. was considered as the control for this trial for both fields. The reason to test two different tapping times for each field was to swap between the fields in order to supervise, when tapping fell on the same day, due to no tapping in one field.

Both fields were tapped at the S/2 d2 tapping system without rain guards. Trees were randomly selected with a total number of 50 trees and there were five blocks of each with 10 trees for each tapping time. For a given tapping day, each block had been assigned with one tapping time (Table 2) and latex was collected from 10 trees for each block at each time tapped; DRC values were estimated and thereby g/t/t was calculated. In order to alleviate the influence of tree-to-tree variation for latex yield, the tapping time was changed every new day according to a roster for each block. In this manner each set of 10 trees was tapped at all five tapping times in order to minimize any errors. A forehead-mounted torch was used by each tapper to illuminate the tapping cut. The latex volume of individual trees was measured daily by using a measuring cylinder. Metrolac reading was taken daily from the bulk sample after collecting latex from each treatment block and dry rubber content of the latex yield was calculated in relation to gram per tapper per tapping (g/t/t). Rainfall data was recorded from a non-recording type rain gauge which was installed at the Moneragala Substation of RRISL. Statistical analysis was done by the analysis of variance followed by a mean separation procedure, Duncan's Multiple Range test (DMRT), at a probability level of 0.05. SAS statistical software package – version 9.0 (SAS Inc., USA) was used to analyze data.

Table 1. *Details of the two rubber fields used for the trial*

Field	Location	Clone	Extent (Ha)	No of rubber trees	Year of planted	Year of tapping	Tapping panel	Type of rubber manufactured	Trial period
1.	Unawatuna Buttala	RRIC 121	1.01	520	2007	2016	A	Sell latex	From 07.04.2017 to 15.11.2018
2.	Yundaganawa Buttala	RRIC 121	0.81	440	2006	2016	A	Produce RSS	From 18.05.2017 to 06.03.2018

Table 2. *Starting time of tapping of two rubber fields*

Field 1	Field 2
1.30 am	1.00 am
2.30 am	2.00 am
3.30 am	3.00 am
4.30 am	4.00 am
5.00 am	5.00 am

Results

The yield data collected for the period specified for the two fields are given in Fig. 1a and 1b. Since the two fields were not identical in tapping time, micro-climate, and agro management, yield data are presented separately in Figs.1a and 1b.

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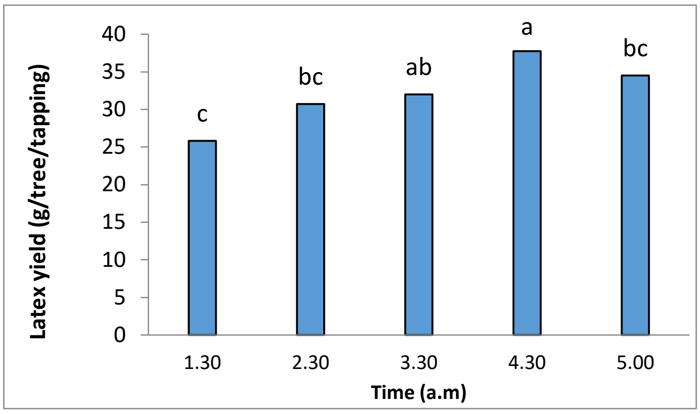


Fig. 1a. The mean yield of trees tapped at hourly intervals from 1.30 am to 5.00 am (Field 1)

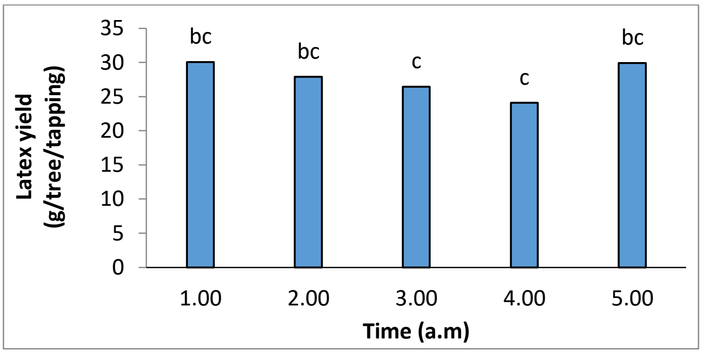
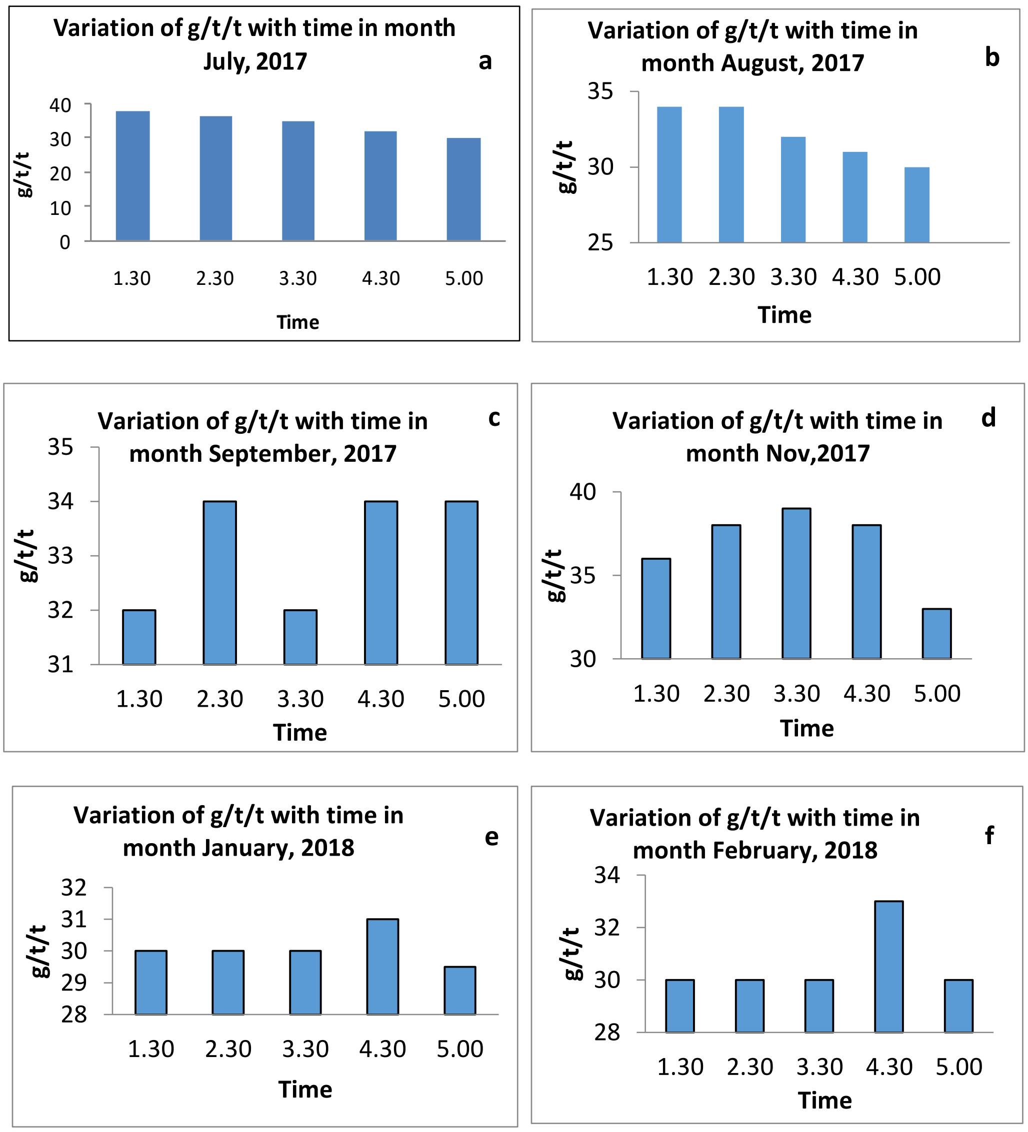


Fig. 1b. The mean yield of trees tapped at hourly intervals from 1.00 am to 5.00 am (Field 2).

The data collected on tapping revealed that there was variation in latex yield obtained at different tapping intervals (Figs. 1a &b). In field 1, a yield increase could be obtained when tapped from 4.30 as compared to that from other tapping times (Fig. 1a). In Field 2, latex yield is comparably low ($p \leq 0.05$) when compared with the Field 1 due to the changes in micro-climates (Fig. 1b). Data collection in Field 2 was terminated after 10 months due to lack of interest of the smallholder with price dropdown at the study period. Data collection was continuously done in Field 1 for one year starting from July 2017 to June 2018. The mean latex yield of the trees of Field 1, tapped from 1.30 am to 5.00 am from July 2017 to June 2018 period are shown in Figs. 2a to 2i.

Tapping was undertaken throughout the study period except for a few days during the heavy rains that prevailed in second inter-monsoonal and North-East monsoon periods from October and December 2017 to April 2018. According to Figures 2a to 2i, there was no marked yield increase observed during the early hours before 4.30 am. Monthly rainfall during the study period was recorded from the meteorological station established at the Moneragala Substation of Rubber Research Institute of Sri Lanka. Monthly rainfall data are shown in Fig. 3.


Variation of g/t/t with time in month July, 2017
a
g/t/t
40
30
20
10
0
1.30
2.30
3.30
4.30
5.00
Time
Variation of g/t/t with time in month August, 2017
b
g/t/t
35
30
25
1.30
2.30
3.30
4.30
5.00
Time
Variation of g/t/t with time in month September, 2017
c
g/t/t
35
34
33
32
31
1.30
2.30
3.30
4.30
5.00
Time
Variation of g/t/t with time in month Nov,2017
d
g/t/t
40
35
30
1.30
2.30
3.30
4.30
5.00
Time
Variation of g/t/t with time in month January, 2018
e
g/t/t
32
31
30
29
28
1.30
2.30
3.30
4.30
5.00
Time
Variation of g/t/t with time in month February, 2018
f
g/t/t
34
32
30
28
1.30
2.30
3.30
4.30
5.00
Time

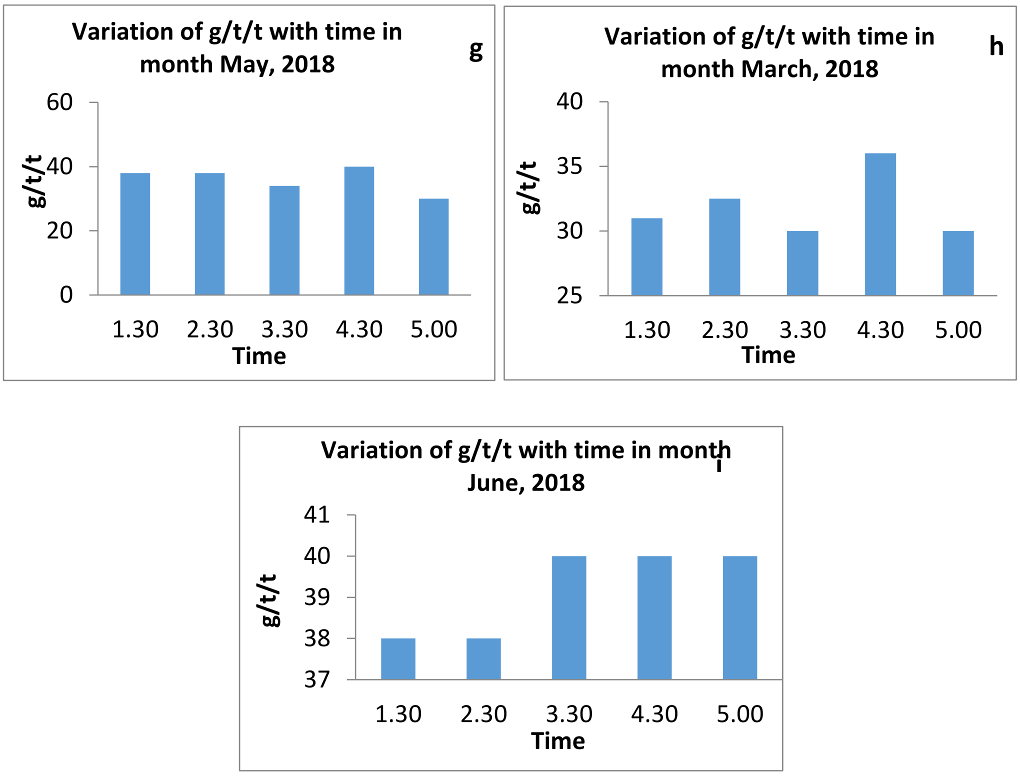


Fig. 2. Latex yield variation of Field 1, tapped from 1.30 am to 5.00 am from July 2017 to June 2018 (**a -i**)

A high monthly rainfall variation was observed during the study period with distinct dry months *i.e.;* July-September 2017, January-February, and June 2018 (Fig. 3). Tapping was not commenced in some months (October and December) under heavy rains as the trees were not rain-guarded. Nearly 60% of the rainfall was experienced during the second inter-monsoonal (October – November) and North-East monsoon rainy season from December to February (Fig. 3).

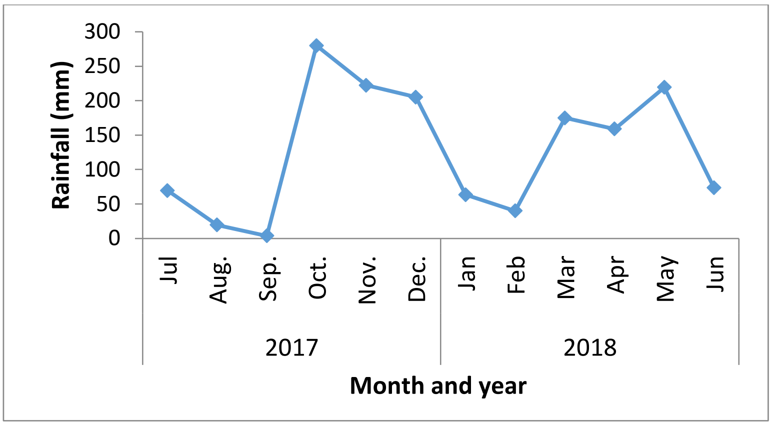


Fig. 3. Monthly rainfall during the study period (from July 2017 to June 2018)

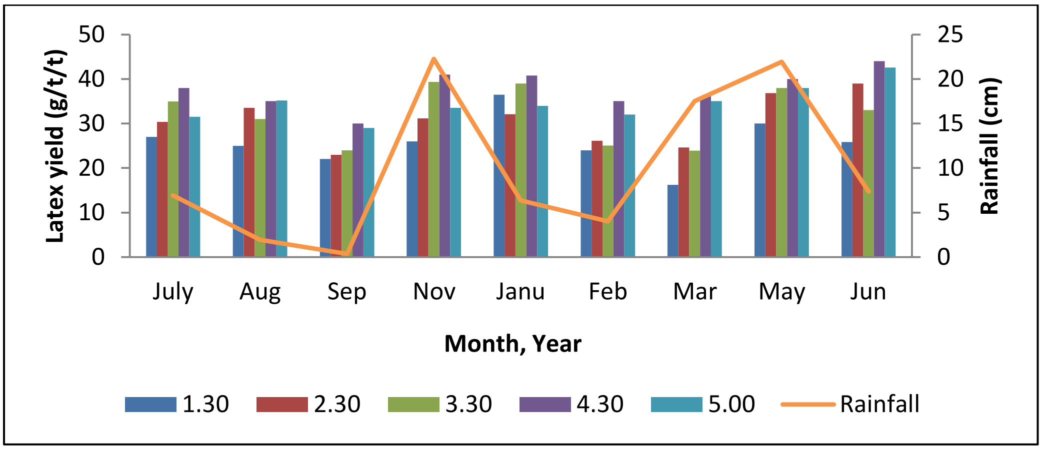


Fig. 4. The latex yield when tapped at different times from 1.30 am to 5 am in Field 1 from July 2017 to June 2018 (Bars show SEM values)

As seen in Fig. 4, it is revealed that the latex yields are given at the tapping time before 4.30 am comparatively low in pre-dawn tapping. The yield difference in every month is occurred due to a positive correlation between monthly variations in rainfall.

Discussion

Results obtained from the trial conducted over a period of one year revealed that there was a variation in latex yield when tapped from 1.00 am to 5.00 am in both fields in the smallholder fields in the Moneragala district. The time of tapping is generally determined by the time of dawn of the day as the tapper should see the tapping cut clearly in order to do proper tapping without wounding the bark or to avoid shallow tapping. A higher yield obtained at dawn was discussed by Priyadarshan (2011) due to its direct bearing on the turgidity of the tree with transpiration being at a minimum in the early hours of the day under high atmospheric pressure and high relative humidity. However, the climatic conditions in the Moneragala area are adverse and marginal with regard to rainfall experiencing with marked dry periods that affect the growth of rubber and latex yields.

A similar study was conducted by Nayanakantha *et al.* (2022) in Wet and Intermediate Zones for a one-year period in 2019 to see if there is any effect of time on the latex yield in Wet and Intermediate Zones of Sri Lanka. Accordingly, they have tested 3.00 am to 8.00 am in hourly tapping intervals for Wet Zone and 2.00 am to 7.00 am for the Intermediate Zone. The overall results of that study revealed that there were no significant differences among different time intervals in latex yield. According to Nayanakantha *et al.* (2022), there was a very insignificant increase in latex yield by 0.04% when trees were tapped from 5.00 am to 7.30 am in wet regions. However, the results reported in the same study revealed that, about 5.1% yield increment resulted for trees tapped between 2.00 am and 4.30 am in the Intermediate Zone. Another study conducted in the wet region of Kalutara district, in Dartonfield estate for about one year revealed a negative impact on the yield when tapped at 3.00 am (-5.76%) and 4.00 am (-1.87%) and a higher yield when tapped at 5.00 am (Anon, 2019). A study conducted by the Rubber Research Institute of Sri Lanka in the Eastern province has reported that tapping before 6.00 am yields a higher latex volume. But dry rubber content of latex has increased after 6.00 am tapping attributed to the water status of the tress which is governed by evapotranspiration (Kudaligama *et al.*, 2019). As reported by Haridas (1985) rubber plantations transpire 4-6 mm of water vapor daily when soil moisture availability is adequate and only 2-4 mm when it is inadequate. Rao and Vijayakumar (1992) also explained that the distribution of rainfall, temperature, sunshine, and humidity are the major conditions contributing to yield variability. About 40-62% of the total variation in monthly rubber production could be explained by prevailing environmental factors as reported by Jiang (1988). This is on par with the monthly variation observed in the latex yield than the variation or the pattern observed among different times of tapping in the present study (Figs. 3 & 4), indicating a stronger relationship with the rainfall and its related parameters reported for the month. Rao *et al.*, (1998) have carried out a commercial-scale trial with RRII 105 which is considered drought tolerant. Trees have been tapped at S/2 d2 and the tapping operation has been taken place from 7.00 am to 11.00 am. They have concluded that the maximum temperature, sunshine duration, vapor pressure deficit, and pan evaporation had a negative correlation but a significant positive correlation with rainfall.

Latex yield variation within the day is often correlated with the turgor pressure in lactiferous phloem tissues. There are more weather related factors such as relative humidity influencing turgor pressure. Riches and Gooding (1952) have reported early morning pressure falling during the day and recovering at night. They correlate the diurnal pressure changes with atmospheric relative humidity. Negative correlations with changes in temperature, evaporation, leaf water deficit, and stomatal opening are also demonstrated by them. The loss in turgor is explained probably as the result of the withdrawal of water from the phloem tissues under transpiration stress. A general turgor gradient from the base to the crown does not preclude mass flow in sieve tubes in the opposite direction provided that the rates of loading and unloading are such that a sufficient osmotic gradient is maintained in them in the required direction, as reported by them. The latex contains 65-70% of water and hence the moisture stress influences the yield. Heavy rainfall for a prolonged period, however, can also have a negative effect on yield. Shorter duration of sunshine, associated with heavy rainfall results in low photosynthetic efficiency. Heavy rainfall also occurs nutrient loss. Therefore, the results obtained during the study do not indicate any marked effect of tapping time on latex yield. But seasonal changes associated with climatic factors were experienced. Furthermore, the quality of tapping has a high impact on the latex yield than many other factors such as time of tapping. Tapping is a skilled job and good eyesight is considered an important factor to perform proper tapping. Similarly, the availability of enough light for the tapper to see the tapping cut is equally important. This is the main reason to commence the tapping operation as the day dawn. With the personal experience gained in conducting the present trial, even though head-mounted torches were used to tap in the dark, it was difficult to maintain the correct depth of the cut and the angle of the tapping which resulted in low yields. The poor tapping quality resulted after about a year or two made some estates abandon early tapping, which was done at 3.30 - 4.00 am in the Wet Zone. Even though the early tapping saves the tapper's time more at day time, the terrain in the rubber fields in the Wet Zone does not permit tapping in the dark due to the difficulty faced by the tapper to walk from tree to tree in the uneven and rocky fields and the other, venomous and carnivorous animals such as scorpions, snakes, *etc.* found under the weeds in the dark hours. Snake bites are often reported by rubber tappers and taking them to a hospital may challenging at night. Though the terrain is flat in the Moneragala area, the dangerous animals searching for prey and water at the night is common. In estates and most of the time in the smallholder sector also, only the tapper is in the field without a helper or a supporter. A higher percentage of tappers is female and night tapping may create social security issues too. In conclusion, the present trial indicated that the latex yields given at different tapping times before 4.30 am are comparatively low in pre-dawn tapping.

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