

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 processability 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 Plasticizers 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

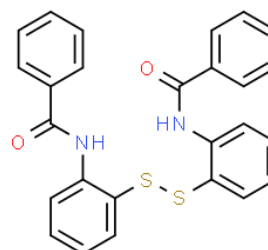


Fig. 1. Chemical structure of 2,2'-Dibenzamido Diphenyl Disulfide (Pepton 4040)

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 photodegradation 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 along 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).

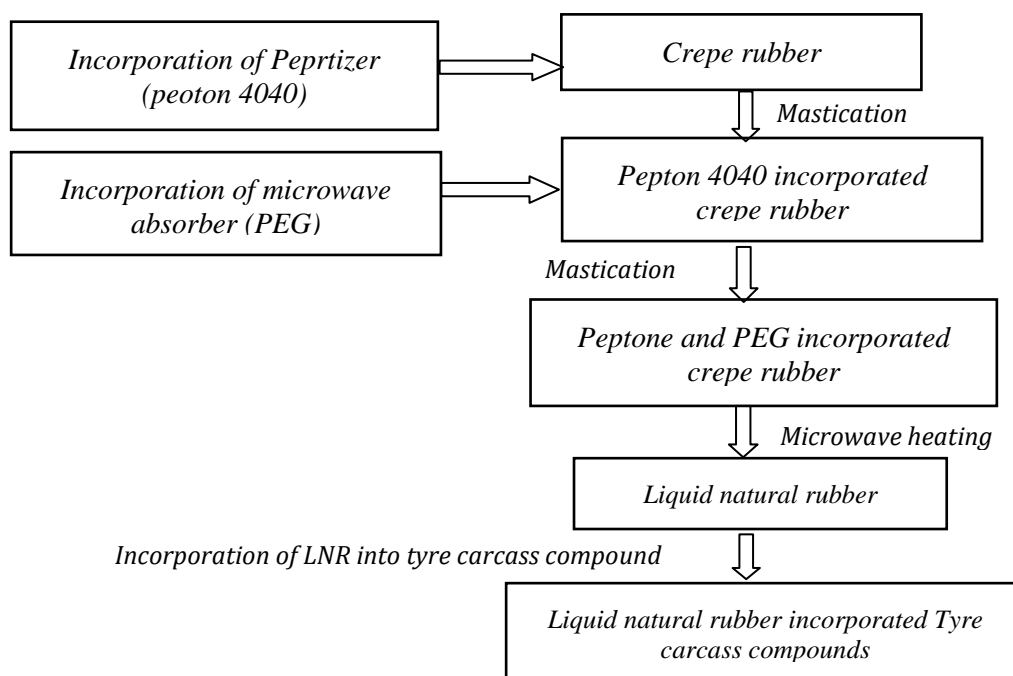


Fig. 2. Preparation of LNR

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 $28^{\circ}\text{C} \pm 2$ 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°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°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,

^c 2,2'-Dithiobis(benzothiazole);

^e Tetramethylthiurammonosulphide,

^b 2,2,4 - Trimethyl-1,2-Dihydroquinoline,

^d N - Cyclohexyl-2-benzothiazole Sulfenamide;

^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 °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 °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.

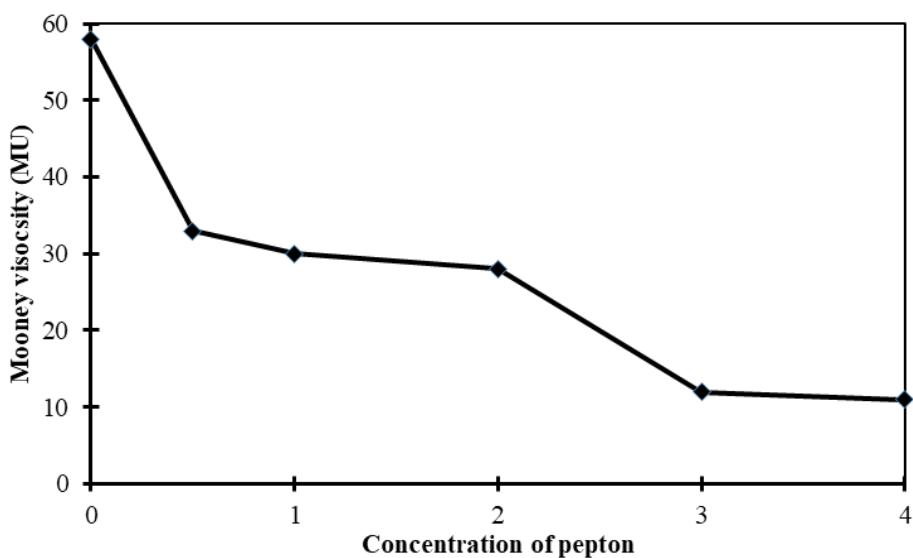


Fig. 3. Effect of Pepton loading on molecular weight

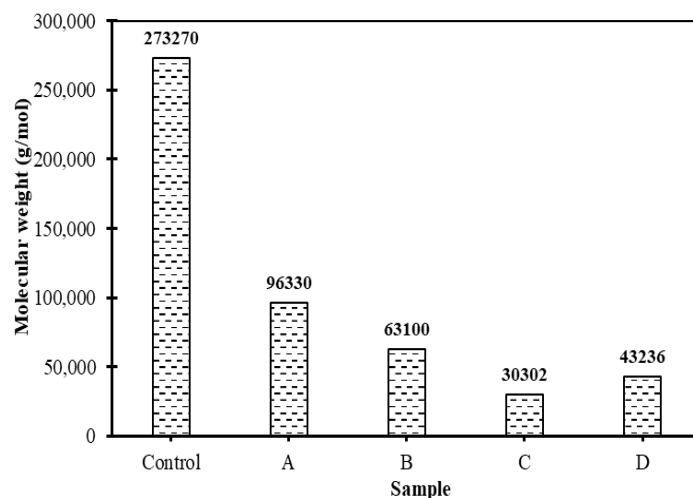


Fig. 4. Effect of PEG loading on molecular weight

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 1446cm^{-1} , out-of-plane bending vibrations of C-H 835cm^{-1} [22-23]. FTIR spectrum of LNR (sample "C") shows extra peaks appear around $3000\text{-}3500\text{cm}^{-1}$ and $1700\text{-}1750\text{cm}^{-1}$. These peaks relate to O-H, (3400cm^{-1}) and C=O (1720cm^{-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 887cm^{-1} attributed to vinylidene group ($-\text{C}=\text{CH}_2$) is a clear evidence of oxidation C=C

bond. Other major observations were reduced intensity of 1667cm^{-1} , 1444cm^{-1} and 836cm^{-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. $\text{CH}_2\text{-CH}_2$ 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.

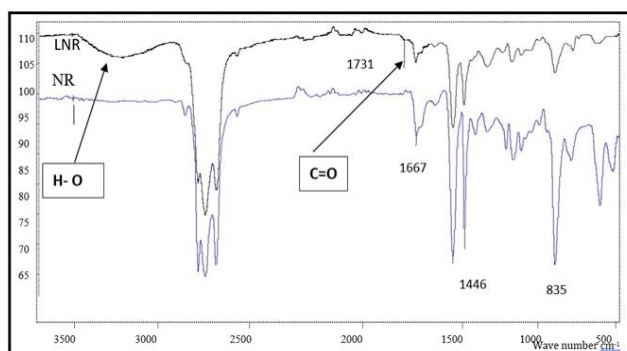


Fig. 5. FTIR spectra of NR and LNR

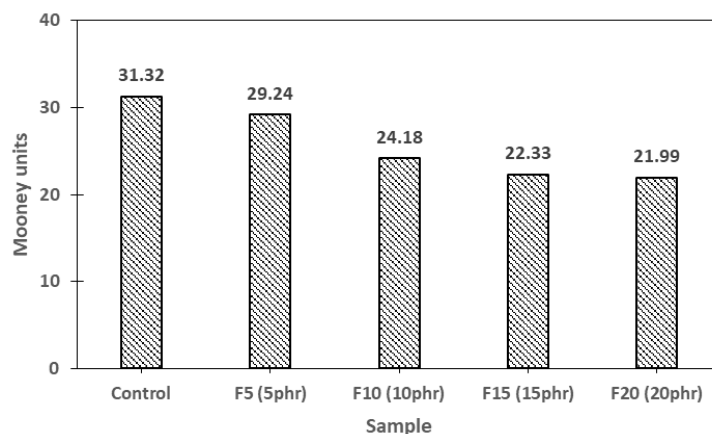


Fig. 6. Mooney viscosity of LNR modified rubber compounds

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 processability, 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
t _{S2}	2.36	2.41	2.34	2.56	2.45
t _{90%}	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.

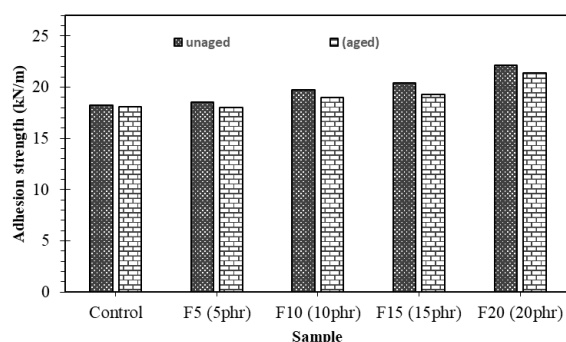


Fig. 7. Mooney viscosity of LNR modified rubber compounds

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 processability and improved properties.

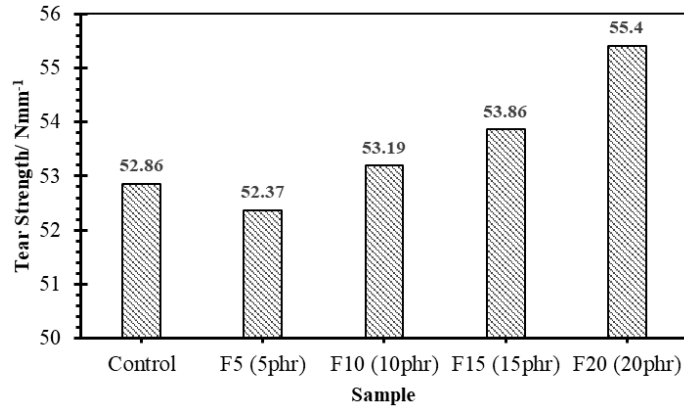


Fig. 8. Tear strength results of LNR modified Sample

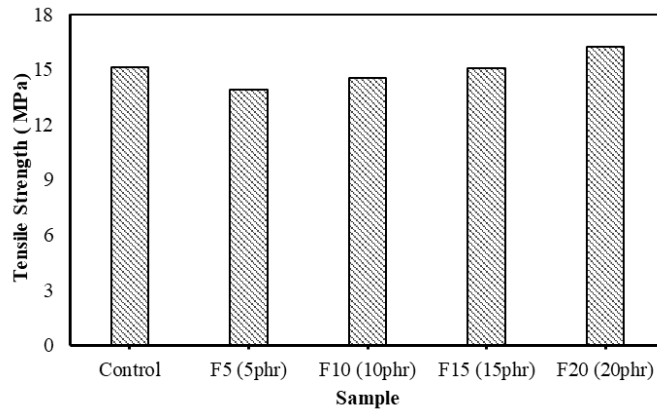


Fig. 9. Tensile strength of LNR modified Sample

Conclusion

NR was masticated in the presence of a peptizer (Pepton 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 Pepton to obtain the maximum efficiency. Optimum concentrations of Pepton 4040 and PEG were found to be 3 and 4 prh respectively. Therefore, a microwave assisted LNR manufacturing process with following conditions could be proposed to produce LNR;

- (i). Formula: NR: 100; Pepton 4040: 3prh; PEG 4000: 4prh,

- (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 unaged adhesion properties, tensile and tear properties while improving the processability of the compound.

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