

## **INFLUENCE OF KRAFT LIGNIN ON THE PROPERTIES OF RUBBER COMPOSITES**

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### **ABSTRACT**

The influence of lignin content on reclaimed rubber (RR)/natural rubber (NR) blend composite properties has successfully been studied. Scanning electron microscopy (SEM) were used to understand morphology. Fourier-transform infrared spectroscopy (FTIR) for the possible chemical interaction, whereas thermogravimetric analysis (TGA) and tensile tester were used to predict strength and elongation for possible practical applications. The results indicated that the presence of lignin forms cavities which seemed to arise from complex interactions of the blend with the lignin. Those cavities dominated tensile fractured surface and the increase in lignin indicated inconsistencies of interfacial interactions. Lignin RR/NR blend composites revealed a drop in tensile strength and shift in glass transition temperature, except for the highest lignin containing blend composite. More active interactive constituent of the blend appeared to be NR. The interaction has not favored the thermal stability and crosslinking density.

**KEYWORDS:** Blend composites, polymer composites, lignin, biomass, lignin-polymer composites.

### **INTRODUCTION**

Recently, there has been renewed interests in the utilization of lignocellulosic components such as cellulose lignin and hemicellulose to advance green applications of materials (Mohomane et al. 2017, Sibiyi et al. 2018). Some researchers compared modified characteristics of celluloses from different sources (Linganiso et al. 2019), whereas some tested potential of the modified surfaces in different polymeric matrices (Sibiyi et al. 2018). Of more interest

is the employment of lignin, a complex, amorphous-thermoplastic material as a filler, modifier and reinforce in polymeric materials such as rubbers and plastics. Due to its abundance and versatility compared to inorganic fillers, lignin has gained a lot of attention (Datta et al. 2017, Frigerio et al. 2014, Yu et al. 2015, Liu et al. 2015, Botros et al. 2005, Barana et al. 2016, Sen et al. 2015). Lignin has been reported to have interesting properties which include flame retardant abilities, antioxidative characteristics, biodegradability, antimicrobial behaviour and adhesive capabilities. Furthermore, the structure of lignin contains a variety of chemical functional groups that positively influence its reactivity, making it able to meet the needs of most rubber industries (Sen et al. 2015, Setua et al. 2000, Jiang et al. 2014, Košíková et al. 2007, Košíková et al. 2005, Gregorová et al. 2006, Kumaran et al. 1978).

A lot of research has been done on the incorporation cellulose to formulate composites that could be applied industrially. For instance, Che et al. (2018) utilized a water-soluble copolymer for wood modification, while Furuno et al. (2004), Klüppel and Mai (2013), Yu et al. (2011) small molecular sizes, low molecular weight urea formaldehyde and phenol formaldehyde resins. Those materials can effectively penetrate through wood micropores and grafting and/or crosslinking with wood cells to enhance the dimensional stability and other properties of wood. Lignin is one component of lignocellulosic material which also received fair attention as a filler in polymer blends. For instance, as a filler in natural rubber (NR) Yu et al. (2015), Setua et al. (2000), Jiang et al. (2014) and Košíková et al. (2005, 2007) studied NR composites filled with sulphur-free lignin under sulphur vulcanization. The addition of lignin up to 30 phr into the NR rubber composites resulted in improved physico-mechanical properties such as 100% increment in modulus, elongation at break and tensile strength. It was also reported that lignin influenced the cure characteristics (increased scorch time and decreased optimum cure time) of the NR compounds. They further demonstrated that the significant improvement in the study was observed when the NR was filled with 20 phr lignin. This observed positive effect on the mechanical properties of NR vulcanizates was attributed to the relatively low molecular mass and polydispersity of the lignin which allowed for a good lignin-NR miscibility. Similar results were observed by Gregorová et al. (2006). They argued that the lignin influences the retention of physico-mechanical properties of non-aged natural rubber system in the small concentrations. Setua et al. (2000) also showed that reinforcing nitrile rubber with modified lignin resulted in better thermal stability when compared against phenolic resins or carbon black. It was shown that lignin is a more capable replacement to the costlier petroleum derived carbon black and phenolics subsequently imparting on the lignin-rubber composite superior oil resistance. Košíková et al. (2007) has demonstrated through SEM micrographs, the morphological properties of NR vulcanizates with and without lignin which showed that lignin enhanced the dispersion of other compounding ingredients in the mix.

There is little or/and no information regarding the influence of lignin on the blend of reclaimed rubber (RR)/ natural rubber (NR). The novelty of this study is based on systematic incorporation of lignin in the blend. The blend composites were analyzed by scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR) thermogravimetric analysis (TGA) and tensile tester.

## MATERIAL AND METHODS

### Materials

Kraft (alkali) lignin in the form of brown powder, was obtained from Sigma Aldrich, South Africa. It has average molecular weight of about 10 000. Reclaimed rubber was obtained

from EnvanDe Rubber, Pietermaritzburg, South Africa. It has specific gravity of 1.15, tensile strength of and elongation at break of natural rubber (SMR 20) was obtained from S&N Rubber, Port Elizabeth, South Africa.

### Preparation of blends and composites

All samples were prepared by twin roll mill at room temperature for 15 min before hydraulic pressing into sheets. The blend homogeneity was achieved at about 15 min prior lignin addition, at which the time counting of mixing blend composites was started. Preliminary results recommend 90:10 ratio due to homogeneity as confirmed in literature (Sen et al. 2015, Kumaran et al. 1978, Tibenham et al. 1954). A mixture design was used to formulate the different blend composites as shown in Tab. 1 below.

*Tab. 1: Formulations used in preparation of lignin-rubber blends and composites by mixed design.*

Abbreviations	Reclaimed rubber (g)	Natural rubber(g)	Lignin (%)
RR/NR blend	90	10	0
10 ppfr	90	10	10
20 ppfr o	90	10	20
30 ppfr	90	10	30

### Scanning electron microscopy (SEM)

Morphological analysis of the composites was characterised by SEM. The micrographs were taken using an FEI Quanta 200 (FEI Co., Eindhoven, the Netherlands) electron microscope operated at an accelerating voltage of 15 kV. All samples were fractured in liquid nitrogen, sputter-coated with gold and allowed to dry before taking images.

### Fourier-transform infrared spectroscopy (FTIR)

The FTIR spectra were collected by using a Perkin Elmer FTIR spectrometer in the diffuse reflectance mode. The samples were analysed in the spectral region between 4000 and 400  $\text{cm}^{-1}$  with a 4  $\text{cm}^{-1}$  resolution.

### Thermogravimetric analysis (TGA)

Thermogravimetric analyses were performed using TGA analyser unit (Perkin Elmer), under flowing nitrogen atmosphere at flow rate of 20  $\text{ml}\cdot\text{min}^{-1}$ . Approximately 10-15 mg of sample was heated from 30°C to 700°C at a heating rate of 10°C  $\text{min}^{-1}$ .

### Dynamic mechanical analysis (DMA)

The dynamic mechanical analysis (DMA) Q800 of the PP and composites were investigated from 40 to 180°C by 3-point bending mode with 15 mm sample size at a heating rate of 5°C $\cdot\text{min}^{-1}$  and a frequency of 1 Hz.

### Tensile testing

Measurements of tensile strength were carried out on three dumb-bell shaped samples, width of 1 mm using a Universal Testing Machine QC 505 with a crosshead speed of 500  $\text{mm}\cdot\text{min}^{-1}$  and a 500 N load cell. Median values of at least 5 samples per test were used for data analysis

### Swelling experiments

Swelling was studied in toluene according to ASTM D 471-79. A sorption-desorption

method was used to determine the swelling behavior of the rubber samples; this being demonstrated as the mole percent uptake of toluene by a gram of rubber at room temperature. The rubber sample was cut into 20 x 20 mm for measuring and calculation of swelling percentage.

## RESULTS AND DISCUSSION

### Scanning electron microscopy (SEM)

SEM micrographs of the tensile fractured surface of lignin RR/NR blend composites are shown in Fig. 1. The 10 pphr (Fig. 1a) of lignin in the blend composite generally showed smaller pore sizes, compared to 20 pphr (Fig. 1b) and 30 pphr ( Fig. 1c), ranging from approximately 50 to 173  $\mu\text{m}$ . Whereas 20 pphr lignin indicated pore sizes ranging from approximately 70 to 400  $\mu\text{m}$ . Surprisingly the pores generally seemed to reduce (most are smaller from 50 to 294  $\mu\text{m}$ ) in sizes for the 30 pphr blend composites and the surface appeared rougher. In fact, there are clear potholes or pits in Figs. 1b,c which maybe an indication of a departure of a material during fracturing. There is a limited information on a study involving lignin blend with reclaimed rubber, however Ramarad et al. (2015) studied lignin reinforced rubber composites. Formation of pits, large cavities and grooves were dominating their tensile fractured surface. They related the observation to the interaction of alcohol and phenolic groups in lignin with mostly unvulcanised part of the rubber. The same explanation is highly possible in the current study, however the natural rubber seemed to be more interactive with lignin than the reclaimed rubber. In fact, this is logical because reclaiming rubber is known for reducing molecular weight and promotes a partial degradation that limit pendent groups for more interactions (Barana et al. 2016, Gregorová et al. 2006, Asrul et al. 2014). Therefore, some of pores may have resulted from an escape of natural rubber when interacting with the lignin.

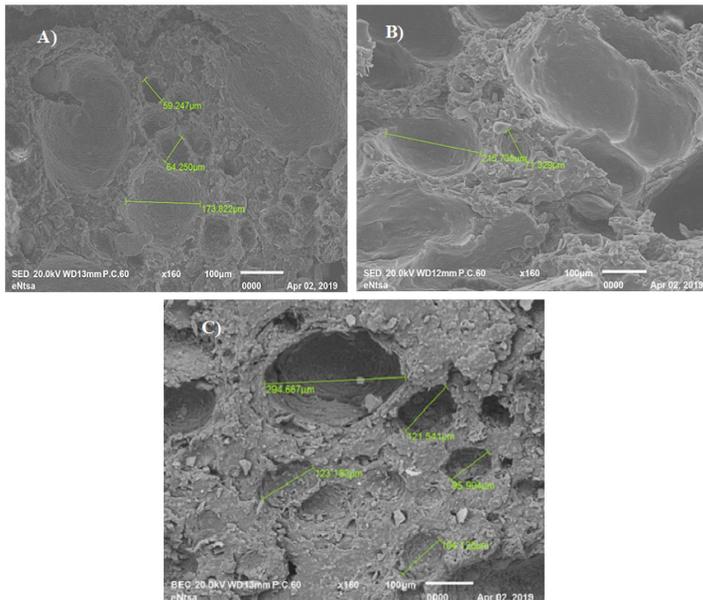


Fig. 1: SEM image of lignin RR/NR blend composites containing: a) 10 pphr, b) 20 pphr, and c) 30 pphr of lignin.

### Fourier-transform infrared spectroscopy (FTIR)

FTIR spectra of RR/NR blend and blend composites are presented in Fig. 2. The blend exhibited peaks at  $3100\text{--}3000\text{ cm}^{-1}$ ,  $1596\text{--}1680\text{ cm}^{-1}$ , and  $1384\text{--}1000\text{ cm}^{-1}$  and a hump at  $3400\text{ cm}^{-1}$  which are known for CH, C=C, C-C and N-H vibrations respectively (Ramarad et al. 2015, Yu et al. 2015, Liu et al. 2010, Botros et al. 2005). The peaks are typical in literature for a blend of the two rubbers (Ismail et al. 2002). The presence of lignin in the blend generally led to inconsistent effect. For instance, 10 pphr exhibited a suppression of C=C vibrations that seemed to resurrect at highest lignin content. In fact, there are virtually double peaks at approximately  $1384\text{ cm}^{-1}$  for all except for 10 pphr containing blend composite. Nonetheless it could be convincing to consider the inconsistencies as the elements of interfacial interaction that promoted pores as seen from SEM.

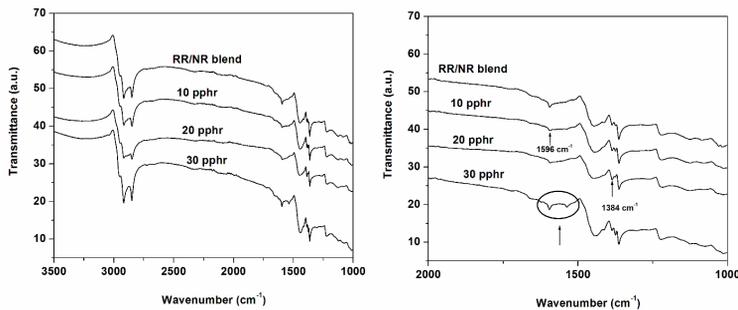


Fig. 2: FTIR spectra of RR/NR blend and lignin RR/NR blend composites.

### Thermal gravimetric analysis (TGA)

Fig. 3 shows the TGA and DTG graphs of lignin RR/NR blend composites. The presence of lignin generally decreased the thermal stability of the composites.

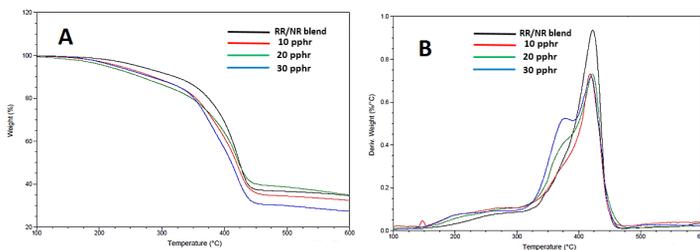


Fig. 3: TGA(A) and DTG (B) graphs of the lignin RR/NR blend composites.

This observation is not extraordinary in literature as a presence of lignin in natural fibres is known for diminishing thermal stability of the fibres. In most cases researchers centralised the rational to phenolic content, low molecular weight and the origin of lignin thereof (Datta et al. 2017, Frigerio et al. 2014, Yu et al. 2015, Liu et al. 2010, Botros et al. 2005, Barana et al. 2016). But in our case, the interfacial interaction appeared to be promoted by compatibility of the lignin and the major phase (see SEM results Fig. 1b,c). That could account for the observed drop in thermal stability. There seems to be no clear order of the lignin content against thermal stability of the blend composites, nonetheless the 30 pphr lignin RR/NR blend composite has

shown lowest thermal stability and char content than the rest. It is worth noting that lignin has a potential to increase char content of the blend composites to almost equal that of the pure blend. In fact, irregular pore sizes and complex interfacial interaction of lignin and reclaimed rubber as confirmed by SEM and FTIR could account for the lack of order in both thermal stability and residual content.

### Mechanical properties

The mechanical properties of the blend composites such as tensile strength, elongation at break and modulus were investigated. Fig. 4 shows that with increasing amount of lignin RR/NR blend composites revealed a drop in tensile strength, except for the highest lignin containing blend composite which has shown increased tensile strength that is even higher than of the pure blend composite by almost 40%.

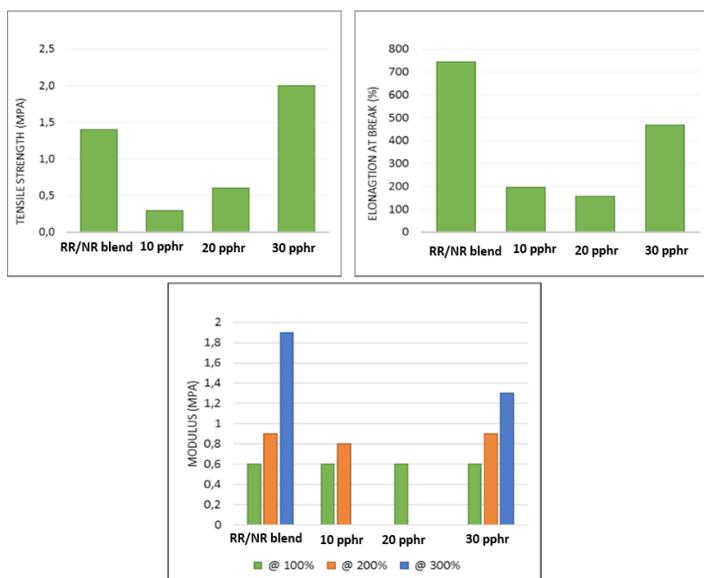


Fig. 4: Mechanical properties of the lignin RR/NR blend composites.

Almost the similar trend was also observed in the case of elongation at break, nonetheless in opposite direction for each content. For instance, the elongation at break of the clean blend composite decreased by more than 50% compared to 10 and 20 pphr and almost 45% for 30 pphr lignin containing blend composites. Moduli were measured at 100%, 200%, and 300% elongation. There was no much changes in modulus at 100% elongation, but at 300% elongation there was a surprise decrease in modulus of 30 pphr blend composites compared to the clean blend. This could be the results of inconsistencies as the elements of interfacial interaction and formation of pores as confirmed by SEM and FTIR. In fact, such defects are known for promoting lowering molecular weight of NR to shorter segments for a reduced tensile strength in RR/NR blend composites (Premachandra et al. 2011, Datta et al. 2017, Frigerio et al. 2014, Yu et al. 2015, Liu et al. 2010). Therefore, it may be virtually fair in this study to regard lignin as a contributing agent that lowered molecular weight of NR to render reduced

mechanical properties of the blend composites. Furthermore, SEM suggested the formation of the pores which are known for lowering a load transfer within the rubber matrix. In fact, Kumaran et al. (1978) explain it as a reduced elastic behavior of the elastomer by the addition of lignin.

### Dynamic mechanical analysis (DMA)

The DMA analysis was used to characterize the dynamic mechanical behaviour of the prepared lignin rubber composites. The variation of tangent delta ( $\tan \delta$ ) and storage modulus ( $E'$ ) as a function of temperature are reported in Fig. 5.

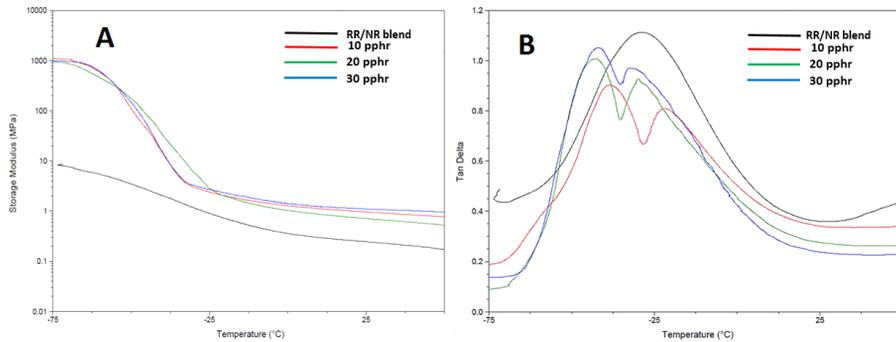


Fig. 5: Storage modulus and  $\tan \delta$  curves of RR-NR-LG composites.

The figure shows the influence of lignin on the storage modulus and damping of RR-NR blends. The addition of lignin resulted in the increase in storage modulus and that can be explained by reduced chain mobility due to the lignin addition. This is in agreement with the mechanical properties particularly the highest containing lignin composite that indicated increased storage modulus. As shown in Fig. 4, the  $\tan \delta$  curves revealed that the RR-NR-LG composites exhibit two glass transition temperatures. One occurring at low temperatures is associated to RR-NR blends and the second at high temperatures corresponds to the lignin. Both the  $T_g$ s slightly shifted to higher temperatures with increasing lignin loading. Furthermore, it can be observed that the  $\tan \delta$  peak values decreased each by approximately 10% with the increase in lignin content, suggesting a continuous decrease in rubber chain mobility and this is due to the strong interaction between the lignin and RR-NR matrix.

### Crosslink density

Fig. 6 represents blend and lignin blend composites. The presence of lignin generally increased the crosslinking density except for the highest lignin containing blend composite.

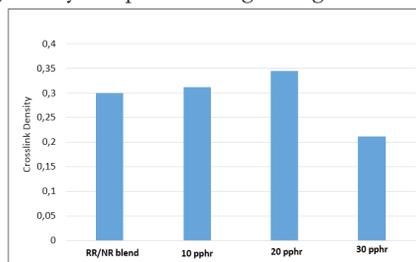


Fig. 6: Crosslinking density of the lignin RR/NR blend composites.

There was no much change in crosslinking density, nonetheless 30pphr blend composite revealed a decrease in the density by almost 40%. For 20 pphr, the observed slight increase in crosslinking density could have emanated from the complex interaction of lignin with the crosslinking sites. This is could be confirmed by suppression of C=C vibrations that seemed to revive at highest lignin content and disappearance of a peak observed from FTIR and/or cavities from SEM. It is recorded in literature that a material with a higher crosslinking density could have high thermal stability apparently due to crosslinking sites which delay a chain mobility (Ismail et al. 2002, Rattanasom et al. 2005, Yahya et al. 2011, Košíková et al. 2007). The explanation clarifies somehow TGA results in this study because it has the similar trend to crosslinking density. By the way modulus also has shown decline at 30 pphr. Perhaps lowering molecular weight of NR to shorter segments also confirms the loss of crosslinking site which rendered lower density ultimately.

### CONCLUSIONS

The effect of the lignin on properties of reclaimed rubber/natural rubber blend composites were successfully prepared and investigated. SEM revealed clear pores which varied in sizes which according to FTIR emanated from the presence of the symmetric aromatic skeletal vibration by lignin macromolecules. Of course, the pores resulted into the general decrease in mechanical properties to almost 50% and crosslinking density of reclaimed rubber/natural rubber blend composites. On the other hand, the observation is a resilient proposal that the lignin could be a potential foam agent in the blend without a blowing agent. The presence of lignin has also resulted in a general decrease in thermal stability. In fact, is worth noting that lignin has a potential to increase char content of the blend composites significantly. The observed char content trend provokes some applications for flame retardants material.

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