

# Cure Behaviour and Tensile Properties of Pineapple Leaf Fibre Reinforced Natural Rubber Composites

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ARTICLE INFO	ABSTRACT
<b>Article history:</b> Received 28 November 2023 Received in revised form 24 January 2024 Accepted 7 February 2024 Available online 22 March 2024	Short natural fibres replace synthetic fibres as filler in natural rubber (NR) as they are environmentally beneficial and sustainable. This study investigates the cure behaviour and tensile properties of pineapple leaf fibre (PALF) reinforced NR composites at various fibre contents. The fibre contents are varied at 0, 10, 20 and 30 parts per hundred rubber (phr). PALF reinforced NR composites are prepared using a two-roll mill. Surface morphology of tensile fractured specimens is examined using scanning electron microscopy (SEM). The results demonstrated that the optimum cure time decreases significantly with greater fibre content. The hardness value increases gradually with increasing filler content. The stress-strain graphs show an increasing trend in stress at higher fibre content particularly at low strain regions. On the contrary
<i>Keywords:</i> Tensile properties; natural rubber; pineapple leaf fibre; cure characteristics; scanning electron microscopy	the tensile strength reduces when the fibre content particularly at low strain regions. On the control y, the tensile strength reduces when the fibre content is increased up to 30 phr. SEM analysis reveals that the fibre-matrix adhesion is considerably poor due to the fibre pullout phenomenon observed. It is indicated that higher fibre content could be possibly reinforced to NR to achieve high deformation stress at incredibly low strain regions.

#### 1. Introduction

NR is a vital raw resource which is essential for producing a wide range of industrial products [1]. Aside from its dynamical qualities, it offers various benefits resulting from its exceptional tensile and tear strengths [2]. Resulting from strain-induced crystallisation, excellent tensile strength and break elongation are incomparable characteristics of NR. The resistance to crack propagation is excellent. However, raw NR has weak mechanical properties. Additionally, it has poor resistance to oil, ozone, and fracture initiation. There are several ways to overcome these limitations, including cross-linking and vulcanization [3]. Rubber is frequently combined with ingredients including accelerators, activators, crosslinking agents, and fillers to get the desired mechanical qualities [4].

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As environmental consciousness grows, there is an increasing need for the introduction of natural fibres as fillers in NR matrix [5]. Natural fibres have certain extra benefits over petroleum-based materials, such as accessibility, biodegradability, low density, affordability, recyclability, and non-toxicity [6–9]. Besides, they have a comparable mechanical property with glass and carbon fibres [10,11]. Addition of short fibre to NR have drawn increased attention among the numerous types of natural fibre due to the characteristics such as low specific weight, use of renewable resources, simplicity of processing and economic benefits [12]. These unique characteristics cannot be offered by addition of synthetic fibres to NR. Besides that, rubber composites with cellulosic fibre reinforcement can yield materials with a high anisotropy. Particulate fillers could not provide this special feature other than fibre reinforcement [13]. The orientation of the constituent materials in a composite affects properties like strength and stiffness in different directions [14]. Therefore, strong adhesion and uniform filler dispersion at the rubber-filler interaction are two essential elements in utilising the special abilities of fillers to strengthen rubber effectively [15]. Among the other fibres, PALF is fine enough to perform as a desirable rubber reinforcement [13].

Different kinds of fibre are reinforced into NR and have been reported in the literature to compare the tensile properties and hardness. For instance, the influence of PALF content on the tensile properties of PALF reinforced nitrile rubber was discussed in a report by Ukrit Wisittanawat et al., The reinforcement of PALF resulted in a very large improvement at low strain region as the values of all modulus are higher with greater addition of fibre content. However, the composites failed at relatively low strain [16]. In addition, Ukkadate Moonart et al., investigated the influence of hemp fibre loading on the hardness and tensile properties of hemp fibre reinforced NR composites. In comparison to unfilled NR composites, the study demonstrates that the hardness of hemp fibre filled NR composites increases with greater fibre loading. Hemp fibre reinforcement increases the modulus of rubber composites at 100 percent strain levels. The study also demonstrates that the tensile strength of hemp fibre is substantially lower compared to unfilled vulcanized NR mostly due to the crystallization under strain of NR [17]. Additionally, Palanisamy et al., studied the influence of Phormium tenax fibre content on the tensile properties of NR composites. The study found out that the tensile strength declines compared to unfilled NR composites [18]. Carla Almeda Correia et al., studied the influence of jute fibre loading on the hardness and tensile properties of NR composites. The hardness of the composites increased with greater content of jute fibre. With the inclusion of fibres, a minor decrease in the tensile strength value is observed [19].

In this study, the investigation of short PALF in compounding NR is reported. The consequences of fibre content on the cure behaviour and tensile properties of NR composites are investigated. The research aims to examine the characteristics of PALF as a filler for NR composites.

## 2. Methodology

## 2.1 Pineapple Leaf Fibres

The pineapple leaves were harvested from a plantation in Changlun, Kedah, which is located in the northern part of peninsular Malaysia. The scrapping method was employed to extract the fibres. Firstly, the leaves were cleaned and its upper layer was scratched with a dull tool to remove the waxy layer. The fibre was pulled out from the leaves and dried at room temperature for 2 days. After that, the fibre was collected in a bundle and cut into an average grain size of 6mm for further process.

# 2.2 Composite Fabrication

The NR used in this research was vulcanized standard Malaysia rubber, SMR CV (60). It had a constant viscosity of 60 and it was supplied from a local supplier, GMR Trading (M) Sdn. Bhd. The NR compounding ingredients, namely zinc oxide (ZnO), stearic acid, sulphur and N-cyclohexyl -2-benzothiazole sulfenamide (CBS) were obtained from A. R. Alatan Sains Sdn. Bhd. NR, PALF and other compounding ingredients were fabricated on a two-roll mill. The mixing duration was kept constant to prevent cross-linking and adhesion of the rubber compound to the mill rollers during mixing. The speed ratio of mill rolls, nip gap and the order of addition of NR compounding ingredients were remained constant throughout all formulations. The formulations of all NR compounds and the order of mixing are displayed in Tables 1 and 2 respectively. Firstly, the NR was masticated for 10 minutes followed by the mixing of PALF and compounding ingredients. The fibres were mixed thoroughly to ensure the fibres were evenly dispersed in the NR compounds. The NR compounds were vulcanized in a hydraulic press at 150 °C and 10 tonnes of pressure to create 3mm thickness of test specimens. The compression time was set in accordance to the optimum cure time,  $t_{c90}$ , the moment which 90 percent of curing has occurred.

Formulations of NR composite production						
Compound	Amount (phr)					
	NR	PALF	ZnO	Stearic acid	CBS	Sulphur
NR	100	0	5	2	1	4
NR/10PALF	100	10	5	2	1	4
NR/20PALF	100	20	5	2	1	4
NR/30PALF	100	30	5	2	1	4

Tab	le	1
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Sequence o	f mixing for NR compounds	
Order	Ingredients	Mixing time (min)
1	NR	10
2	PALF	6
3	Stearic acid + Zinc	2
4	Sulphur + CBS	2
5	Removal of sheet	3
Total time		23

# 2.3 Experimental Procedure

Curing behaviour of the unvulcanized NR compounds were studied at 150 °C with moving die rheometer (MDR2000, Alpha Technologies, USA). Tensile properties and hardness of the composites were determined in accordance to ASTM D412 and ASTM D2240 respectively. The tensile test specimens were made into dumbbell shape using type C die cutter. The tensile test is conducted at room temperature by using a universal testing machine (Shimadzu, model AG-X, Japan). A cross head speed of 500 mm/min was used. The tensile strength and modulus at 10% strain were determined. Hardness value was obtained with Shore A durometer (Teclock, GS-719N, Japan). Measurements were taken on 5 different positions and their average value is reported.

# 2.4 Scanning Electron Microscopy (SEM)

The SEM analysis was conducted to identify the morphological changes on the tensile fractured surface of the PALF reinforced NR composite using a TESCAN VEGA SEM (Novatiq Scientific Sdn. Bhd., Kuala Lumpur, Malaysia). A thin layer of gold was coated to the sample surfaces using an ion coating process to avoid electrical charge accumulation. The analysis was carried out at a 100x magnification. The objective of the current study was to identify the rubber-matrix adhesion in the morphology and tear mechanisms of the fibres in the NR matrix.

# 3. Results

## 3.1 Cure Characteristics

Curing behaviour of PALF reinforced NR compounds are displayed in Table 3. The relative viscosity of NR compounds is measured by the minimum torque,  $M_L$  [20]. Correspondingly, a lower minimum torque denotes a lower compound viscosity [21]. The values of  $M_L$  increases gradually with greater filler contents. In other words, the viscosity increases when the filler contents increase. Good filler-to-matrix adhesion is shown by the rise in  $M_L$  with greater filler content, which limits the free mobility of rubber chains [22]. Similar observation had been reported by Justyna Miedzianowska *et al.*, [23].

At the vulcanization temperature, the maximum torque,  $M_H$  is a measure of the stiffness or shear modulus of the test specimen that has been entirely vulcanised [24].  $M_H$  increases with greater filler contents and it displays the highest value at 30 phr of fibre content. The rubber-filler interaction, which includes intercalation and exfoliation, is responsible for the  $M_H$  [21]. The increase phenomenon of  $M_H$  suggests that the NR compounds in PALF reinforced NR compounds have a higher network density [24].

The scorch time,  $t_{s2}$  is the time at which the vulcanization of the NR compounds starts [24].  $t_{s2}$  of the prepared NR compounds shows a decrease in trend up to 20 phr and slightly increase at 30 phr of PALF content. The decrease of  $t_{s2}$  with greater fibre content may be owing to fibre-related factors such as fibre surface reactivity, fibre surface area, and fibre particle size [22].

The optimum cure time,  $t_{c90}$  for NR compounds decreases significantly with greater fibre content. Reduce in  $t_{c90}$  corresponds to a higher cure rate of the NR compounds [25]. In other words, the presence of fibre serves as curative components that improve the cure rate. Numerous researches have noted a decrease in  $t_{c90}$  when filler contents are greater [19,26–29].

Cure characteris	stics of NR compou	nds		
Compound	$M_L$ (dNm)	$M_H$ (dNm)	<i>t<sub>s2</sub></i> (min)	t <sub>c90</sub> (min)
NR	0.35	5.07	8.6	13.9
NR/10PALF	0.54	8.98	5.1	9.8
NR/20PALF	0.59	16.14	3.9	8.8
NR/30PALF	1.40	17.41	4.3	7.7

Table 3Cure characteristics of NR compounds

# 3.2 Hardness

Table 4 displays the Shore A hardness of PALF reinforced NR composites at different PALF content. It shows an increase in trend with greater PALF loading compared to raw NR composites. The reinforcement of fibre significantly increases the hardness values of the composites. This indicates that the incorporation of fibres reduces the ductility and increase the stiffness of the composites [30,31]. The highest value is 54.2 Shore A at 30 phr of PALF content in NR composites.

According to studies, addition of fibres greatly increases the hardness of NR composites [17,19,32,33].

Table 4		
Hardness Shore A of NR composites		
Compound	Hardness (Shore A)	
NR	46.8	
NR/10PALF	49.6	
NR/20PALF	53.2	
NR/30PALF	54.2	

#### 3.3 Tensile Properties

This present research aims to study the tensile properties of NR composites at different fibre contents. Figure 1 displays the relationship of stress versus strain of PALF reinforced NR composites containing various fibre contents. In general, PALF reinforced NR composites show a significant increase in stress at lower strain areas, indicating the composites stiffen as the fibre content increases [12]. Increased fibre content resulted in a decrease in NR matrix content, whereas decreased ductility resulted in a stiffer composite [34]. Compared to the raw NR composite, the greater the fibre content, the greater the increase of slope at low strain region. However, the strain and tensile strength decrease as the fibre content increases. This is due to the reinforcement of fibrous fibres has restrict the mobility of rubber chains. [32]. Similar observations have been reported for NR composite reinforced with short PALF and aramid fibre [12,35].

The observed reduction in tensile strength with increasing PALF addition is mainly caused by the poor fibre-matrix compatibility, which resulted in a poor rubber-filler interaction. The poor compatibility of the hydrophobic rubber chains and the reinforcement of lignocellulosic fibre interferes with the arrangement of rubber chains followed by reducing the amount of strain that causes the rubber to crystallise which results in reduction of the tensile strength of NR composites [36,37]. Stresses from the NR matrix cannot be consistently transmitted through the filler. Thus, the tensile strengths are further reduced [22].



**Fig. 1**. Stress strain curves of PALF reinforced NR composites at varying fibre contents

Further mechanical property could be extracted from the stress-strain curves is the Young's modulus at 10% strain of the composites. The values are shown against the filler contents as illustrated in Figure 2. It demonstrates a clear increment with greater content of PALF. The effect of increased PALF loading leads to an increase stiffness of rubber composites followed by modulus in rubber modulus. In other words, the modulus of the composite increases when the filler has a higher rigidity than the matrix [38]. Similar effects have been reported for short aramid fibre reinforced NR and PALF reinforced NR composites [12,35,38].



Fig. 2. Modulus at 10% strain

## 3.4 Scanning Electron Microscope

The present study employed SEM to examine the surface morphology and microstructural changes of samples of PALF reinforced NR composites after tensile test at different fibre contents. The SEM images after tensile test for each specimen are presented in Figure 3(a)-(c).

SEM images demonstrated that the fibre distribution in the matrix is random. NR composites resist to failure caused by matrix cracking, which is most typically seen in traditional stiff composites. However, the NR composites demonstrate the phenomena of matrix tearing [39].

It was observed in the SEM images that PALF reinforced NR composites produced fibre tear, fibre pullout, regions of matrix tearing and debris after tensile test is performed at room temperature. Numerous voids are left on the surface of the composites after the fibres were separated from the rubber matrix after tensile test is performed. The fibres debone, shear at the interface, and pullout due to the greater tensile forces at the fibre ends than the composites could tolerate. This illustrates that the bond is insufficient between the fibre and NR matrix [40].

The deeper tear line and rougher surface appearances exhibited in Figure 3a are caused by the increased crack propagation resistance during the tensile test [41]. By creating deformation regions, the composites are resistant to fracture [42]. The presence of the fibre remained in the rubber matrix indicates the strong fibre-matrix adhesion [40]. Additionally, the tensile strength is increased due to the effective load transmission from the matrix to the reinforcement [28].

A conclusion is drawn from the SEM images that the poor adhesion at the fibre-matrix interphase where fibre pull-out, voids formed after fibre pullout are observed, may initiate tearing to cause the composite to fail in tensile modes.



**Fig. 3**. SEM images of tensile fractured specimens at fibre contents of (a) 10 phr, (b) 20 phr, (c) 30 phr

## 4. Conclusions

In conclusion, this research provides valuable insights into the tensile properties of PALF reinforced NR composites at varying fibre contents. The objective of the current study is to comprehend the influence of fibre contents on the tensile properties of composites, and the results directly address this aim. Specifically, the modulus at 10 percent strain and hardness exhibit higher values at greater fibre content. Compared to raw NR, PALF reinforced NR composites show a reduction in tensile strength. Reinforcement of short fibres into the composites enhances stress transfer at low strains. Furthermore, the incorporation of PALF in NR enables the composite to endure very high deformation stress in regions characterized by extremely low strain. SEM analysis highlights a significant deficiency in fibre-matrix adhesion. The outcomes of this work underscore the potential for utilizing materials derived from industrial crops in the development of NR composites. The findings presented herein offer guidance for fabricating short natural fibre-reinforced rubber composites that are more environmentally friendly.

## Acknowledgement

This research was supported by the Ministry of Education, Malaysia through the Fundamental Research Grant Scheme (Ref: FRGS/1/2021/STG05/UNIMAP/02/3). The authors also acknowledge the Faculty of Mechanical Engineering & Technology, Universiti Malaysia Perlis (UniMAP), for providing equipment and financial and technical assistance. The authors are grateful for the fruitful discussions and input of the UniMAP staff.

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