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Synthesis and Characterization of Liquid Natural Rubber (LNR) from SIR-10 as a Basic Material for Making Rubber Compounds

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ABSTRACT

Liquid Natural Rubber (LNR) is synthesized from Standard Indonesian Rubber (SIR-10) through the process of deproteinization and oxidative degradation using phenylhydrazine/O₂. Deproteinization was carried out by the Soxhlet extraction method using acetone solvents to reduce protein content that can inhibit the degradation process. Furthermore, the rubber was oxidatively degraded and characterized using Fourier Transform Infrared (FTIR) and intrinsic viscosity measurements with an Ostwald viscometer. The FTIR results showed the emergence of new absorption bands at 3273 cm⁻¹ (O-H) and 1713 cm⁻¹ (C=O) indicating the formation of oxidized functional groups. Viscosity analysis showed a significant decrease in molecular weight from 3.717 × 10⁵ g/mol (SIR-10) to 2.375 × 10³ g/mol (LNR). These results confirmed that SIR-10 was successfully converted into LNR which has the potential to be used as a base material in the manufacture of rubber compounds.

Keywords: liquid natural rubber, SIR-10, deproteinization, oxidative degradation, FTIR

1. INTRODUCTION

Natural rubber (*Hevea brasiliensis*) is an important biopolymer extensively utilized in many industrial applications, including tire manufacturing, medical gloves, and vibration isolation components, due to its excellent elasticity and mechanical properties.¹ However, natural rubber has limitations such as low thermal and oxidative resistance due to the high level of unsaturation of polyisoprene chains.² In order to improve thermal stability and expand its application, various chemical modifications have been developed, one of which is the formation of Liquid Natural Rubber (LNR). LNR is a natural rubber derivative with a shorter polymer chain so that it has low viscosity, is easy to mix, and can function as a compatibilizer in the manufacture of rubber compounds.³

One of the potential raw materials for LNR synthesis is Standard Indonesian Rubber (SIR)-10, which has low impurities and good mechanical properties compared to other types of SIR.⁴ However, the protein content in SIR-10 can inhibit the oxidative degradation process because the protein is capable of neutralizing free

radicals that play a role in the breaking of polymer chains.⁵ Therefore, a deproteinization process is required to remove proteins and non-rubber components, resulting in raw materials with better thermal stability. Previous research has shown that the process of deproteinization with acetone solvents is able to significantly lower protein levels and produce rubber with higher oxidative stability.⁶ After the process, the polymer chain of natural rubber can be chemically broken using phenylhydrazine/O₂-based oxidative degradation methods to form LNR with lower molecular weight and the emergence of new oxygenation groups such as carbonyl (C=O) and hydroxyl (O-H).⁷

Several previous studies have reported LNR synthesis using various degradation systems, such as ozonolysis, radiation, and redox reactions.^{8,9} However, the phenylhydrazine/O₂ oxidation method is considered more efficient because it takes place at relatively low temperatures and results in a uniform distribution of molecular weight.¹⁰ LNR produced from deproteinized SIR-10 has the potential to be the basic material for the manufacture of rubber compounds because they can improve compatibility between natural rubber matrices and fillers such as carbon black.¹¹ In this study, LNR synthesis from SIR-10 was carried out which has gone through a deproteinization process with acetone solvent, followed by an oxidative degradation reaction using phenylhydrazine/O₂. Characterization was carried out by Fourier Transform Infrared (FTIR) analysis to identify the modified functional groups and molecular weight analysis using the intrinsic viscosity method (Ostwald Viscometer). This study aims to provide an overview of the influence of deproteinization on the success of LNR formation and its potential application as a basic material for making rubber compounds with better mechanical performance.

2. EXPERIMENTAL

2.1. Chemicals, Equipment and Instrumentation

The equipment used included Standard Indonesian Rubber/SIR 10, Acetone (C₃H₆O) 99% (p.a. Merck), Xylene (C₈H₁₀) 99% (p.a. Merck), Phenylhydrazine (C₆H₅NHNH₂) 99% (p.a. Merck), Oxygen Gas (O₂), Toluene (C₇H₈) 99% (p.a. Merck), Methanol (CH₃OH) 99% (p.a. Merck), distilled water.

The tools used include glassware, beaker, three-neck round-bottom flask, condenser, *Ostwald* viscometer (*Pyrex*), *Hot plate* (*Cimarec*), Analytical balance (*Scientech*), Oven (*Memmert*), A set of oxygen regulators and gas cylinders, stands and clamps, a Soxhlet apparatus, a distillation apparatus, *Fourier Transform Infrared Spectroscopy* (FTIR; *Bruker Invenio II*).

2.2. Research Procedure

A. Deproteinization of Natural Rubber (DPNR)

The sample used in this study is SIR-10. The deproteinization process was carried out using a Soxhlet extraction method. SIR-10 was cut into small pieces and then ground using a Blending Mill. After that, it was extracted using 100 mL of acetone solvent at 56°C for 24 hours. The extraction results are then dried using the oven for 2 hours. Then analyzed using FTIR spectroscopy.

B. Manufacture of Liquid Natural Rubber (LNR)

Cut 15 grams of SIR-10 into small rubber granules to aid in the dissolution process. Then, 375 mL of xylene was added, mixed, and heated into a three-neck flask at 90°C until SIR-10 is completely dissolved. Then, 27 mL of phenylhydrazine was added, then oxygen was introduced and refluxed for 24 hours. Next, the

reflux results were precipitated using 1.2 L of methanol, followed by two washings and drying at 60°C for 4 hours to produce liquid natural rubber, which was then examined through FTIR spectroscopy.⁷

C. Molecular Weight Analysis Preparation

The Ostwald Viscometer is prepared by inserting 10 mL of toluene into the Ostwald Viscometer. Using a rubber ball, suck the toluene solution up to the marked line, then after the suction is stopped, record the time it takes for the toluene to go down from the top mark to the bottom mark. Next, add 0.1 g of LNR to 10 ml of toluene to make a 0.01 g/ml polymer solution, and further prepare 0.02 g/ml, 0.03 g/ml, 0.04 g/ml, and 0.05 g/ml solutions. Then, insert the polymer solution into the Ostwald Viscometer, following the same procedure as mentioned earlier, repeat the process three times to determine the intrinsic viscosity value and calculate the molecular weight.⁷

3. RESULTS AND DISCUSSION

3.1. Analysis of Characterization Results

A. Deproteinization of Natural Rubber (DPNR)

The deproteinization process is carried out by Soxhlet extraction, the results of FTIR analysis are as follows.

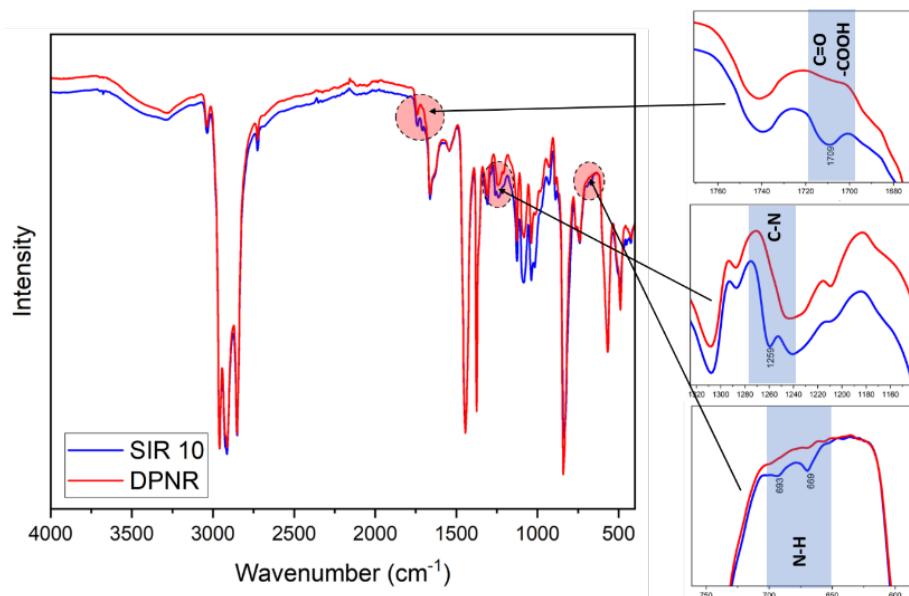


Figure 1. FTIR spectra SIR-10 before (blue) and after (red) extraction with acetone for 24 hours

The results of FTIR analysis on SIR 10 before (blue) and after Soxhlet extraction (red) the FTIR spectra of SIR-10 after extraction exhibited a noticeable reduction in absorption intensity associated with amide-related functional groups, indicating a significant decrease in protein content that could be observed from the decrease in intensity at the wavenumber around 1726 cm⁻¹ from the presence of C=O and -COOH functional groups of carboxylic acids bound to protein molecules.^{12,13} Then the existence of the C-N group of amides is also no longer found, where before Soxhlet method it was still found to be absorbed at the wavenumber 1252 cm⁻¹. This is also supported by the loss of absorption at the 650-700 cm⁻¹ wavenumber after Soxhlet extraction which is the presence of the N-H functional group.¹⁴

A. Manufacture of Liquid Natural Rubber (LNR)

Visually, there is a physical change from natural rubber (SIR-10) to liquid natural rubber (LNR) after drying in the oven as shown in Figure 2. After drying, there is a phase change in natural rubber where its elasticity decreases so that the rubber turns more fluid and brittle. In addition, there are differences in the solubility of natural rubber in various organic solvents; for example, Natural rubber was highly soluble in non-polar solvents such as xylene and toluene, while LNR exhibits limited solubility in polar solvents such as acetone., whereas LNR was only slightly soluble in polar solvents such as acetone. This difference indicates changes in the chemical and physical structure of the rubber due to drying and modification processes that affect the solubility and consistency of the material.

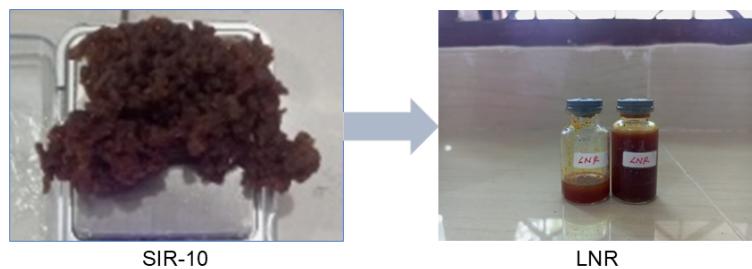


Figure 2. Changes in the Physical of Natural Rubber (SIR-10) and Liquid Natural Rubber (LNR)

The results of the analysis of the FTIR spectra of natural rubber on LNR can be seen in figure 3 in the form of the percent transmittance of natural rubber SIR-10 and the results of LNR chain breaking. The spectral comparison showed a change in chemical structure between SIR-10 and LNR. The emergence of new absorption peaks at the peak of the O-H group at wavenumber 3273 cm^{-1} and the peak of carbonyl absorption C=O at wavenumber 1713 cm^{-1} . In addition, the peak of absorption at the peak of the double bond $=\text{C-H}$ wavenumber 3034 cm^{-1} . A comparison of the changes in the absorption peaks of the SIR-10 and LNR natural rubber spectra formed can be seen in figure 4. In more detail, the magnitude of the peak absorption intensity for each type of absorption can be seen in table 1.

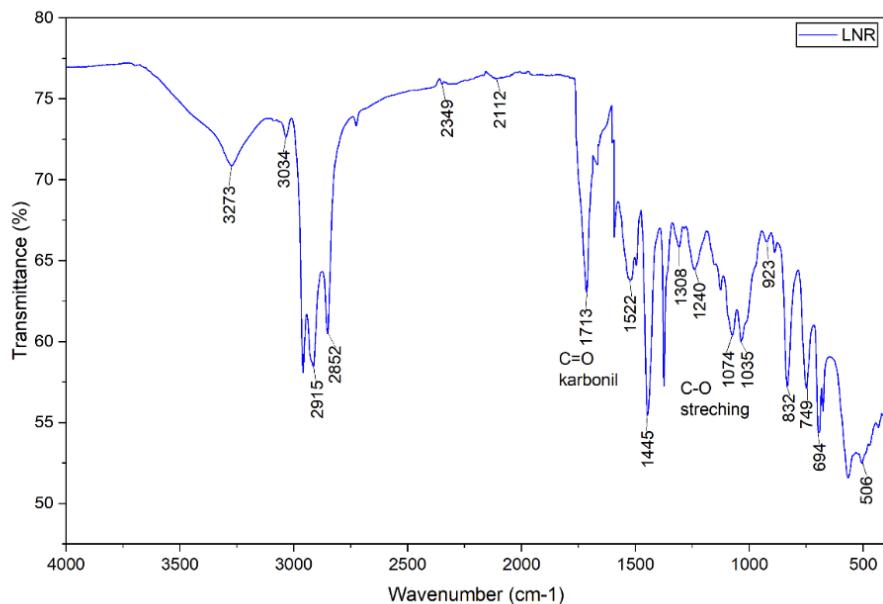


Figure 3. Spectra of degradation of Liquid Natural Rubber using phenylhydrazine and oxygen

Based on the FTIR spectra in Figure 3 show that the differences in absorption intensity and peak position of absorption between SIR-10 natural rubber and Liquid Natural Rubber (LNR) can be observed, reflecting changes in chemical structure due to oxidative degradation processes. To clarify the identification of functional groups and differences in their absorption characteristics, the data on the number of waves and the intensity of each peak are summarized in Table 1.

Table 1. Absorption Type and Absorption Intensity, FTIR spectra of Wavenumber Natural Rubber SIR-10 and Liquid Natural Rubber

Name	Figure	Peak (cm ⁻¹)	Intensity	Role
NR	Figure 2	3282	Broad, medium (O-H stretching)	O-H stretching (hydrogen bonding) can cover N-H
		3036	Medium – sharp (stretching)	=C-H stretching on double bonds
		2959, 2912,	Strong – sharp (stretching)	CH ₃ & CH ₂ asymmetric/symmetric stretching
		2850		
		2725	Very weak (stretching)	-CHO stretching
		1741, 1662	Medium – strong, sharp (stretching)	C=O stretching (amide/carbonyl oxide ring)
		1542	Weak – bending	N – H bending + C – N stretching
		1444, 1375	Medium – bending	CH ₂ scissoring/CH ₃ bending
		1243	Weak (mixture of bending & stretching)	C – N stretching
		1126, 1082, 1037	Strong – bending	Out-of-plane cis-C–H bending
LNR	Figure 3	888, 929	Medium–strong – bending	cis-1,4 out-of-plane bending
		840, 739	Medium – bending	trans-1,4 C–H bending
		3282	Broad-medium (stretching)	O-H stretching (water/mild oxidation)
		3030	Medium – sharp (stretching)	=C–H stretching
		2950, 2920,	Strong – sharp (stretching)	CH ₃ & CH ₂ asymmetric/symmetric stretching
		2850		
		1660	Weak–medium (stretching)	C=C stretching or light carbonyl
		1450, 1375	Medium – bending	CH ₂ scissoring & CH ₃ bending
		1240	Weak (bending/stretching)	C–C/C–O stretching
		1125, 1080, 1030	Strong – bending	Out-of-plane cis-C–H
		890 – 900	Medium–strong (bending)	cis-1,4 isoprene
		835 – 740	Medium – bending	trans-1,4 isoprene

A comparison between the obtained LNR spectra and that reported by Tamrin et al. (2021) revealed both similarities and significant differences, particularly in the intensity of the carbonyl absorption region, suggesting a higher degree of oxidative modification in the present study, both samples showed an absorption band in the range of 3200–3400 cm⁻¹, which indicates the presence of a hydroxyl group (O–H) and shows that

the oxidation process effectively inserts the polar group into the natural rubber chain. Both samples showed prominent absorption bands in the range of 2968–2850 cm^{-1} , which are related to the C–H elongated vibrations of the methyl and methylene groups in the polyisoprene structure. Furthermore, the presence of bands near 2726–2728 cm^{-1} indicates the potential for aldehyde group formation, albeit at relatively low intensity.¹⁵

A marked contrast was observed in the carbonyl region, in this LNR result, a significant absorption band was observed near 1713 cm^{-1} , indicating the presence of a carbonyl group (C=O), while in the Tamrin LNR, the carbonyl band was less prominent, highlighting the absorption at 1661 cm^{-1} associated with the double bond (C=C). This suggests that these LNR results undergo additional oxidation, which results in a larger number of carbonyl groups compared to the reference. Both spectra show a typical C=C stretching band near 1660 cm^{-1} and a CH_2/CH_3 bending band in the range of 1450–1370 cm^{-1} , suggesting that the basic structure of polyisoprene remains intact.

Furthermore, both LNR exhibit absorption bands in the range of 1300–1000 cm^{-1} associated with the C–O stretching group, which further supports the indication of oxidative modification. The typical cis-1,4 double bonding band appears at about 894–880 cm^{-1} in both spectra, indicating that the basic structure of natural rubber remains largely intact despite oxidation. However, in the reference LNR, there were additional bands at 743 and 707 cm^{-1} that were less pronounced in the study LNR, which may be related to the vibration outside the C–H bond plane.

This comparison shows that the results of the study produce LNR with features consistent with the reference, especially related to the presence of the O–H, C=C, and C–O groups. However, the clearer carbonyl band intensity in the study's LNR findings shows that the oxidation process produced a higher level of alteration compared to the reference LNR.

B. Molecular Weight Test Analysis

The average molecular weight (\overline{M}_v) of *Liquid Natural Rubber* (LNR) was calculated using the *Ostwald* viscometer through the *Mark and Houwink* equation.¹⁶ The following table 2 shows the data in the form of LNR flow rate time obtained with the *Ostwald* viscometer:

Table 2. LNR and SIR-10 Flow Rate Time with Ostwald Viscometer

Solution	Flow rate time 1 (sec) (t ₁)	Flow rate time 2 (sec) (t ₂)	Flow rate time 3 (sec) (t ₃)	Average flow rate time (sec)
Toluene	35,45	35,34	35,78	35,523
LNR 1 %	53,46	53,87	53,22	53,516
LNR 2 %	75,23	75,77	75,14	75,38
LNR 3 %	106,45	106,73	106,82	106,667
LNR 4 %	150,35	151,93	151,61	151,296
LNR 5 %	185,11	183,84	184,27	184,406
SIR-10 1 %	360,10	360,14	360,12	360,12
SIR-10 2 %	480,30	480,36	480,34	480,34
SIR-10 3 %	620,5	620,82	621,61	620,976
SIR-10 4 %	1068,47	1069,53	1073,60	1070,53
SIR-10 5 %	3965,50	3965,56	3965,54	3965,54

Based on the above equation, the relative viscosity, specific viscosity, and reduction viscosity of LNR and SIR-10 are obtained in the following table 3:

Table 3. Relative Viscosity, Specific Viscosity and Reduction Viscosity Data of LNR and SIR-10

Concentration (%)	Relative viscosity (10^4 kg/ms) (η_r)	Specific viscosity (η_{sp})	Viscosity reduction (η_{red})
LNR 1 %	84.063	0,5065	50,65
LNR 2 %	118.407	1,1219	56,095
LNR 3 %	167.553	2,0027	66,7566
LNR 4 %	237.657	3,259	81,475
LNR 5 %	289.667	4,1911	83,822
SIR-10 1 %	56,5681	9,1376	913,67
SIR-10 2 %	74,4524	12,3427	617,135
SIR-10 3 %	97,5437	16,4809	549,363
SIR-10 4 %	168,1602	29,1362	728,405
SIR-10 5 %	622,9122	110,633	2212,66

Through the data in table 3 above, the intrinsic viscosity value of LNR and SIR-10 was obtained from the resulting linear graph, where the intercept value of the plot is the intrinsic viscosity value. From the graph, the LNR intercept value is 9.1724 and the SIR-10 is 270.93 in the following graph:

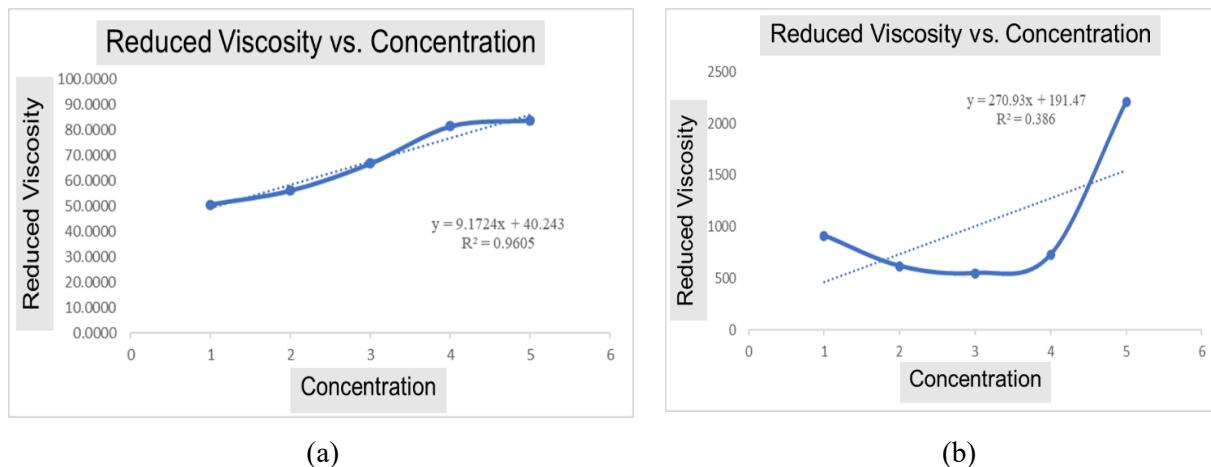


Figure 4. Viscosity Reduction Graph of (a) LNR and (b) SIR-10

The above intrinsic viscosity [η] was obtained by intercepting the plot (in a linear graph) based on the Huggin equation, obtained from the molecular weights in the table below:

Table 4. Average Molecular Weight of LNR and SIR-10

Polymer Types	Average Molecular Weight (\bar{M}_v)
<i>Liquid Natural Rubber</i>	$2,37465 \times 10^3$
<i>Natural Rubber</i>	$3,71706 \times 10^5$

Based on the results in table 4, molecular chain scission occurred due to the presence of phenylhydrazine with O₂ gas flowing until there is a significant decrease in molecular weight in the average molecular weight of SIR-10 natural rubber of 3.71706×10^5 g/mol to 2.37465×10^3 g/mol in the LNR.

4. CONCLUSION

The SIR-10 deproteinization process using acetone solvent through the soxhletation method was proven to reduce protein content, which is indicated by the reduced absorption band of amide groups in the FTIR spectra. Furthermore, oxidative degradation using phenylhydrazine and O₂ flow successfully fractured the natural polymer chain of natural rubber and produced Liquid Natural Rubber (LNR) with the formation of new functional groups such as O-H and C=O. The results of viscosity analysis showed a significant decrease in molecular weight from 3.717×10^5 g/mol to 2.375×10^3 g/mol. This proves the success of the LNR synthesis of SIR-10, which has potential for use as a base material in the manufacture of rubber compounds with more reactive and flexible properties.

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