Short Communication:

Nitration of Liquid Natural Rubber by Concentrated Nitric Acid in the Presence of Acetic Anhydride

Khai Minh Doan[1*], Trung Bao Tran[1], Tuan Duy Nguyen[1], and Vuong Quoc Ly[2]

[1]*Faculty of Special Equipment, Le Quy Don Technical University, 236 Hoang Quoc Viet, Hanoi 100000, Vietnam*

[2]*Institute of Materials, Biology and Environment, Academy of Military Science and Technology, 17 Hoang Sam, Hanoi 100000, Vietnam*

** **Corresponding author:***

tel: +84-977291866
email: khaihv@lqdtu.edu.vn

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Abstract: *Liquid natural rubber (LNR) is commonly used as a binder in energetic composites. One of the key areas of interest is the development of energetic polymers to enhance adhesion efficiency and energy. In this study, an energetic liquid polymer—nitrated liquid natural rubber (N-LNR)—was successfully synthesized via nitration of LNR using concentrated nitric acid (HNO_3) in the presence of acetic anhydride (Ac_2O) and dichloromethane as solvent. The effects of key parameters, including the HNO_3/LNR ratio and reaction time, on the degree of nitration and reaction yield were systematically investigated. The degree of nitration was found to increase linearly with the HNO_3/LNR ratio, ranging from 7.97% to 32.9%, with a maximum yield of 76.1%. The optimal reaction conditions were identified as an Ac_2O/HNO_3 molar ratio of 2:1, a reaction time of 1.75 h, and a temperature of 20 °C. Structural analyses by FTIR, ^{1}H-NMR, and ^{13}C-NMR confirmed the presence of* cis*-1,4-isoprene chains partially substituted with $–NO_2$ and $–ONO_2$ groups, verifying successful nitration at the C=C bonds. These results demonstrated a controllable and efficient method for preparing energetic LNR-based polymers with potential application as binders in propellants and plastic explosives.*

Keywords: *liquid natural rubber; nitration; nitric acid; energetic polymer*

■ INTRODUCTION

Liquid natural rubber (LNR), which is a type of liquid polymer, is mainly prepared by oxidation by a redox system or by photochemical UV light. The LNR contains *cis*-1,4 isoprene units in the main chain and may have functional groups at the end of its chain, such as hydroxyl, carbonyl, or carboxyl. Additionally, the LNR also contains a few epoxy groups in its molecular chain [1-3]. Furthermore, LNR can also be hydrogenated to reduce the degree of unsaturation, thereby improving physical properties, stability against thermal aging, oxidation, and photochemical degradation [4]. Ritonga et al. [5] investigated the enhancement of mechanical, thermal, and morphological properties of LLDPE/natural rubber composite materials incorporating organically precipitated calcium carbonate. The LNR is capable of functioning as a binder (10–15% by weight) for solid rocket propellants [6] or plastic explosives [7]. A recent trend is to replace the above polymers with an energetic polymer to improve adhesion and enhance energy for solid rocket propellants and explosives [8]. The energetic polymer typically contains an amount of nitro, nitrate, nitramine, or azide functional groups in the main chain. These polymers are obtainable from monomers or by the derivatization of liquid polymers. In this publication, the second route has been followed to prepare nitrated LNR (N-LNR).

The nitration of polydiene polymers was carried out using only a few methods. Nitromercuration of diene polymers has been performed on polyisoprene and polybutadiene [9]. Hydroxyl-terminated polybutadiene (HTPB) has been nitrated using N_2O_5 in dichloromethane [10]. Nitro-HTPB has also been synthesized using a $NaNO_2/I_2$ reagent in ethyl acetate [11-12]. Nitro-functionalized hydroxyl-terminated polybutadiene has

been prepared using $NaNO_2$ in the presence of *N*-iodosuccinimide [13]. An LNR was nitrated via iron-mediated radical chloro-nitration [14]. These publications generally suggest that about 10–15% of the monomer units should be functionalized with nitrate groups to retain the elastic properties of polydiene rubbers. Additionally, the nitration of alkenes using various methods has been reported. The alkenes are reacted with nitric acid (HNO_3) to give the intermediate product, hydroxy nitroalkanes. Then, hydroxy nitroalkanes are dehydrated to form nitroalkanes. In addition, alkenes can be nitrated by a mixture of HNO_3 and acetic anhydride (Ac_2O) to form β-nitroacetates, nitroolefins, and β-nitronitrates. Mixtures of nitric acid with strong acids, such as H_2SO_4 or HF, have also been studied as a nitrating agent for alkenes [15]. Nitrohalogenankenes may be formed when alkenes react with a mixture of $Fe(NO_3)_3$ and $FeCl_3$ salts at high temperatures. At the reaction temperature, the $Fe(NO_3)_3$ and $FeCl_3$ salts will be decomposed to form NO_2* and Cl* radicals, which are the main agents for the reaction [16]. No documented research has been found concerning the nitration of LNR using a combination of concentrated HNO_3 and Ac_2O. In this study, LNR was functionalized via nitration in the presence of Ac_2O using dichloromethane as the solvent. The chemical structure of the resulting N-LNR was characterized by Fourier-transform infrared (FTIR) and nuclear magnetic resonance (NMR), and various reaction parameters were systematically investigated.

■ EXPERIMENTAL SECTION

Materials

The LNR was a hydroxyl-terminated polyisoprene prepared in our laboratory from natural rubber (NR) by the photochemical method. The obtained product had a molecular weight of 6,800 g/mol and a viscosity of 19.8 Pa·s at 50 °C. The structural characterization of LNR: ^{1}H-NMR ($CDCl_3$, δ ppm): 5.05 (1,4-unit –C**H**=), 3.40 (terminated –C**H**$_2$OH), 2.62 (epoxy –C$_2$**H**$_2$O), 1.98 (1,4-unit –C**H**$_2$–), 1.6 (unit –C**H**$_3$), 1.2 (terminated –C**H**$_3$). ^{13}C-NMR ($CDCl_3$, δ): 23.4 (unit –**C**H$_3$), 26.4 and 32.2 (unit –**C**H$_2$–), 125.0 and 135.2 (*cis*-unit –**C**=**C**–), 60.9 (terminated –**C**H$_2$OH) and 64.6 (terminated >**C**HOH). FTIR (KBr, cm^{-1}): 3448 (–OH), 1664 (>C=C<), 888 (epoxy), 837 (*cis*-1,4). Additionally, hydrogen peroxide 30%, toluene, tetrahydrofuran, and hydroquinone were purchased from XiLong, China. HNO_3 98% was supplied by 95 Chemical Company, Vietnam. Dichloromethane, Ac_2O, and methanol were supplied by Shanghai Macklin Biochemical Co., Ltd, China.

Instrumentation

The FTIR spectra of the sample were recorded using a Spectrum 400 FTIR spectrometer in the range of 400 to 4000 cm^{-1} with 32 cumulative scans at a resolution of 4 cm^{-1}. The sample was coated on a KBr disk to form a thin film. The samples were measured at 25 °C and a humidity of 35%. ^{1}H-NMR and ^{13}C-NMR spectra were recorded by using an EvanceNEO instrument with a frequency of 600 MHz and a temperature of 303 K. N-LNR samples were dissolved in $CDCl_3$ solvent. The nitration degree of N-LNR was quantified by the Bergman-Junk method. According to this method, the N-LNR was decomposed to form NO_2 gas by heating at 132 °C. This NO_2 gas was absorbed by a 5% hydroperoxide solution. The NaOH solution was used to titrate the absorbed NO_2 content in the solution. The scheme for the reaction of NaOH with NO_2 is shown in Eq. (1).

$$2NaOH + 2NO_2 \rightarrow NaNO_3 + NaNO_2 + H_2O \quad (1)$$

The NO_2 content was calculated through titration with NaOH in the presence of the phenolphthalein indicator. The mole of NO_2 was equal to the mole of the nitro group in the N-LNR, calculated as Eq. (2).

$$n_{NO_2} = V_{NaOH} \times C_{NaOH} \quad (2)$$

So the nitration degree of N-LNR (f, %) was calculated based on the mole of NO_2 calculated using Eq. (3).

$$f(\%) = n_{NO_2} \times \frac{68}{m_{LNR}} \times 100 \quad (3)$$

Procedure

Preparation of LNR

NR was dissolved in toluene to obtain a 5% (w/v) solution under continuous stirring in a 500 mL flat-bottom flask. Subsequently, methanol was introduced into the solution at a toluene-to-methanol volume ratio

of 20:3. Hydrogen peroxide was then added dropwise to the mixture at a hydrogen peroxide-to-toluene ratio of 1:20. The resulting mixture was stirred and exposed to irradiation from a mercury vapor lamp (HSW-250 W) for 50 h. After the reaction, the product was precipitated and repeatedly purified three times using mixtures of methanol/toluene (v/v). The purified sample was finally dried in a vacuum oven at 65 °C for 24 h.

Method of nitration

The LNR was dissolved in dichloromethane with a concentration of 5 g/100 mL in a 3-necked flask. Then, Ac_2O was added to the flask. The flask was cooled down to the reaction temperature. A solution was prepared by dissolving 98% HNO_3 in dichloromethane and placing it in a conical flask. This solution was slowly dripped into the 3-necked flask. After the reaction, the resulting mixture was agglomerated with methanol and washed with methanol 5 times. The product was dried under a vacuum for 24 h. The nitration method was illustrated in Fig. 1. To maintain mechanical properties close to the original polymer after functionalization, the degree of functionalization should be approximately 10–15 mol% of the chain units. Based on the calculations, the mass ratio of HNO_3 to LNR was 14:100, corresponding to the introduction of 15 functional groups per 100 isoprene chain units. Based on the experimental results, the following experimental values were selected to achieve the desired degree of functionalization. Therefore, the reaction conditions for the nitration process used in the preparation of N-LNR are shown in Table 1.

■ RESULTS AND DISCUSSION

Structural Characterization of N-LNR

In this work, the structure of the obtained N-LNR is confirmed, and the mechanism of the nitration reaction of LNR with HNO_3/Ac_2O is also proposed. The FTIR spectrum of the N-LNR is shown in Fig. 2. The peaks in the FTIR spectrum of the N-LNR are almost similar to those of the LNR. The same peaks correspond to the 1377, 1448, and 2800–3000 cm^{-1} absorption bands of the C–H bonds in the methyl ($–CH_3$) and methylene ($–CH_2–$) groups, as well as the peak at 1665 cm^{-1} of the C=C in the *cis*-vinylene group (>C=CH–). In addition, new absorption bands appeared in the spectrum of N-LNR at 1552, 1636, 1278, and 849 cm^{-1}. There, the absorption band at 1552 cm^{-1} indicates the presence of of the nitro group ($C–NO_2$). The absorption band 849 cm^{-1} shows the presence of O–N stretching of the

Table 1. Reaction conditions of the nitration process for the preparation of N-LNR

Samples	Ratio of HNO_3 to LNR (g/100 g)	Molar ratio of Ac_2O/HNO_3	Time (h)	Temperature (°C)
M1-1	**14**	1:2	2	10
M1-2	**21**	1:2	2	10
M1-3	**28**	1:2	2	10
M1-4	**42**	1:2	2	10
M1-1	14	**1:2**	2	10
M2-1	14	**1:1**	2	10
M2-2	14	**2:1**	2	10
M2-3	14	**3:1**	2	10
M3-1	14	2:1	**0.5**	10
M3-2	14	2:1	**1**	10
M3-3	14	2:1	**1.5**	10
M3-4	14	2:1	**1.75**	10
M4-1	14	3:1	2	**0**
M2-3	14	3:1	2	**10**
M4-2	14	3:1	2	**20**
M4-3	14	3:1	2	**30**

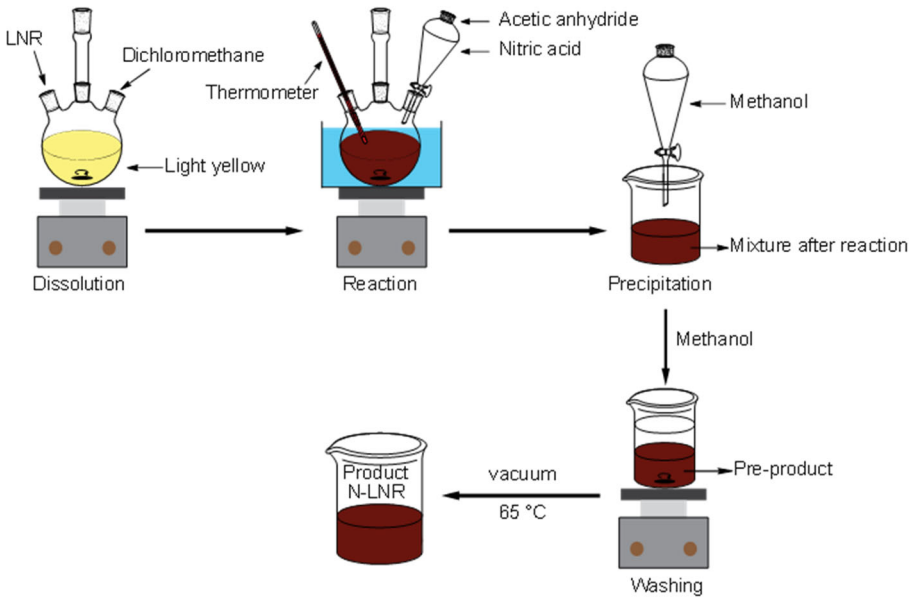


Fig 1. Diagram for the synthesis of N-LNR

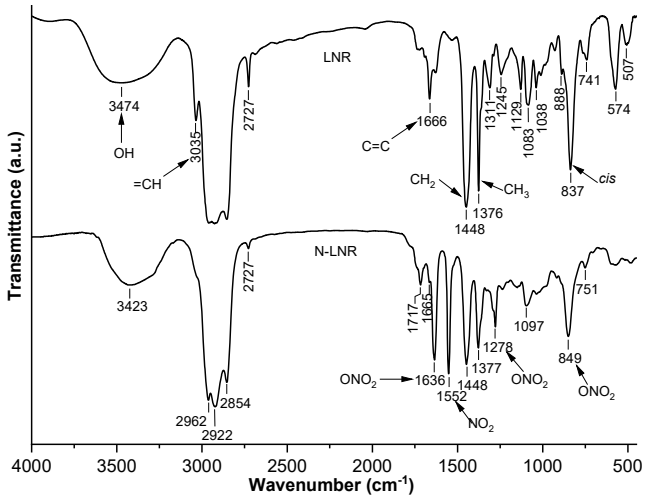


Fig 2. FTIR spectra of LNR and N-LNR

ONO_2 group. The absorption bands at 1636 and 1278 cm^{-1} confirm the presence of the nitrate group (C–ONO_2) [17]. Thus, this result demonstrates the successful nitration reaction resulting in the introduction of nitro and nitrate groups into the LNR chain. The structure of N-LNR was also elucidated through ^{1}H-NMR and ^{13}C-NMR spectra of the N-LNR, as shown in Fig. 3.

The ^{13}C-MNR and ^{1}H-NMR spectra of the N-LNR exhibit different shifts compared to those of the LNR. Like the NMR spectrum of the LNR, the ^{13}C-NMR of the N-LNR has a shift at 16.05 ppm of carbon in the –CH_3 group, shifts at 23.47 and 39.78 ppm of carbon in the–CH_2–group; shifts at 124.26 and 134.97 ppm of the >C=CH– group of the *cis*-1,4-isoprene units. Additionally, the N-LNR exhibits new shifts of 26.71 and 32.02 ppm of carbon bonded to nitro and nitrate groups (C–NO_2, C–ONO_2). The ^{1}H-NMR of the N-LNR has a shift at 1.59 ppm of the proton in the–CH_3 group, a shift at 1.97 ppm of the proton in the –CH_2– group, and a shift at 5.1 ppm of the proton in the >C=CH– group of the *cis*-1,4-isoprene units. Notably, there are shifts at 1.67 and 2.03 ppm of protons in the methylene groups next to the nitro and nitrate groups, in the N-LNR.

Based on the FTIR, ^{13}C-NMR, and ^{1}H-NMR spectra, the N-LNR has a structure consisting of the majority of the main chain units in the *cis*-1,4-isoprene form and a portion of the nitration units containing nitro and nitrate groups. The mechanism of the nitration of LNR by nitric acid can be proposed as follows: the reactive agent in the HNO_3 and Ac_2O mixture is formed via a pathway as illustrated in Eq. (4). In the pathway of Eq. (4), HNO_3 reacts with Ac_2O to form nitronium nitrate. The agent undergoes addition with the C=C double bonds of chain units, leading to two types of products, as shown in Eq. (5). According to Markovnikov's rule, the product formed in direction (Eq. (5a)) is the major product, while the product formed in direction (Eq. (5b)) is the minor product. Additionally, the presence of a small amount of

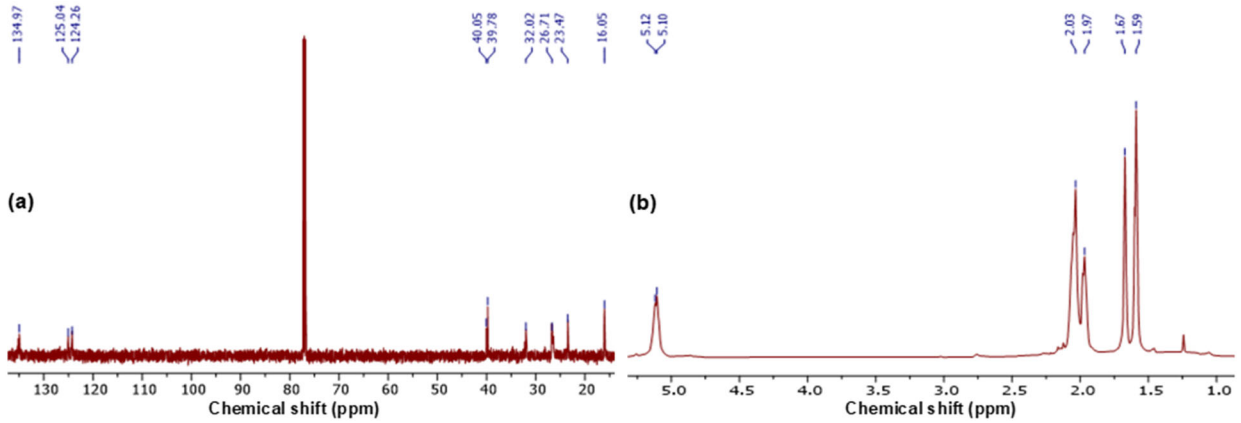
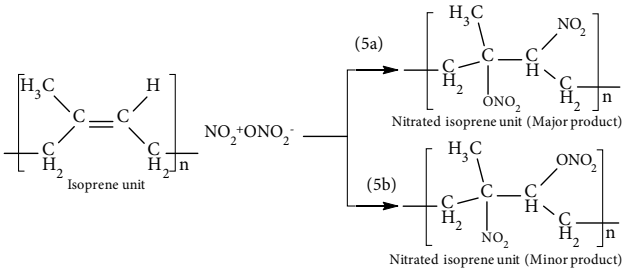


Fig 3. (a) ^{13}C-NMR and (b) ^{1}H-NMR spectra of the N-LNR

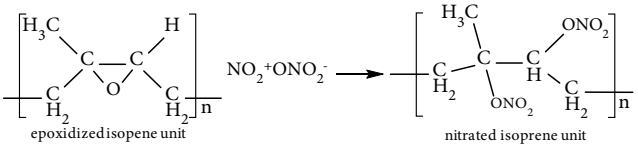
epoxy also participates in the reaction to form nitrate-type products, as described in Eq. (6).

$$Ac_2O + 2HNO_3 \longrightarrow NO_2^+ONO_2^- + 2CH_3COOH \quad (4)$$

Nitroni nitrate



(5)



(6)

Effect of the HNO_3/LNR Ratio on the Nitration

The degree of nitration is a parameter that determines the change in the physical and chemical properties, as well as the energy, of N-LNR compared to LNR. The degree of nitrate depends mainly on the ratio of HNO_3 to LNR. In this work, the reaction ratios of HNO_3 to the LNR were investigated to evaluate the degree of nitration and reaction yield. The reaction ratio of HNO_3 to LNR units was varied from 14 to 42 g/100 g while other conditions were fixed as follows: Ac_2O/HNO_3 ratio of 1:2, LNR/CH_2Cl_2 concentration of 0.05 g/mL, temperature of reaction of 10 °C, and period of reaction of 2 h. The results are shown in Fig. 4.

As shown in Fig. 4, the degree of nitration and yield of reaction go up when increasing the reaction ratio of

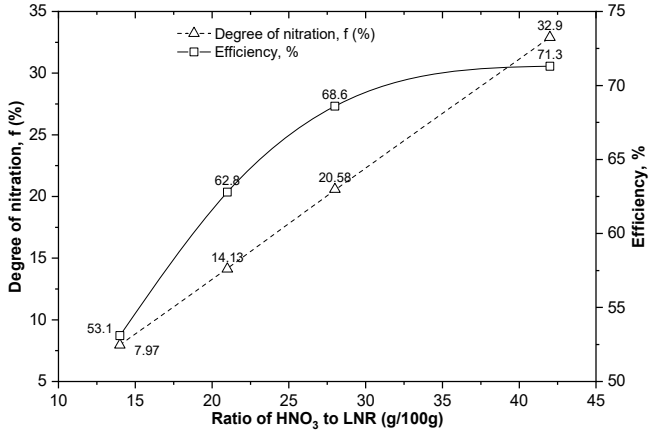


Fig 4. Effect of HNO_3-to-LNR ratio on the nitration

nitric acid to LNR. In particular, the degree of nitration seems to vary linearly with the change in the ratio of nitric acid to LNR. This linearity suggests that, up to a certain point, the concentration of the nitrating species remains sufficiently high to react stoichiometrically with the available reactive sites in LNR. Under the fixed conditions, the degree of nitration is largely governed by the initial concentration of HNO_3. Ac_2O serves to reduce water formation, thereby stabilizing the generation of NO_2^+ and maintaining an effective nitrating medium. Consequently, each incremental increase in the HNO_3/LNR ratio directly leads to an equivalent increase in the probability of nitration per unit, producing an almost linear dependence. The nearly linear relationship confirms that the nitration degree of LNR can be predictably tuned by adjusting the HNO_3/LNR ratio, offering a straightforward means of controlling the energetic content of N-LNR. This relationship also

provides valuable kinetic insight: within moderate acid ratios, the reaction follows pseudo-first-order behavior with respect to HNO_3 concentration. Additionally, the yield increases faster at low ratios and begins to slow down at higher ratios. This result suggested that the presence of the NO_2-functional group in N-LNR negatively affected the nitration of the next units. Thus, under these conditions, N-LNR with a degree of nitration ranging from 7.97% to 32.9% could be prepared by adjusting the ratio of HNO_3 to LNR.

Effect of the Ac_2O/HNO_3 Ratio on the Nitration

The Ac_2O interacts with nitric acid to form reagents for the reaction. To study the effect of Ac_2O on the nitration, the LNR is reacted with a mixture of acids with various ratios of Ac_2O to HNO_3 (Table 1). The degree of nitration and the yield of the nitration are shown in Table 2. The results showed that increasing the ratio of Ac_2O-to-HNO_3 resulted in a significant improvement in the degree of nitration and the yield of the reaction. However, the degree of nitration did not increase significantly when the ratio was increased from 2:1 to 3:1. It could be explained by the shift in the equilibrium of the reaction of the ONO_2-group formation when the Ac_2O, in addition to interacting with HNO_3, also acted as a reactant with the water produced by nitration [18-19]. This result suggests that the Ac_2O/HNO_3 ratio should be chosen as 2:1.

Effect of Reaction Time on Nitration

The influence of reaction time was investigated using 5 samples, spanning a range of 0.5 to 2 h. The remaining parameters were fixed as HNO_3/LNR of 14%, Ac_2O/HNO_3 of 2:1, LNR/CH_2Cl_2 of 5%, and a temperature of 10 °C. The degree of nitration and the yield of the reaction are determined and presented in Table 3.

The research results demonstrate a clear relationship between reaction time and the effectiveness of the process, as reflected by the degree of nitration (f%) and yield (%). Specifically, as the reaction time increased from 0.5 to 1.5 h, the degree of nitration rose from 8.74 to 10.12%, while the yield significantly increased from 58.3 to 67.5%. However, when the reaction time was extended to 1.75 and 2.0 h, the degree of nitration plateaued or slightly declined (11.10 and 10.03%, respectively), and the yield showed no significant improvement (67.1 and 66.9%, respectively). This indicates that the reaction likely approached equilibrium after approximately 1.75 h, and extending the reaction time further did not bring substantial benefits and could even reduce overall efficiency. Therefore, the optimal reaction time is identified as approximately 1.75 h, at which both degrees of nitration and yield reach their maximum values. This finding provides a crucial basis for optimizing the reaction process, enabling the efficient use of time and energy while maintaining high product yields.

Table 2. Effect of Ac_2O/HNO_3 ratio on the nitration

Samples	Ac_2O/HNO_3	Degree of nitration, f (%)	Yield%
M1-1	1:2	7.97	53.1
M2-1	1:1	9.71	64.7
M2-2	**2:1**	**10.03**	**66.9**
M2-3	3:1	10.48	69.9

Table 3. Effect of reaction time

Samples	Time (h)	Degree of nitration, f (%)	Yield%
M3-1	0.50	8.74	58.3
M3-2	1.00	9.61	64.1
M3-3	1.50	10.12	67.5
M3-4	**1.75**	**11.10**	**67.1**
M2-2	2.00	10.03	66.9

Effect of Temperature on Nitration

The temperature affects the rate of nitration [20-21]. The reaction temperature was varied from 0 to 30 °C to investigate this effect. The degree of nitration and the reaction yield at each temperature were determined and given in Table 4.

The results indicate that reaction temperature has a significant impact on both the degree of nitration and

Table 4. Effect of reaction temperature

Samples	Temperature (°C)	Degree of nitration, f(%)	Yield%
M4-1	0	7.47	49.8
M2-3	10	10.48	69.9
M4-2	**20**	**11.41**	**76.1**
M4-3	30	10.84	72.3

the yield of the product. Specifically, as the temperature increased from 0 to 20 °C, the degree of nitration rose from 7.47 to 11.41%, accompanied by a notable increase in yield from 49.8 to 76.1%. However, further increasing the temperature to 30 °C resulted in a slight decrease in both parameters, with a degree of nitration dropping to 10.84% and yield declining to 72.3%. This trend suggests that the optimal values for both the degree of nitration and yield may be achieved at around 20 °C. Moreover, the positive correlation between the degree of nitration and yield implies that an increase in the degree of nitration may contribute to enhancing reaction efficiency. The slight decrease at higher temperatures could be attributed to the formation of side reactions, reduced selectivity, or product degradation. Therefore, 20 °C appears to be the optimal temperature condition for maximizing yield within the studied range.

■ CONCLUSION

The N-LNR was successfully synthesized via the reaction of liquid natural rubber with concentrated HNO_3 in the presence of Ac_2O. The process parameters significantly affected the nitration degree and yield. By varying the HNO_3/LNR ratio from 14 to 42%, the degree of nitration was tunable between 7.97 and 32.9%, with a maximum yield of 76.1%. The optimal conditions are an Ac_2O/HNO_3 ratio of 2:1, a reaction time of 1.75 h, and a temperature of 20 °C. FTIR and NMR spectra confirmed that nitration occurred primarily at the C=C bonds of the *cis*-1,4-isoprene backbone, forming $-NO_2$ and $-ONO_2$ functionalities. The study establishes a reliable and scalable nitration method for LNR, providing a foundation for developing energetic polymeric binders for propellant and explosive formulations.

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■ CONFLICT OF INTEREST

The authors declare no conflicts of interest.

■ AUTHOR CONTRIBUTIONS

Khai Minh Doan and Trung Bao Tran conducted the experiment and wrote the manuscript. Tuan Duy Nguyen and Vuong Quoc Ly revised the manuscript. All authors agreed to the final version of this manuscript.

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