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TEMPERATURE EFFECT ON RAMI (*BOEHMERIA NIVEA L*) FIBRE-BASED NITROCELLULOSE SYNTHETIC

Kudrat Sunandar[1*], Lin Marlina[1], Marcellinus Christwardana[1], Rafi Ramanto[1], and Willy Dwi Novian[1], Hwai Chyuan Ong[2]
[1]Department of Chemical Engineering, Institut Teknologi Indonesia,
Jl. Raya Puspiptek Serpong, South Tangerang, 15320, Indonesia
[2]Future Technology Research Center, National Yunlin University of Science and Technology, Taiwan
* k_sunandar@iti.ac.id

Abstract.
Nitrocellulose as the basic component of a single base propellant type for rocket booster fuel has been developing for a long time, since 2010 the process of making nitrocellulose from pineapple leaves, palm frond, cotton fibers, and coconut fibers. Based on this, this research was directed to synthesize nitrocellulose from ramie (*Boehmeria nivea* L. Gaudich) fiber due to its cellulose content being relatively very high at around 97% through nitration reaction. The process of nitrocellulose synthesis was carried out in two stages. First, the process of separating cellulose from fiber, starting with the process of hydrolysis, delignin, and bleaching. Second, the reaction of nitration with nitric acid and sulfuric acid as a catalyst with temperature variation of 7ºC, 12ºC, 17ºC, 22ºC, and 27ºC with nitration reaction time is 15-60 minutes. The final step is a analysis using the Fourier Transform InfraRed (FTIR) tool and analysis using the Kjeldahl method nitrogen content. The results showed that the highest absorption of Nitro (-NO2) groups at the temperature 7ºC and the reaction time of 30 minutes with the percentage of Nitrogen in the cellulose is 10.791% N.

Keywords: *ramie, cellulose, nitration, nitrocellulose*

Introduction

Nitro cellulose can be used as a membrane, varnish in the paint manufacture, and a single base propellant [1]. Research on nitrocellulose has been developing for a long time, in 2010 the process of making nitrocellulose from pineapple leaves (*Ananas comosus*) was achieved yield is 86.2% with a nitrogen content is 11.56% at a ratio of 95% H2SO4 to 65% HNO3 of 3:1 and nitration time 90 minutes [2], from a palm brunch with a nitrogen content is 6.8% [3]. Research with cotton fibers states that the most produced nitro groups, at the reaction time is 30 minutes, reaction temperature 15^{O}C, and the composition of the acid mixture 60 ml H2SO4 and 45 ml HNO3 [4]. In 2017, nitrocellulose from coconut fibers [5] showed optimum results at ratio of 7:3 acid mixture (H2SO4:HNO3) with 10.85% of nitrogen content. Some of the studies mentioned above are quite good achievements, the theoretical maximum nitrogen content in nitrocellulose is 14.14% [6]. Some of research encourages to increase nitrogen content of nitrocellulose and trying other raw materials for cellulose. Ramie plants (*Boehmeria nivea* L. Gaudich) is natural fibers that have characteristics similar to cotton and used as textile raw materials. Another advantage of ramie is its productivity per hectare is much higher compared to cotton [7]. The ramie fiber production is directed towards the main target as raw material for the textile industry (cotton substitution as an imported product). It cannot be fully absorbed by the existing textile industry, only less than 25% can be absorbed by the national textile industry. Ramie: plant, stem and fibers are shown in Figure 1.

Figure 1.*Plant, stem and fiber*
(sources: www.semanticscholar.or/ramie)

For export, Indonesian ramie fiber cannot compete because the processing of ramie fiber is generally traditional [8]. To

anticipate the abundance of ramie fiber is use it as a pulp base to produces cellulose that processed into nitrocellulose (NC).

Research Methodology

Nitrocellulose is made by nitration reaction using nitric acid as raw material and H2SO4 catalyst. Based on this, this research is directed to synthesize nitrocellulose from ramie fiber through nitration reaction. The process consists of three stages, first, the process of isolating cellulose from ramie fiber. The process of cellulose isolation from flax fiber starts from the drying process, the blending and filtering process for 60 mesh sizes, the process of prehydrolysis, delignification with an alkaline solution (NaOH, 17.5%), and bleaching with H2O2. Second, the nitration process with H2SO4 and HNO3 nitration solution, the water content of the sample should not be exceeded 10%. Third, testing with FTIR and Kjedal to determine NO2 levels found in nitrocellulose, and nitrogen content. Through setting the temperature variation and the duration of the nitration reaction with a temperature of 7ºC, 12ºC, 17ºC, 22 ºC, and 27ºC and reaction times are 15 to 60 minutes.

Result and Discussion

The results of fiber analysis are quite high at 91.35% with 97.50% alpha cellulose, completely shown in Table 2, while the results of the analysis of nitrocellulose specifications are presented in Table 3. The amount of alpha cellulose content in fiber shows that it is very potential to be used as a source of cellulose in the production of nitrocellulose. The nitrogen content of the nitration reaction is still low because the delignification process was still incomplete as shown in Table 2. Lignin is very influential in the process of nitrogen penetration in cellulose.

Table 2. *Ramie fiber composition*

No	Parameters	Quantity (%)
1	Extractives	4.81
2	Hemicellulose	1.92
	Alpha cellulose	97.5
	Beta cellulose	2.32
	Gamma cellulose	0.18
3	Cellulose	91.35
4	Lignin	0.98
5	Ash Content	0.01

On the other hand, there was a possibility to use higher ratio of nitric acid and sulfuric acid, theoretically nitrogen would penetrate more to get higher nitrogen content. This is consistent with the phenomenon that occurs from the results of K. Hartaya's research in 2010 [9]. According to the Kjeldahl test, nitrogen content completed showed in Table 3 and FTIR result at 7ºC and 30 minutes showed at Figure 2.

Table 3*. Nitrogen content*

No	Temperature (ºC)	Times (minutes)	N (%)
1	7	15	6.6807
		30	10.791
		45	9.9925
		60	7.8798
2	12	15	9.8212
		30	9.707
		45	8.6221
		60	8.1082
3	17	15	4.4538
		30	9.5357
		45	9.0218
		60	5.4816
		15	9.3644

4	22	30	8.565
		45	9.6499
		60	9.1931
5	27	15	9.8212
		30	4.9677
		45	6.3381
		60	8.1082

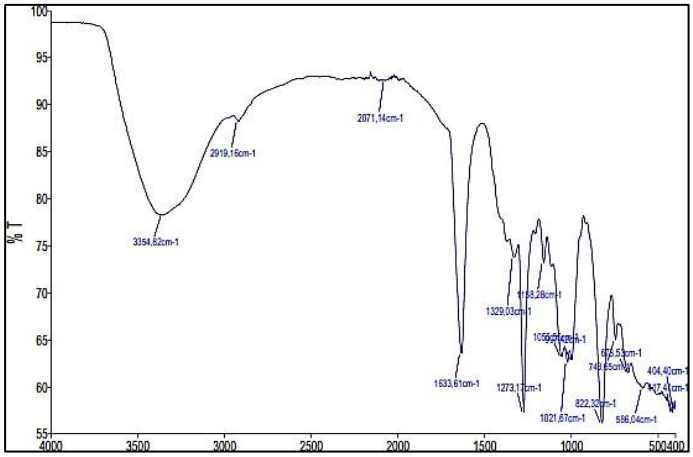

Figure 2. *FTIR for Nitrocellulose at 7°C and 30 minutes*

Meanwhile, the nitrogen content of nitrocellulose resulting from the reaction was still relatively low, which is 9.99 - 10.79% still below the nitrogen content requirement for propellant which is 12.7% as showed at Table 4.

Table 4. *Nitrocellulose specifications*

No	Parameters	Units	Value	
			Research	Standard
1	Nitrogen Content	%	10.79	12.5 - 12.7
2	Viscosity	mPas	120	700 - 1500
3	Moisture	%	28.8	27-32
4	Acetone Insoluble	%	0.35	0.4 max
5	Ash Content	%	0.33	0.4 max
6	Fineness	ml/10 gr dry	75-80	80 - 110

Conclusion

The cellulose content of ramie fiber is 91.35% with alpha cellulose 97.50% which is very potential to be used as a sources of cellulose in making nitrocellulose as a propellant raw material. The highest level of nitrogen is produced from the nitration reaction process at 7°C for 30 minutes which is 10.79%.

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References

[1] Saragih, E., Padil, and Yelmida. (2014). Pembuatan Nitroselulosa Dari Selulosa Hasil Pemurnian Pelepah Sawit dengan Hidrogen Peroksida (H2O2) Sebagai Bahan Baku Pembuatan Propelan. presented at the Teknik Kimia Universitas Riau, Teknik Kimia Universitas Riau, 2014.

[2] Tafdhila, B. P. I., Mahfud, and Prihatini, R. P. (2012). "Pembuatan Nitroselulosa dari Kapas (Gossypium Sp.) dan Kapuk (Ceiba Pentandra) Melalui Reaksi Nitrasi," *Teknik Pomits,* vol. 1, 1-6.

[3] Nuraini, Padil, and Yelmida. (2012). "Proses Pembuatan Nitroselulosa Dari Limbah Pelepah Sawit Dengan Variasi Waktu Dan Temperatur Nitrasi," presented at the Laboratorium Kimia Organik, Universitas Riau.

[4] Mudyantini, W. (2008). "Pertumbuhan, Kandungan Selulosa, dan Lignin pada Rami (Boehmeria nivea L. Gaudich) dengan Pemberian Asam Giberelat (GA3)," *Biodiversitas,* vol. 9(4), 269-274.

[5] Mulyadi, M., Setiadi, and Kusmartono, B. (2017). "Optimasi Proses Nitrasi pada Pembuatan Nitroselulosa dengan Memanfaatkan Limbah Serabut Kelapa Sebagai Bahan Baku dalam Upaya Mewujudkan Sumber Energi Bersih dan Terbarukan (Variabel Waktu Nitrasi dan Rasio Asam Penitrasi)," in *Prosiding Seminar Nasional XII "Rekayasa Teknologi Industri dan Informasi 2017*, Sekolah Tinggi Teknologi Nasional Yogyakarta.

[6] Suret, A. H. (2010). "Proses pembuatan pulp dari batang roella dengan proses soda "*Jurnal Teknik Kimia,,* vol. 17, 3.

[7] Tarmansyah, K. U. S. (2012). "Pemanfaatan Serat Rami Untuk Pembuatan Selulosa," presented at the Tim Puslitbang Indhan Balitbang Dephan.

[8] Setiadi, Y. Mulyadi, and Kusmartono, B. (2017). "Optimasi Pembuatan Nitroselulosa dari Daun Nanas dengan Proses Delignifikasi Dalam Upaya Mewujudkan Sumber Energi Bersih dan Terbarukan (Variabel Waktu Nitrasi dan Rasio Asam Penitrasi)," in *Prosiding Seminar Nasional XII "Rekayasa Teknologi Industri dan Informasi 2017*, Sekolah Tinggi Teknologi Nasional Yogyakarta.

[9] Hartaya, K. (2010). "Korelasi Kadar Nitrogen Dalam Nitroselulosa," *Majalah Sains dan Teknologi Dirgantara,* vol. 5(2), 74-85.