ASEAN
Journal of Scientific and Technological Reports
Online ISSN:2773-8752

Research article

Pyrolysis of Latex Sediment from Concentrated Latex Industry and Properties of Pyrolytic Products

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Citation:
Wongdaeng, F.; Chotisuwan, S.; Jariyaboon, R.; Kongjan, P. Pyrolysis of latex sediment from concentrated latex industry and properties of pyrolytic products. *ASEAN J. Sci. Tech. Report.* **2024**, 27(2), 21-28. https://doi.org/10.55164/ajstr.v27i2.250798.

Article history:
Received: September 3, 2023
Revised: December 2, 2023
Accepted: January 11, 2024
Available online: February 1, 2024

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Abstract: Concentrated latex is an intermediate rubber product in Thailand. It is an important industry that generates income for the country and Its southern region. The main products of the latex factory were concentrated latex and skim blocks. The industrial waste from the concentrated latex factory is called latex sediment. It appears to be a white mixed light-yellow sludge or a dark brown color, contains magnesium compounds, and phosphorus is an important element. Therefore, finding appropriate measures for rubber production and environmentally friendly industrial waste management is important. Therefore, this study investigates the pyrolysis of latex sediment as an alternative to industrial waste management. Pyrolysis of 100 g of latex sediment was performed at 300 °C, 500 °C, and 700 °C. From the experiment, it was found that the properties of the liquid product from the pyrolysis process at 500 °C were alkaline, with a pH of 9.45. However, acetic acid was the highest amount of organic substance in the liquid product. Substances in the amine and amide groups were also observed, a base result for the liquid product exhibiting an alkaline value. The pH is an important factor in the decomposition of organic substances under anaerobic conditions. Therefore, a 1 wt% liquid product was used in a volume of 13.5 mL, which is the most appropriate amount because it uses the least amount of liquid product to adjust the alkalinity of the skim latex serum before entering the anaerobic fermentation system. Pyrolysis is an environmentally friendly option for managing factory latex sediments for sustainable production and creating a good image for the organization.

Keywords: Pyrolysis; Latex Sediment; Concentrated latex; Skim latex serum

1. Introduction

Thai rubber products will expand from 2021 to 2023 in line with the related industries likely to grow. In addition, exports of concentrated latex still have the opportunity to grow as demand from The world market expands. Concentrated latex is an intermediate rubber product in Thailand. This important industry generates income for the country and southern region [1]. Since 2003, Thailand has been the world's largest producer of natural rubber. In 2011, Thailand produced latex for the first time. Approximately 3.4 million tons of fresh latex is tapped and collected for processing into primary rubber products such as concentrated latex, skim block rubber, and smoked rubber sheets. The primary rubber products are then processed into final rubber products such as condoms and gloves. The concentrated latex produced in Thailand is mostly exported to European countries, such as China, India, and Malaysia [2]. Concentrated latex is an intermediate rubber product in Thailand. The main products from the concentrated latex factory were concentrated latex and skim block rubber. Two types of waste are generated from the production processes of such rubber products.

1) Wastewater generated from the production of concentrated latex and skim blocks. The wastewater from both production processes is treated and collected into the wastewater treatment systems of all factories. During the anaerobic decomposition treatment process, biogas was produced, which needed to be disposed of by combustion at the flare system. It cannot be utilized as biofuel gas due to the low production rate of biogas and the high concentration of H_2S (20,000-50,000 ppm). Installing a biogas quality improvement system to eliminate H_2S is not cost-effective.

2) Waste from the production process of a factory is called latex sediment, which appears to be white, light-yellow, or dark brown sediment containing magnesium and phosphorus compounds as important elements. This is caused by adding chemicals to field latex in making latex concentrate, causing the magnesium ions in the latex to precipitate at the bottom of the tank. There are also organic and inorganic impurities, such as rubber, flour, dust, fat, protein, nitrogen compounds, and metal ions. The rate of latex sediment generation per ton of concentrated latex produced was calculated as a proportion of 0.6-50.0 kg latex sediment per ton of concentrated latex. Latex sediment is classified as a non-hazardous waste. It can be utilized directly as a raw material for improving soil or as latex sediment as a source of magnesium for plant growth [3]. The research study of [4] found that this latex sediment contains nitrogen, phosphorus, and potassium, which can be used as liquid and granular fertilizers. Alternatively, producing other materials for various uses could be further improved.

Since natural rubber and concentrated latex are majorly exported products of Thailand, information on sustainable production is necessary. Therefore, it is challenging for Thai rubber entrepreneurs to find appropriate measures to produce environmentally friendly rubber in many respects, including water pollution, odor problems, and industrial waste [2]. Latex sediment, an industrial waste, can be used for soil improvement. However, utilization of this latex sediment is not yet widespread. Therefore, the latex sediment generated by the company was granted permission to remain in the factory area while waiting to be transported to the fertilizer producers. As the production capacity of concentrated latex is likely to increase, the amount of latex sediment accumulates increasingly in the company's area. Therefore, improper management of latex sediment may cause increased pollution due to various microbial groups' biodegradation of organic substances in latex sediment. Waste management for maximum benefit and environmental friendliness is necessary for the concentrated latex industry to achieve sustainable production.

This study aimed to investigate the pyrolysis of latex sediment from concentrated latex industries and studied the alkalinity adjustment of skim latex serum from the skim block production process with liquid products from the pyrolysis process.

2. Materials and Methods

2.1 Material preparation

Latex sediment was collected from a concentrated latex industry in Songkhla Province, Thailand. The samples were pre-treated by drying at 105 °C for 24 h. [5] All chemicals in this work were of analytical grade and used without further purification.

2.2 Pyrolysis method

The equipment was set up according to the schematic diagram of the pyrolysis system shown in Figure 1. Initially, 100 g of the pre-treated sample was placed in a furnace tube and then heated for 1 h at 300 °C, 500 °C, and 700 °C. The system was operated at a heating rate of 20 °C/min, nitrogen gas flow of 1 L/min, as shown in Table 1, and a controlled cooling bath at 4 °C cooling temperature. The condensed pyrolysis liquid was collected using a glass liquid collector equipped with a condenser.

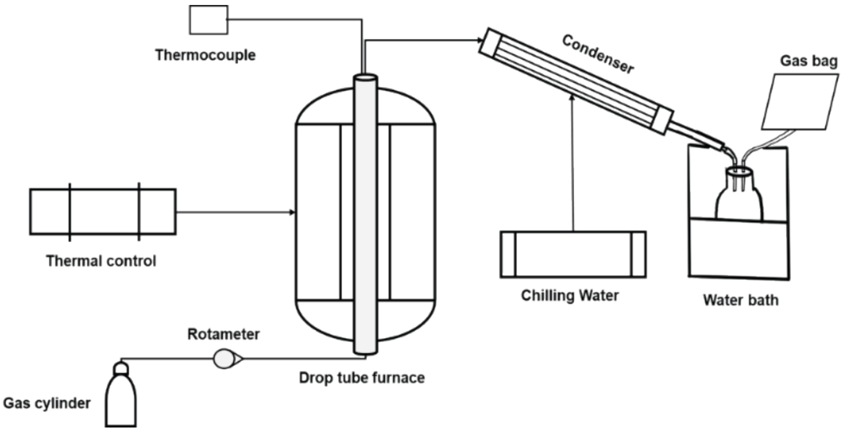

Figure 1. Schematic diagram of the pyrolysis system.

Table 1. Condition of pyrolysis process.

Conditions	Temperature (°C)		
	300	500	700
Initial sample (g)	100	100	100
Reaction time (hr.)	1	1	1
Heat rate (°C /min)	20	20	20
Flow rate (L/min)	1	1	1

2.3 Characterization method

The composition of the latex sediment before pretreatment was analyzed for moisture, total solids, volatile solids, and ash content, as shown in Table 2.

Table 2. Latex sediment composition analysis.

Parameter	Analytical methods/Equipment
Moisture	ASTM Standard D 3173-11
Total Solid (TS)	APHA, AWWA and WEF (1998)
Volatile Solid (VS)	APHA, AWWA and WEF (1998)
Ash Content	ASTM Standard D 3174-12

The pyrolytic liquid product was analyzed for pH, chemical oxygen demand (COD), and chemical compounds, as shown in Table 3. This work analyzed the chemical compounds using a gas chromatograph-mass spectrometer (7890B) and a mass spectrometer (5977AMSD), Agilent, USA.

Table 3. The pyrolytic liquid product analysis.

Parameter	Analytical methods/Equipment
COD (g/l)	ASTM Standard D 7544-12
pH	ASTM Standard D 7544-12
Chemical compound	ASTM Standard D 3173

2.4 The pyrolytic liquid product application

The pyrolytic liquid product was used to adjust the pH of skim latex serum before entering the wastewater treatment system, as shown in Table 4. The experiment began with pyrolytic liquid product samples were prepared by diluting with distilled water at concentrations of 1%, 3%, and 5%, and then used as a titrant in skim latex serum titration to determine the appropriate amount of liquid product for pH adjustment. Skim latex serum samples were prepared by adding sulfuric acid to 1000 g of skim latex to coagulate rubber. The rubber was then separated from skim latex serum.

Table 4. Experiment on skim latex serum pH adjustment.

Pyrolytic product	Vol. of skim latex serum (mL)	pH final
1% Liquid product	100	6.8
3% Liquid product	100	6.8
5% Liquid product	100	6.8

3. Results and Discussion

3.1 Composition of latex sediment

Sample of latex sediment from a concentrated latex factory in Songkhla Province. The two points where latex sediment was collected are the sludge sump pit and latex sediment collection area. The sludge sump pit collects latex sediment from the fresh latex receiving process and concentrated latex production department. The latex sediment was a white light-yellow solid. There is high moisture because the washing water from the production process flows into the pond at all times, as shown in Figure 2. The latex sediment was generated by adding ammonia to the latex field. This causes Mg to precipitate after leaving the latex overnight. The other part is from the centrifuge or disc centrifuge used to produce concentrated latex. The latex sediment collection area is wide open, with gutters around the storage area to accommodate latex sediment leachate and a pump system to transfer leachate into the factory wastewater treatment system. The latex sediment has less moisture because it is exposed to the sun, as shown in Figure 3.

Figure 2. Latex sediment from the sludge sump pit.

Figure 3. Latex sediment from latex sediment collecting area

Table 5. Compositions of latex sediment.

Parameter	Value	
	Sludge sump pit	Latex sediment collecting area
Moisture content (%w/w)	67.07 ± 3.78	62.78 ± 0.57
Total solid TS (%w/w)	38.20 ± 0.26	36.82 ± 0.14
Volatile solid VS (%w/w)	14.08 ± 0.14	14.40 ± 0.05
Ash (%w/w)	24.11 ± 0.18	22.42 ± 0.01

From Table 5, latex sediment from the concentrated latex production plant in January 2022 in the sludge sump pit had moisture, total solids, volatile solids, and ash content of the latex sediment equal to 67.07 (%w/w), 38.20 (%w/w), 14.08 (%w/w) and 24.11 (%w/w), respectively. Simultaneously, the latex sediment collecting area had moisture, total solids, volatile solids, and ash content of the latex sediment equal to 62.78 (%w/w), 36.84 (%w/w), 14.40 (%w/w) and 22.42 (%w/w), respectively. The experiment was able to criticize that latex sediment contained the least amount of volatile solids; therefore, the products obtained from pyrolysis had a low chance of being liquid and obtained the most solid product owing to the presence of inorganic substances as the main components in the latex sediment. At the latex sediment sampling site, the humidity of the latex sediment was higher in the sludge sump pit than in the latex sediment collecting area. Therefore, latex sediment in the latex sediment collection area is more appropriate for factory pyrolysis than latex sediment from the latex sediment storage tank.

3.2 Product yield

The pyrolysis experiments were conducted at different temperatures (300, 500, and 700 °C) for 1 h with a nitrogen gas flow rate of 1 L/min and a heating rate of 20 °C/min. The products have three phases: solid, liquid, and gas. The three product phases yielded unequal proportions at different temperatures. The products obtained by pyrolysis at various temperatures are listed in Table 6. Pyrolysis at 300 °C yielded the most solid product and the lowest amount of liquid products, 71 wt% and 19 wt%, respectively. Most of the gas obtained was nitrogen from the pyrolysis process. Pyrolysis at 500 °C showed that the highest amount of liquid product was 33 wt%, followed by 25 wt% at 700 °C. The liquid product obtained from pyrolysis was the main product required for this research. Therefore, the study of the conditions used in pyrolysis The most liquid product was sludge pyrolysis at 500 °C for 1 h.

Table 6. Product yield of pyrolysis of latex sediment at difference temperatures.

Temperature °C	Sample (g)	Product yields (wt%)		
		Solid product	Liquid product	Syngas
300	100.29 ± 0.08	71.39 ± 1.05	19.05 ± 0.34	9.85 ± 0.84
500	100.39 ± 0.14	56.58 ± 0.48	32.86 ± 0.74	10.95 ± 0.66
700	100.99 ± 0.07	47.94 ± 0.53	25.39 ± 0.65	27.66 ± 1.21

3.3 Properties of the pyrolytic liquid product

The liquid from the pyrolysis at 500 °C was a dark brown solution with a pungent odor. There were two layers: an aqueous phase and an organic phase. Some properties are shown in Table 7 and Figure 4. The pH value in this study was 9.45. This contradicts the research of [6] and [7], in which liquid products were acidic with pH equal to 4.50 and 2.40, respectively, possibly due to different starting materials. Consequently, the chemical compositions of the products were different. From Table 8, It was found that the highest organic substance in the liquid product was acetic acid, but substances in the amine and amide groups were also found, with the property of being a base. As a result, the liquid part of the product has high pH.

From the analysis of the COD value of the product, it was found that the COD value obtained was 26.75 g/L, which is similar to the research of [5], who studied various properties and compared the volume of methane accumulated from the decomposition process between wastewater and pyrolysis liquid from sludge pyrolysis at temperatures of 250, 350, 450, and 550 °C at the ratio (S/I) 30%:70% (by volume) from the mesophilic group. It was found that the highest volume of methane gas produced was at temperatures of 250 ˚C, 350 ˚C, 450 ˚C, and 550 ˚C, where the methane yields were 461.0 mL/g-VS, 366.1 mL/g-VS, 233.1 mL/g-VS and 207.8 mL/g-VS were inconsistent with the preliminary properties study. These include total solids, volatile solids, and COD. Because the highest amount of organic matter is found at 550 °C, the highest methane gas production should be obtained. Increased organic matter may inhibit microorganisms that produce methane gas. Another reason is the pH of the system because, at a temperature of 550 °C, there is a pH as high as 8.88, which is not suitable for the growth of microorganisms. Because acid-producing and methane-producing microorganisms can work together well, the pH value must be maintained within an appropriate range of approximately 6.8-7.2 [8]. Therefore, it can be concluded that using liquid products in fermentation systems for direct anaerobic treatment may not be appropriate as it may be toxic to the system.

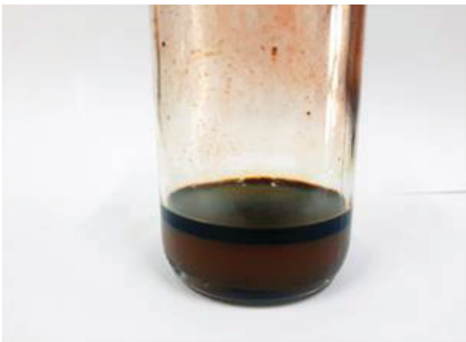

Figure 4. Pyrolytic liquid products at 500 °C.

Table 7. Properties of pyrolytic liquid products.

Properties	Asadullah et al. (2007) at 500 °C	Sara B. (2018) at 500 °C	Lutfiyah S. (2018) at 550 °C	This study at 500 °C
pH	4.50	2.40	-	9.45
COD (g/L)	-	-	23.83	26.75

Table 8. Identified compounds in latex sediment pyrolytic liquid at 500°C.

Compound Name	Formula	%Area
Anhydrosecoisolariciresinol	$C_{20}H_{24}O_5$	0.23
Pyridine	C_5H_5N	2.96
2,3-Dimethylpyridine	C_7H_9N	0.74
3-Methylpyridine	C_6H_7N	1.86
2,3-Dimethylpyridine	C_7H_9N	0.92
N-(2-Aminoethyl)-N-methyl ethylenediamine	$C_5H_{15}N_3$	0.39
Acetic acid	$C_2H_4O_2$	7.20
Pyrrole	C_4H_5N	3.30
Isobutyric acid	$C_4H_8O_2$	0.52
Butanoic acid	$C_4H_8O_2$	0.45
4-Methylhex-5-en-4-olide	$C_7H_{10}O_2$	1.58
Acetamide	C_2H_5NO	3.29
Propionamide	C_3H_7NO	0.90
2-Pyridinamine	$C_5H_6N_2$	1.09
(3S,4S)-3-fluoro-2-phenylheptan-4-ol	$C_{13}H_{19}FO$	0.54
2-Pyrrolidinone	C_4H_7NO	1.95
3,4,5-Trimethylpyrazole	$C_6H_{10}N_2$	2.04
1,2,3-Propanetriol	$C_3H_8O_3$	4.55
3-Pyridinol	C_5H_5NO	3.57
2-Methoxy-6-methyl pyrazine	$C_6H_8N_2O$	0.95
Parthenin	$C_{15}H_{18}O_4$	0.45
Benzeneacetamide	C_8H_9NO	0.06
Dibutylformamide	$C_9H_{19}NO$	0.60
5,5-Dimethylhydantoin	$C_5H_8N_2O_2$	2.40
5-Ethyl-5-methylhydantoin	$C_6H_{10}N_2O_2$	1.28
N,N-Dipropyl-1-butanamine	$C_{10}H_{23}N$	0.74

Table 8. Identified compounds in latex sediment pyrolytic liquid at 500°C (continued).

Compound Name	Formula	%Area
5-Ethyl-5-methylhydantoin	$C_6H_{10}N_2O_2$	1.28
N,N-Dipropyl-1-butanamine	$C_{10}H_{23}N$	0.74
5-Isopropyl-2,4-imidazolidinedione	$C_6H_{10}N_2O_2$	5.12
5-Isopropyl-2,4-imidazolidinedione	$C_{11}H_{18}N_2O_2$	0.43
5-Isopropyl-2,4-imidazolidinedione	$C_6H_{10}N_2O_2$	1.44
3-Isobutylhexahydropyrrolo[1,2-a]pyrazine-1,4-dione	$C_{11}H_{18}N_2O_2$	0.69
N,N'-Methylenebisacrylamide	$C_7H_{10}N_2O_2$	0.44

3.4 The alkalinity of skim latex serum was adjusted using liquid products from the pyrolysis process.

The pH is an important factor in organic decomposition systems under anaerobic conditions. The optimum pH range was approximately 6.8-7.2. If the pH value is higher or lower, the system performance will decrease rapidly; if the pH value is lower than 6.6 or higher than 7.6, the system performance will quickly decrease [8]. From a study of the use of the liquid product to adjust the alkalinity of skim latex serum before entering the anaerobic fermentation system, as shown in Figure 5, it was found that skim latex serum from the skim block production process had a pH of 4.95, which has the condition somewhat acidic and is toxic to anaerobic fermentation systems. Adjusting the alkalinity of skim latex serum from a pH of 4.95 to 6.80 before entering the organic decomposition system under anaerobic conditions is necessary and extremely important. From the experiment, as shown in Table 9, it was found that using the liquid product at 1 wt% liquid product (13.5 mL) was the most appropriate amount because it used the least amount of liquid product to adjust the alkalinity of skim latex serum.

Table 9. Pyrolytic liquid products application.

Pyrolytic product	Vol. of skim latex serum (mL)	pH initial	pH final	Vol. of pyrolytic product (mL)
1% Liquid product	100.01 ± 0.02	4.95 ± 0.00	6.80 ± 0.01	13.53 ± 0.15
3% Liquid product	100.04 ± 0.01	4.95 ± 0.00	6.81 ± 0.02	12.20 ± 0.20
5% Liquid product	100.02 ± 0.02	4.95 ± 0.00	6.81 ± 0.01	11.17 ± 0.15

Figure 5. Preparation of skim latex serum and adjustment of alkalinity of serum water with liquid products from pyrolysis at a temperature of 500 °C.

As shown in Figure 5, serum was prepared from 1,000 grams of latex, and sulfuric acid was added to coagulate the rubber. Skim latex serum was collected after coagulation. The product was obtained as a yellow liquid. Has a relatively low pH. Skim latex serum from the skim block production plant was released into a stabilization pond before entering the anaerobic digester. This can result in a rapid decrease in the pH of the digester. The factory solves this problem by adjusting the alkalinity condition by adding chemicals such as lime because the cost is lower than that of sodium bicarbonate and sodium hydroxide; however, the factory requires a large amount of water to dilute the lime. It was found that lime did not dissolve completely and precipitated. When entering the anaerobic digester, the sediment accumulates at the bottom of the pond, causing the pond capacity to decrease, and the amount of lime solution used to adjust the pH appropriately requires a large amount. This increases the amount of wastewater that wastes energy during treatment. Therefore, using a liquid product from the pyrolysis of the latex sediment at a temperature of 500 °C is an option to adjust the alkalinity of the wastewater appropriately to increase the efficiency of the anaerobic digester. This could be a beneficial guideline for managing and utilizing sediment residue generated from the concentrated latex production process.

4. Conclusions

The batch process of pyrolysis of latex sediment from concentrated latex industries at temperatures ranging from 300-700°C was successfully demonstrated. The alkaline pH of the pyrolytic liquid was around 9.45. Therefore, the liquid pyrolytic product is suggested to be added to acidic serum wastewater for pH improvement before feeding to the anaerobic digestion system.

5. Acknowledgements

The authors thank the financial support from the Research Grant for Master Thesis, Graduate School and Faculty of Science and Technology, Prince of Songkla University.

Author Contributions: F.W. conducted the research, analyzed the data, and wrote the paper; S.C., R.J., and P.K. validated the results and reviewed a manuscript; S.C., R.J., and P.K. reviewed the manuscript and original draft preparation; all authors had approved the final version.

Funding: This research was supported by a Thesis Research Grant for graduate students, Department of Applied Chemistry, Faculty of Science and Technology, Prince of Songkla University.

Conflicts of Interest: The authors declare no conflict of interest.

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