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EFFECT OF CENTRIFUGATION SPEED OF PINEAPPLE EXTRACT AS NATURAL COAGULANT ON RUBBER CHARACTERISTICS

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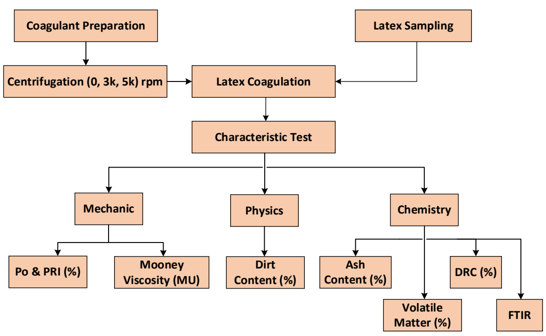
Graphical abstract



Abstract

Natural rubber is one of the leading plantation commodities widely cultivated in Indonesia to produce products useful for human needs. This research aims to determine the effect of centrifugation speed on the properties of PB 260 clone rubber using pineapple extract coagulant. Among rubber farmers, there are obstacles in processing natural rubber, such as reliance on chemical coagulants and the lack of cheap and safe alternatives. Therefore, it is necessary to find coagulants that are affordable, safe, and effective. Pineapple extract was used as the natural coagulant in this research. The latex used was 150 mL of PB 260 clone rubber, with 75 mL of 100% natural coagulant and 2% of formic acid. The rubber properties tested included Initial Plasticity (P_O), Plasticity Retention Index (PRI), Mooney viscosity, dirt content, ash content, volatile matter, Dry Rubber Content (DRC), and FTIR analysis. The research results showed that the highest values for P_O, PRI, DRC, ash content, dirt content, and mooney viscosity values of 39.5%, 88.61%, 30.95%, 0.48%, 0.19%, and 62 MU respectively, were obtained using 0 rpm pineapple extract (without centrifugation), with. Meanwhile, volatile matter content was highest at 0.37% with pineapple extract centrifuged at 5000 rpm. This study revealed that higher centrifugation speeds increased the pH of the natural coagulant and decreased the concentration of H^+ ion, which led to an accelerating of the latex coagulation process. So it can be concluded that pineapple extract can be used as a substitute for chemical coagulants as it effectively coagulates latex. The rubber from coagulation process using pineapple extract complies with Indonesian National Standard (SNI) for rubber characteristics. Centrifuged pineapple extract offers lower value of impurities and ash content while non centrifuged extract yields higher value of Po and PRI.

Keywords: Centrifugation, Latex, Natural coagulant, PB 260 clone

1.0 INTRODUCTION

Natural rubber, which comes from the rubber tree with the Latin name *Havea brasiliensis*, is a material used in the manufacture of various products useful to humans [1]. Based on data from the Central Statistics Agency [2], natural rubber production in Indonesia reached 2.65 million tons in 2023, with an area of 3,690,000 hectares. This number represented a decrease of 2.22% from the previous year when production reached 3.14 million tons in 2022. This large amount of production can become a source of livelihood for the community, as rubber is now used not only to make toys and household products but as well as construction, packaging, and health products [3].

Trade specifications, widely known as the Indonesian Rubber Standard (SIR), are used for rubber, requiring the selection of a type of rubber that is of good quality and meets established standards. An example of this is clone PB 260, which is the result of a cross between clones PB 5/51 and PB 49, which are known to produce latex. Clone PB 260 is known for its high metabolic rate, efficient latex regeneration system, and satisfactory latex production. Other advantages include high latex productivity, rapid derivation, and very rapid growth [4].

The rubber processing process faces several challenges, particularly the use of chemical coagulants such as formic acid to coagulate latex, which has negative impacts on rubber farmers, processing equipment, and the environment [5]. Formic acid is a hazardous chemical when used without proper handling. Formic acid can irritate if it comes into direct contact with the skin, and hurts the sustainability of the ecosystem if it is not disposed of properly. Discovering alternative natural coagulants that are environmentally friendly and do not hurt rubber farmers and rubber processing infrastructure are aims of this research.

In this research, pineapple (*Ananas comosus*) has been used as a natural coagulant in the coagulation or rubber-curing process. The intention is to find out how variations in centrifugation speed affect the separation of pineapple fruit extract.

In our previous research [6], latex was coagulated with various types of natural coagulants such as Angifera Odorata, Garcinia atroviridis, Mangosteen, Physalis, and Rambutan. All natural coagulants were centrifuged at 3000 rpm for 1 hour and compared with those without centrifugation. The results of the rubber characteristics obtained met the SNI 06-1903-2017 standard for SIR 20. In addition, centrifuged natural coagulants obtained better rubber characteristics compared to chemical coagulants such as formic acid. This is the basis for our further research to determine the effect of centrifugation speed on the characteristics of the rubber produced.

The natural coagulant extract used is often not properly separated or filtered between the filtrate and the grains or impurities, which can cause the rubber produced to be dark in color and have characteristics that do not meet SNI standards due to high levels of impurities. For this reason, this research aims to separate impurities from natural coagulants using centrifugation.

The centrifugation method is a separation process that utilizes the effects of centrifugal force and rotational movement on each molecule of the suspension substance. The centrifugal force generated when an object rotates pushes molecules away from the center of rotation [7]. High centrifugation speeds can deposit relatively large and dense particles [8].

Another aim is also to analyzing the effect of using natural coagulants on rubber properties such as Initial Plasticity of Rubber (P_o), Plasticity Retention Index (PRI), Mooney Viscosity, Dirt Content, Ash Content, Volatile Matter, Dry Rubber Content (DRC) and Fourier Transform Infrared Spectroscopy (FTIR) by SNI 06-1903-2017 and meet the requirements of SIR 20 rubber standard as also the aims of this research.

2.0 METHODOLOGY

The natural coagulant centrifugation process was prepared and conducted at the Microbiology Laboratory of Institut Teknologi Sumatera. While, testing of rubber characteristics was performed at PT Diagnostic Laboratory, Perkebunan Nusantara VII, Way Berulu unit, Pesawaran, Lampung. The latex samples were collected from subdivision 1 of the Way Berulu unit using a D3 rubber tapping system, which was conducted once every three days, and coagulants from pineapple and formic acid were used. The rubber clone used was PB 260, planted in 2007 (16 years old), with tapping starting in 2012. The research diagram is attached in Figure 1.

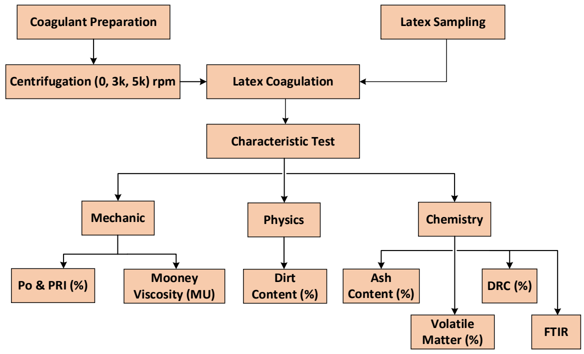


Figure 1 Experiment flow diagram

2.1 Preparation of Coagulant

The natural coagulant used was pineapple, while the chemical coagulant used as a control variable was formic acid at a concentration of 2%. The pineapple was washed with water and then put into a blender. The mashed pineapple was filtered to separate the extract from the pulp. The fruit extract was centrifuged at speeds of 0 rpm (without centrifugation), 3000 rpm, and 5000 rpm for 60 minutes. The pH of the fruit extract was then measured at 0 rpm (without centrifugation), 3000 rpm, and 5000 rpm. The fruit extracts obtained at 0 rpm, 3000 rpm, and 5000 rpm were placed in beakers with a volume of 75 mL for each extract to thicken the latex.

2.1.1 Latex Sampling Procedure

Latex sampling was carried out on PB 260 clone rubber trees periodically, once every three days, using a 3-tap D3 tapping system. The tapped latex was placed in a sample bottle and then filtered to avoid contamination with dirt. For each sample, 150 mL of latex was collected and weighed for testing.

2.1.2 Latex Coagulation Process

150 mL of weighed latex was placed in a coagulation vessel. The natural coagulant and latex were mixed in a 1:2 ratio of 75 mL and then stirred until evenly distributed. The latex coagulation time was recorded with a stopwatch until the latex formed a clot. After coagulation, the rubber was separated from the coagulated water using a measuring cup. The pH of the water or serum resulting from coagulation was measured, and any color changes that occurred were observed.

2.1.3 Characterization of Rubber

In this research, tests were conducted on the properties of rubber, including mechanical, physical, and chemical tests. Mechanical testing included the initial plasticity test (P_O), Plasticity Retention Index (PRI) according to the SNI ISO 2013 standard, and Mooney viscosity. Physical testing included dirt content testing according to SNI 8383:2017. Chemical testing included ash content testing according to SNI ISO 247:2012, volatile matter testing according to SNI 8356:2017, Dry Rubber Content (DRC), and Fourier Transform Infrared Spectroscopy (FTIR).

3.0 RESULTS AND DISCUSSION

3.1 Effect of the Type of Coagulant on the pH of Latex Coagulation

Coagulants are used to accelerate the clumping of latex, which is caused by lowering the pH of the latex. The purpose of pH measurement is to identify the type of acid and the concentration of H^+ ions in each natural coagulant extract. The results of how the type of coagulant affects the pH of the latex coagulation can be seen in Figure 2.

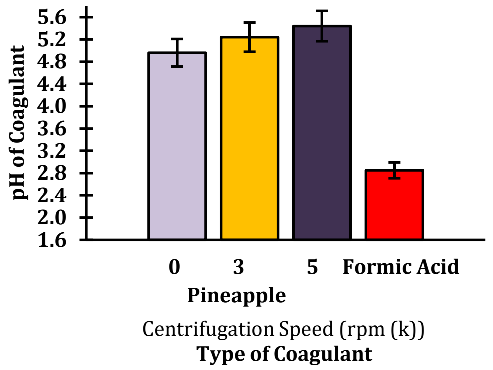


Figure 2 Effect of coagulant type on latex coagulation pH

Treatment of natural pineapple coagulant extract with centrifugation speeds of 0 rpm, 3000 rpm, and 5000 rpm affects the pH value. In this study, the highest pH value was observed in the natural pineapple coagulant that was centrifuged at 5000 rpm, reaching 5.44, while the lowest pH value was found in the 2% formic acid chemical coagulant, reaching 2.85. These results confirm that natural coagulants centrifuged at 3000 rpm, and 5000 rpm had higher pH values than those not centrifuged. A higher level of centrifugation speed induce to an increased in the pH of the natural coagulant and a decrease in the H^+ ions. By the reason of organic acids such as ascorbic acid, malic acid, citric acid, and oxalic acid present in natural clotting factors are precipitated during centrifugation, reducing the amount of these organic acids in the clotting factor [9].

3.2 Effect of the Type Of Coagulant on The Coagulation Time of Latex

The coagulation time is measured from the time the coagulant is added to the latex until the latex coagulates. The natural pineapple coagulant used can coagulate latex due to the natural coagulant used contains organic acids in varying amounts [9]. The effect of coagulant type on latex coagulation time is shown in Figure 3.

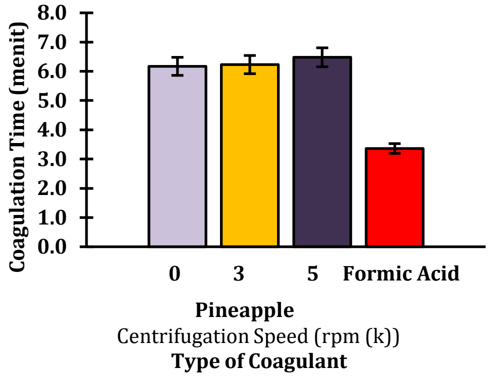


Figure 3 Effect of coagulant type on latex setting time

Figure 3 shows that the coagulation time for latex with 2% formic acid as the chemical coagulant was the fastest at 3.36 minutes and the longest coagulation time for natural pineapple coagulant treated with 5000 rpm centrifugation was 6.48 minutes. These results indicate that the coagulation time of chemical formic acid coagulants is faster than natural pineapple coagulants owing to the lower pH of formic acid compared to pineapple. Pineapple coagulant without centrifugation treatment (0 rpm) was faster than pineapple coagulant treated with centrifugation at 3000 rpm, and 5000 rpm. The longer coagulation time when natural coagulants are centrifuged is in consequence of the precipitation of the organic acids they contain. In contrast to natural coagulants without centrifugation treatment, latex requires a shorter coagulation time due to the higher organic acid content. The pH and compound content of the coagulant affect the coagulation time of latex. According to Rusiardy, et al. [10], the lower the pH of the coagulant, the greater the strength of the H^+ ions to bind OH^- ions in the latex, thereby accelerating the coagulation time of the latex.

3.3 Effect of Coagulant Type on P_O

Initial Plasticity (P_O) is a measure of rubber plasticity that is tested directly without any special pre-treatment and is usually measured using a Wallace Plastimeter [11]. P_O analysis is performed to obtain initial plasticity values that reflect the initial resistance to degradation, which are later used in the PRI test [12]. The results of the effect of coagulant type on P_O are shown in Figure 4.

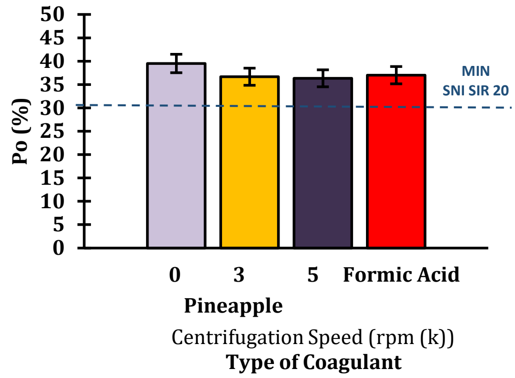


Figure 4. Effect of coagulant type on P_O

Figure 4 shows that pineapple coagulant without centrifugation treatment (0 rpm) has a higher P_O value, namely of 39.5%, while pineapple treated with centrifugation at 5000 rpm has a lower P_O value of 36.33%. Formic acid as a chemical coagulant has a P_O value of 37%. Pineapple coagulant and formic acid met the requirements of the SNI 06-1903-2017 standard for SIR 20 minimum 30%. The centrifugation speed affects the P_O value; the higher the centrifugation speed, the lower the P_O value. This is due to the reduction in organic acids contained in natural coagulants, leading to an increased separation of antioxidant content [10]. Higher the P_O value, the better the rubber properties. A high P_O value makes the rubber molecular chain longer and the rubber more resistant to oxidation, whereas a low P_O value makes the rubber molecular chain shorter. This is because rubber with short molecular chains oxidizes easily and has a soft texture. The P_O value is influenced by several factors, namely improper processing of rubber raw materials and excessive use of acid in the latex coagulation process [13].

3.4 Effect of Coagulant Type on PRI

The plasticity retention index (PRI) indicates how well the raw rubber resists oxidation at high temperatures before and after the drying process at 130 °C for 30 minutes [14]. The results of the effect of the type of coagulant on the PRI are shown in Figure 5.

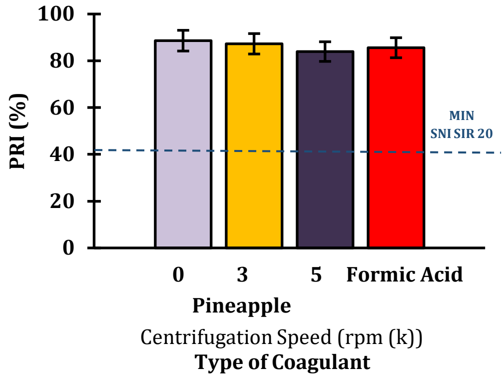


Figure 5 Effect of coagulant type on PRI

Based on Figure 5, the highest PRI value was obtained for pineapple without centrifugation (0 rpm), namely 88.61%, and the lowest PRI value was obtained for pineapple centrifuged at 5000 rpm, namely 83.94%, while for formic acid the PRI value was 85.59%. Pineapple coagulant and formic acid met the requirements of the SNI 06-1903-2017 standard for SIR 20, where the minimum standard value is 40%. The PRI value is influenced by metal ions, organic acids contained in the latex, antioxidants, and the time taken to perform the PRI analysis [15].

The antioxidant content of organic acids in natural coagulant extracts can reduce the degradation of rubber molecules due to the oxidation process at high temperatures [16]. The centrifugation speed affects the PRI value; a higher centrifugation speed will cause the PRI value to decrease. This is because the higher the centrifugation speed, the more the organic acids contained are precipitated and the antioxidant substances contained in the organic acids are increasingly lost. The rubber will then oxidize more easily and the PRI will decrease [17].

3.5 Effect of Coagulant Type on Mooney Viscosity

Mooney viscosity is a property used to test the plasticity of rubber. Mooney viscosity describes the molecular chain length of natural rubber. In this research, Mooney viscosity is used as a parameter to determine the properties of natural rubber [13]. The results of the analysis of the effect of coagulant type on Mooney viscosity are shown in Figure 6.

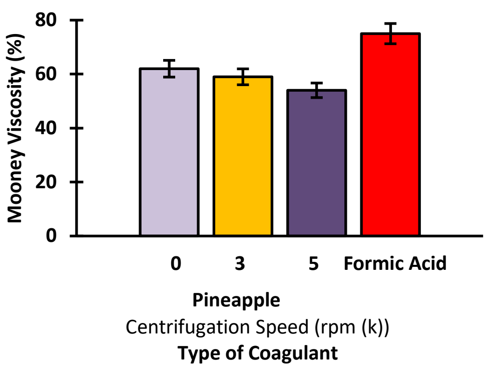


Figure 6 Effect of coagulant type on Mooney viscosity

Based on Figure 6, the highest Mooney viscosity value was obtained for formic acid, namely 75 MU, and the lowest Mooney viscosity value was obtained for pineapple centrifuged at 5000 rpm, namely 54 MU. The high Mooney viscosity value describes the number of natural rubber molecular chains that have branched out, increasing the length of the rubber molecular chain [18]. The Mooney viscosity is influenced by the organic acids present in natural coagulants. Low Mooney viscosity is due to organic acids that are increasingly precipitated during centrifugation, while high Mooney viscosity is due to organic acids that are still abundant in natural coagulants. The higher the centrifugation speed, the lower the organic acid content and the lower the Mooney viscosity. This indicates that the molecular chain of the rubber is becoming shorter [19].

3.6 Effect of Coagulant Type on Dirt Content

The dirt content indicates the amount of non-rubber content that cannot pass through a 325mesh filter [20]. The dirt content is influenced by several factors such as the coagulant and equipment used, the cleanliness of the latex, and the processing machine parts. The results of the effect of the type of coagulant on the dirt content are shown in Figure 7.

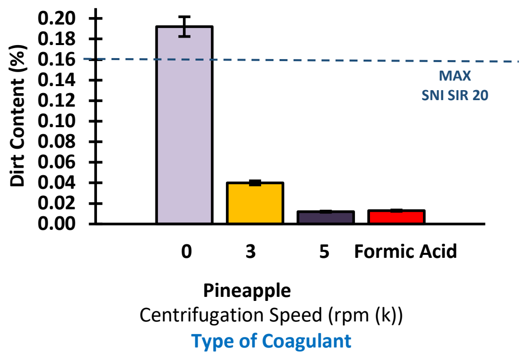


Figure 7 Effect of coagulant type on dirt content

Based on Figure 7, the highest value of impurity content was obtained in a pineapple without centrifugation (0 rpm), namely 0.192%, and the lowest value of impurity content was formic acid with 0.013%. All coagulants accordance to the requirements of SNI 06-1903-2017 for SIR 20, where the maximum value for the dirt content test standard is 0.16%, except for pineapple coagulants without centrifugation treatment (0 rpm). In this analysis, the coagulant impurity content of 0 rpm pineapple was higher compared to the centrifugation treatment due to the high fiber content still present in the pineapple fruit extract. Centrifugation speed affects the level of impurities in the natural coagulant; the higher the centrifugation speed, the more fiber or sand impurities in the natural coagulant will be precipitated, further reducing the level of impurities. High levels of impurities in rubber can reduce the dynamic properties of rubber products, such as the elasticity and flex cracking resistance of natural rubber [21].

3.7 Effect of Coagulant Type on Ash Content

The ash content in rubber comes from the metal content in the form of potassium, magnesium, calcium, sodium, and other mineral elements. The ash content also comes from minerals in the form of silicates [15]. The results of the effect of the type of coagulant on the ash content are shown in Figure 8.

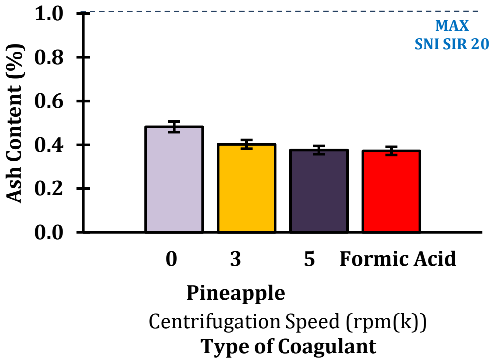


Figure 8 Effect of coagulant type on ash content

Figure 8 shows that the highest ash content value in a pineapple without centrifugation (0 rpm) is 0.482% and the lowest ash content value in formic acid is 0.372%. Pineapple coagulant and formic acid meet the requirements of SNI 06-1903-2017 for SIR 20, where the maximum value for the ash content test standard is 1%. The high ash content before centrifugation is because there is still a large amount of unseparated mineral content in the natural coagulant extract compared to after centrifugation (3000 rpm and 5000 rpm). Meanwhile, the high ash content in formic acid is owing to the dilution process of formic acid with water, which still contains a lot of minerals. Minerals in rubber can affect the dynamic properties of rubber products, such as the elasticity and flex cracking resistance of natural rubber [22].

3.8 Effect of Coagulant Type on Volatile Matter

Volatile matter indicates the amount of water, serum, and other substances remaining in the rubber after oven drying [6]. If the amount of evaporated substances in the rubber is lower, then the rubber has a better quality since it indicates that the water content in it is lower. The results of the effect of the type of coagulant on the level of volatiles are shown in Figure 9.

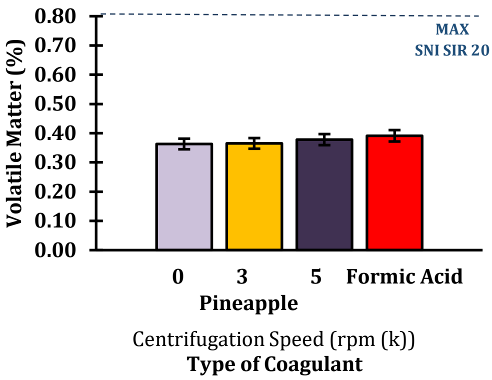


Figure 9 Effect of the type of coagulant on the volatile matter

Figure 9 depicted the highest value of volatile matter in formic acid and the lowest value of volatile matter in pineapple coagulant without centrifugation (0 rpm) is 0.391% and 0.363%, respectively. Pineapple coagulant and formic acid meet the requirements of SNI 06-1903-2017 for SIR 20, where the maximum value for the impurity content test standard is 0.80%. The centrifugation speed affects the volatile content of the rubber. The higher the centrifugation speed, the faster the organic acids will be settled on the natural coagulant, so that the coagulation process will not be completed. In addition, less latex coagulates and retains a greater amount of water. This results in a higher evaporation rate. Meanwhile, with 2% formic acid, the volatile content is higher due to the dilution process uses water, whereas natural coagulants do not use water at all. Volatiles in rubber can cause unpleasant odors and mold growth [23]. High levels of volatiles reduce the quality of the rubber.

3.9 Effect of Coagulant Type on DRC

Dry Rubber Content (DRC) is the number of rubber particles in the rubber latex expressed as a percentage. The higher the DRC value, the better the quality of the sample produced because the dry rubber content in it is also higher, thus increasing the quality of the sample [16]. The results of the effect of the type of coagulant on the DRC are depicted in Figure 10.

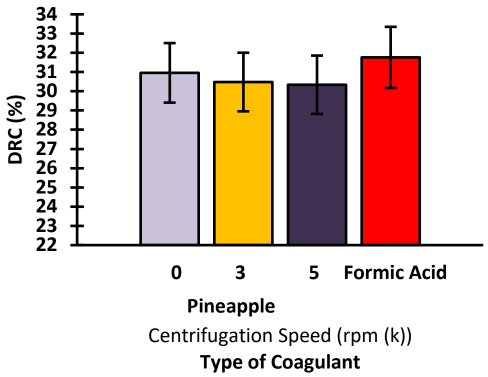


Figure 10 Effect of coagulant type on DRC

Centrifugation speed affects the DRC value, high centrifugation speed will produce a low DRC value. This is because high centrifugation speeds cause organic acids to precipitate, reducing the coagulation power of the coagulant and causing the rubber particles to coagulate less. The lower the rubber particle content, the lower the DRC [6]. Slightly coagulated rubber particles can be identified by the color of the coagulum serum produced, with formic acid producing a clearer serum color and pineapple a whiter serum color. The color of the serum from the coagulum natural pineapple coagulant can be seen in Figure 11. White serum indicates the presence of latex that is not fully coagulated, causing the latex content to be carried away by the serum coagulum and reducing the DRC value [11]. Based on the comparison of coagulants, formic acid has a higher DRC value.

Figure 11 Serum from the coagulum natural pineapple coagulant

3.10 Effect of Coagulant Type on Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy, or infrared spectroscopy, is an analytical method based on the principle of interaction of a chemical compound with electromagnetic radiation, which produces vibrations from a polyatomic chemical bond or functional group of a chemical compound (Cholifah, 2018) [24]. In this study, FTIR tests were carried out on rubber samples using natural pineapple coagulant with centrifugation treatment at 0 rpm, 3000 rpm, and 5000 rpm and formic acid chemical coagulant. The results of the FTIR test on rubber samples for pineapple coagulant are shown in Figure 12.

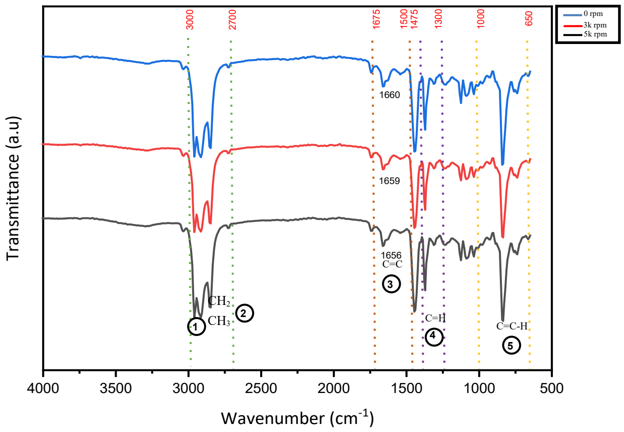


Figure 12 FTIR Spectra of Rubber Samples with Pineapple Coagulant

Figure 12 shows a graph of the FTIR test on pineapple at centrifugation speeds of 0 rpm, 3000 rpm, and 5000 rpm. Based on the above graph, wave peaks are obtained indicating the formation of polyisoprene rubber functional groups, which can be read from Table 1. For comparison, the molecular structure of natural rubber is also depicted in Figure 13.

Table 1 FTIR Signature of Pineaple [25].

Wave number (cm^{-1})		Type of Bond
(1,2)	3000-2700	Stretching $-CH_3$, $-CH_2$, C-H,
(3)	1675-1500	Stretching C=C
(4)	1475-1300	C-H *bending*
(5)	1000-650	C=C-H, Ar-H *bending*

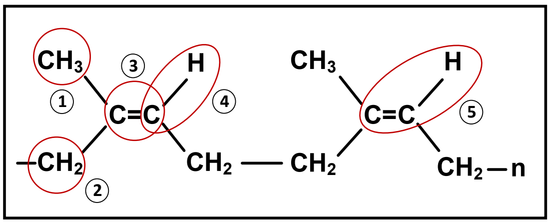


Figure 13 Molecular structure of natural rubber (cis-1,4-polyisoprene)

The pineapple coagulant FTIR test sample showed the peak of the CH_3 functional group, CH_2 was in the wave number range of 3000-2700 cm^{-1}, C=C was in the wave number range of 1675-1500 cm^{-1}, C-H was in the wave number range of 1475-1300 cm^{-1} and C=C-H are in the wave number range of 1000-650 cm^{-1}. This FTIR test ensures that natural rubber samples that have been coagulated using natural pineapple coagulant still have the same functional groups as pure natural rubber, so they do not change the structure of the original functional groups. This can be seen in Figure 12 which shows the results of the FTIR test of pineapple coagulant at speeds of 0 rpm, 3000 rpm and 5000 rpm producing the same functional groups as natural rubber functional groups, namely in Figure 13.

FTIR results at 3100-2700 cm^{-1} are characterized by the presence of CH_3, CH_2, and CH functional group bonds. This bond is a specific bond found in natural rubber compounds, namely isoprene monomers. Meanwhile, wave number 1675 - 1500 cm^{-1} is susceptible to the C=C functional group, which is the result of the stretching vibration of isoprene monomer to polyisoprene or natural rubber. The wave number in the range of 1675-1500 cm^{-1} is the stretching of the C=C functional group, which is also a specific bond found in natural rubber compounds, namely isoprene monomers [25] [26].

The C=C functional group, which has a wavenumber range of 1675 - 1500 cm^{-1}, undergoes a wavenumber shift. Where the value becomes larger or moves further to the left. For the C=C functional group, a centrifugation speed of 0 rpm shows that the wave number range of 1656 cm^{-1} shifts to a larger wave number of 1659 cm^{-1} at a centrifugation speed of 3000 rpm. Meanwhile, a centrifugation speed of 5000 rpm shows a wave number area of 1660 cm^{-1}. However, the wave number shift in this natural coagulant is still in the same functional group region and does not change the rubber functional groups. This is because all types of samples generally have the same constituents such as rubber particles, carbohydrates, proteins, phospholipids, and various other elements [27].

4.0 CONCLUSION

In the coagulation process, the pH and organic acid content are influenced by the high centrifugation speed, which will also affect the resulting rubber properties. The higher the centrifugation speed, the higher the pH of the natural coagulant, because the higher the centrifugation speed, the more organic acids in the natural coagulant are precipitated. Pineapple natural coagulant has characteristic advantages in that it has a higher value in the P_O and PRI tests and a lower volatile content than formic acid. However, pineapple coagulant has disadvantages in the tests for ash content and impurities, namely a higher value and a lower value for mooney viscosity than formic acid. From all characteristic test results, pineapple coagulant and formic acid fulfill SNI 06-1903-2017, except for pineapple coagulant in the impurity test without centrifugation treatment (0 rpm). Pineapple's natural coagulant has a pH of 4-5.5 and an acid content that can coagulate latex, rending it a suitable replacement for chemical coagulants.

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Conflicts of Interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper

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