The effect of additive dosages on crepe quality produced

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ABSTRACT

Thin Pale Crepe (TPC) products are currently increasing in demand by rubber product manufacturers. Its production process is relatively more efficient than crumb rubber, which is Indonesia’s main product. The drying of TPC products still uses air drying from the burning of rubberwood. The use of rubberwood is predicted to be less economical in the future. With an increasing limited natural forest, rubberwood will be an alternative substitute for wood from natural forests. As a result, the economic value of rubberwood will be even greater which will have an impact on the increasing cost of TPC products if it continues to use rubberwood as fuel. Therefore, we need an alternative to rubberwood as an energy source for drying TPC. Sunlight can be an alternative source of energy that can be maximized for the drying process of natural rubber, including in TPC production. This research was conducted in two stages, namely 1) the production of TPC using latex from various clones with several additive dosages, and 2) TPC drying using various renewable energy sources. The results of research activities are in the form of a technology package regarding the TPC production process using renewable energy sources. The results showed that the TPC products produced with various treatments fulfilled SNI 1903-2000. The three clones (BPM 24, PB 260, and GT 1) were able to produce TPC with bright yellow color.

Keywords: crepe, drying, natural rubber, renewable energy, TPC.

INTRODUCTION

There are many types of raw rubber-processed products, either processed directly from fresh latex or processed from latex coagulum (lump). Some raw rubber-processed products from latex include Thin Pale Crepe (TPC), SIR 3L, SIR 3, SIR 5, and Ribbed Smoke Sheet (RSS). Meanwhile, raw rubber processed products produced from coagulum are Brown Crepe (BRCR), SIR 10, and SIR 20. The main product which is produced by the natural rubber processing industry in Indonesia is SIR 20.

From several raw rubber products, TPC products are currently increasing in demand by rubber product manufacturers. This raw rubber product has a relatively more efficient production process than crumb rubber, which is Indonesia’s main product. In TPC processing, the main equipment is only a creeper machine. The drying process is only carried out at low temperature, around 35-40 °C, which means that energy required for the drying process is lower than crumb rubber which is generally dried at a temperature of 120-140 °C.

The drying of TPC products, as done at PTPN XI, still uses dry air from the burning of rubberwood. The use of rubberwood is predicted to be less economical in future. With increasingly limited natural forest, rubberwood will be an alternative substitute for wood from natural forests. As a result, economic value of rubberwood will be even greater which will have an impact on increasing cost of TPC products if it continues to use rubberwood as fuel. Therefore, there is a need for an alternative to rubberwood as an energy source for drying TPC.

Indonesia, which is located in tropical areas, has various other renewable energy sources, such as sunlight, biomass, and wind. The use of renewable energy sources, especially sunlight, is currently not being maximized. With technological developments, sunlight can be an alternative...
source of energy that can be maximized for the drying process of natural rubber. Natural rubber drying using sunlight is still not widely used by the natural rubber industry. This condition is because there has been no research related to the use of sunlight in drying natural rubber, especially on quality. This research will examine the TPC processing process which will later be dried using renewable energy sources, especially sunlight. This drying technology is expected to replace dependence on rubberwood fuel.

MATERIALS AND METHODS

Materials used in this study were fresh latex and lump from the research garden of Sembawa Research Center, formic acid, technical sodium metabisulfite, rubberwood, mineral turpentine, and pH paper. The equipment used were an analytical balance, measuring cup, Erlenmeyer, glass beaker, oven, stopwatch, rapid plastimeter, Mooney viscometer, coagulant tub, creeper machine, prototype drying device based on sunlight, biomass, and solar (Bioyuya). The stages of research activities were shown in Figure 1.

TPC production used latex from various additive doses, it covered 3 doses of additive (sodium metabisulfite). Treatment for dosages of additive consisted of 2.5% (w/w) of dried rubber, 5.0% (w/w) of dried rubber, and control (without adding additives). For clone type, it was mixed clones. For observation parameters in this study were clotting conditions (pH and clotting time), drying room temperature, drying time/duration, percentage of TPC product quality, and technical quality of TPC produced. For technical quality parameters to be analyzed consisted of initial plasticity ($P_0$), plasticity retention index (PRI), Mooney viscosity, and dirt content. In this study, secondary data were also be collected in the form of observational data on climatological conditions during the drying process. This climatological data was obtained from AWS equipment in the form of air temperature and humidity. The drying equipment for processing natural rubber latex into TPC can be seen in Figure 2.

RESULTS AND DISCUSSION

Initial Plasticity

The research activity of TPC production was focused on the dosage treatments of additive (bleaching agent). The additive dosage consisted of 2.5% (w/w), 5.0% (w/w), and 0% (w/w) or no additive as a control. The type of clone used was a mixed clone. From the results of the research that has been done, it is known that all types of clones are capable of producing TPC products with a minimum dose of additive 0.5% (w/w). The resulting TPC product was visually characterized by a bright yellow color. Apart from visual analysis, this research also analyzed the technical quality of TPC products. The technical quality data for TPC products were compared to SIR 20 rubber quality standards.

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Figure 1. Research activities phase.

Figure 2. Solar energy-based drying room (Bioyuya).
Table 1. Requirements for the quality of SNI 06-1903-2000.

| Specifications/(origin of processing materials) | SIR 20 Coagulum |
|------------------------------------------------|-----------------|
| Dirt content, % Max (w/w)                     | 0.20            |
| Ash content, % Max (w/w)                      | 0.50            |
| Content of the substance evaporates, % Max (w/w) | 0.80           |
| PRI, Min                                      | 40              |
| $P_0$, Min                                    | -               |

Source: BSN (2011).

standard based on SNI 06-1903-2000 (Table 1).

Initial plasticity ($P_0$) is a measure of rubber plasticity that indirectly estimates the length of rubber molecular polymer chain or molecular weight (BM) of rubber. $P_0$ value is one of the basic parameters for determining rubber quality (Achmadi et al., 2015). Based on Figure 3, it is known that the $P_0$ value of all TPC products meets the quality standard for SIR 20 product. $P_0$ value for SIR 20 (lowest quality) is at least 30 (Table 1).

Vachlepi and Suwardin (2015) stated that the use of ammonium salt, as a natural rubber additive, ranges from 32 to 35 %. High $P_0$ value of Low Viscosity (LoV) rubber using ammonium salt additive was seemed to occur due to formation of microgels, namely bonds in individual rubber particles (intra particle crosslink). From the microgel, a further cross-linking will occur which is called a macrogel. $P_0$ value of natural rubber is partly influenced by formation of microgels and macrogels (Solichin & Setiadi, 1992). This condition is different from HNS additive where natural rubber produced has a low $P_0$ value. Meanwhile, Solichin and Setiadi (1992) reported that adding Hydroxylamine Neutral Sulfate (HNS) additive to natural rubber can reduce $P_0$ value when compared to those without HNS. Meanwhile, $P_0$ quality parameter is not quality requirements listed in SNI No.1903-2011 for LoV rubber. However, $P_0$ value which is generally preferred by consumers, in this case tire factory is generally more than 30. This is in accordance with research of Solichin (1995) where $P_0$ value of natural rubber tends to decrease during processing. This is thought to occur because of water content in 10% acetaldehyde solution. Presence of water in the stabilizer could cause growth of microorganisms which destroy proteins in natural rubber molecules. Growth of microorganisms that destroy these proteins can decrease $P_0$ values. Vachlepi and Purbaya (2019) stated that addition of maleic anhydride to solid phase will increase $P_0$ value of natural rubber around 26-28. Results of this $P_0$ value analysis illustrated that natural rubber polymer modified with maleic anhydride additives has a longer molecular chain than control natural rubber. During mastication process by heating, natural rubber polymer chains are terminated and form free radicals. Pulungan et al. (2017) stated that free radicals are one or more unpaired electrons which were added with additive mixture of hydrazine hydrate-ammonium sulfate with various doses were generally higher than the control. $P_0$ value of natural rubber using a mixture of hydrazine hydrate and ammonium sulfate additives was around 35.5-43.0. This figure indicates that additive mixture of hydrazine hydrate and ammonium sulfate with various dosages can produce natural rubber with high $P_0$. This ability is more due to the presence of ammonium sulfate which does not decrease the $P_0$ value. These results are in accordance with research done by Vachlepi and Suwardin (2015) which proved that use of ammonium salts, one of which is ammonium sulfate, does not reduce the $P_0$ value of natural rubber. An ammonium sulfate could cover impact of using hydrazine, which has been shown to reduce $P_0$ values.

According to Vachlepi (2019) soaking natural rubber with acetaldehyde for 30 minutes can increase $P_0$ value. This condition is in accordance with research of Solichin (1995) where $P_0$ value of natural rubber tends to decrease during processing. This is thought to occur because of water content in 10% acetaldehyde solution. Presence of water in the stabilizer could cause growth of microorganisms which destroy proteins in natural rubber molecules. Growth of microorganisms that destroy these proteins can decrease $P_0$ values (Solichin & Anwar, 2003). Vachlepi and Purbaya (2019) stated that addition of maleic anhydride to solid phase will increase $P_0$ value of natural rubber around 26-28. Results of this $P_0$ value analysis illustrated that natural rubber polymer modified with maleic anhydride additives has a longer molecular chain than control natural rubber. During mastication process by heating, natural rubber polymer chains are terminated and form free radicals. Pulungan et al. (2017) stated that free radicals are one or more unpaired electrons.
that trigger copolymerization grafts. Addition of maleic anhydride additive would react (graft) with poly isoprene group to form a new functional group.

**Plasticity Retention Index**

Plasticity Retention Index (PRI) is a measure of rubber resistance to wear (oxidation) at high temperatures. PRI value is measured by amount of rubber raw rubber that remains when sample is heated for about 30 minutes at 140 °C. PRI value is percentage of rubber plasticity after heating compared to plastic’s plasticity before heating and is determined by means of a Wallace plastimeter. PRI value is one of basic parameters for determining rubber quality (Achmadi et al., 2015). For PRI value of mixed clones, all TPC products meet the quality requirements of SIR 20 (Figure 4) where the minimum requirement is a PRI value of 50 (Table 1). The higher the PRI value, the better the rubber quality (Montha et al., 2016).

Vachlepi (2018) reported that usage of ammonium salt additives has no effect on PRI value. The lowest PRI value (80) was produced by LoV rubber using additives in form of ammonium chloride and the highest value (89) from rubber without addition of additives. In SNI No.1903-2011 concerning SIR, the quality parameter in form of PRI is also not a requirement for type of LoV rubber. PRI value of LoV rubber with added ammonium salt additive is around 80-86. Main factor affecting PRI value is consideration between prooxidants and antioxidants in rubber (Solichin, 1991). According to Vachlepi and Purbaya (2019) usage of maleic anhydride additives in natural rubber could possibly reduce PRI value, which ranges from 53.57 to 57.69. This figure shows that natural rubber which has been modified with maleic anhydride is not resistant to oxidation process at high temperatures. For low resistance of modified natural rubber occurs due to presence of a new functional group that is not resistant to oxidation.

Vachlepi (2019) reported that PRI value of natural rubber soaked in acetaldehyde ranges from 71.6 to 82.2 which was resistant to high temperature (Solichin, 1991) and is resistant to ozonolysis (oxides by ozone) (Aguele et al., 2015). Another study was also reported by Vachlepi (2018) that usage of a mixture of hydrazine hydrate and ammonium sulfate additives did not damage quality of natural rubber PRI, ranging from 70.2-86.1. It should be noted that in general PRI value of rubber is affected by storage time. Environmental conditions (temperature, pH, and oxygen in the air) during storage affect sensibility of natural rubber to high temperature oxidation. This factor also occurs due to changes in balance between antioxidant compounds (protein, amino acids, tocotrienols) and pro-oxidants (unsaturated free fatty acids and free metal ions) in natural rubber (Intapun et al., 2009).

**Mooney Viscosity**

Mooney viscosity parameter describes chain length of rubber molecules. This quality parameter plays an important role in mixing process when making compound, both for level of dispersion of chemicals in rubber and energy required for
Crepe quality with different dosage of additive...........(Hanifarianty et al.)

Mooney viscosity is usually used as a technological indicator to determine characterization of rubber particles in terms of their ability during further processing, including when making compound (Zheleva, 2013).

Mooney viscosity value of all TPC products was high, ranging from 77-83 (Figure 5). This figure shows that during drying process using hot air in a sun-based drying chamber cross-linking has occurred between aldehyde groups on natural rubber particles. This could also be seen from high P0 and PRI values. This hanging drying technology is also used in crumb rubber (SIR) factories to improve quality of natural rubber, especially P0, PRI, and Mooney viscosity values. Based on research of Vachlepi (2018), Mooney viscosity value and low viscosity rubber (LoV) with various treatments using ammonium salt in manufacture of LoV rubber did not significantly affect Mooney viscosity value and stability viscosity index (SVI), which ranged from 64-69. Mooney viscosity parameter describes chain length of rubber molecule. The higher Mooney viscosity value could possibly create longer chain of natural rubber molecules. Rubber viscosity plays an important role in mixing process when making compound, both for level of dispersion of compound chemicals in rubber and power required for grinding in mixing machine. Viscosity that is too high causes high power consumption of processing engines. Conversely, if viscosity is very low, it causes a low shear force in mixing which results in material tending to agglomerate, so homogeneity is low (Maspanger, 2008). Mooney viscosity is usually used as a technological indicator to determine characterization of rubber particles in terms of their ability during further processing, including when making compound (Zheleva, 2013).

During storage, viscosity of rubber increases spontaneously and irreversibly so that rubber becomes tougher. This hardening phenomenon is accelerated by rubber storage under dry conditions and high temperatures (Subramaniam, 1984). Solichin (1991) stated that storage time affects Mooney viscosity of natural rubber. Different conditions occur in 20 CV SIR rubber which uses a hydrazine as a stabilizer at a dose of 0.4% (w/w). If at the beginning of process (0 days) viscosity is quite high, which is around 71. But during storage process viscosity of rubber has decreased to 64 and meets quality requirements of SIR 20CV according to SNI 1903-2011. This phenomenon occurs because reaction of double bond saturation through hydrogenation is carried out by hydrazine is already happening. Hydrazine compound functions as a hydrogen group donor that can reduce double bond of natural rubber molecules to single (saturated) bonds (Rahman et al., 2002). Decreasing in viscosity value indicates that hydrazine at a dose of 0.4% (w/w) is able to prevent cross-linking between rubber particles. Double bonds found in natural rubber have reacted with hydrazine to form a single bond.

Decreasing in Mooney viscosity value also occurred due to reduced formation of microgels and macrogels (Suparto & Alfa, 1998). Mooney viscosity is usually used as a technological indicator to determine characterization of rubber particles in terms of their ability during further processing, including when making compound (Zheleva, 2013). In contrast to viscosity, SVI parameter describes change in viscosity value of natural rubber mooney during storage before natural rubber is further processed into rubber finished goods. This SVI value shows how stable
The viscosity of natural rubber is during storage. Mooney viscosity value of natural rubber was more influenced by type of coagulant treatment. Mooney viscosity of natural rubber with acid group coagulation material (formic acid and sulfuric acid) is around 81-91. High Mooney viscosity of natural rubber which is coagulated with acid group coagulants is due to cross-linking between natural rubber molecules that occurs naturally and is characterized by an increase in viscosity value of Mooney (Solichin, 1995). This phenomenon proves that acid group coagulants, both formic acid and sulfuric acid, are unable to prevent cross-linking reactions in natural rubber. Natural rubber which is coagulated with liquid smoke has a lower Mooney viscosity (75-76) than formic acid and sulfuric acid. Low Mooney viscosity value indicates that liquid smoke is able to prevent natural rubber from hardening during processing process so that natural rubber remains soft due to chemical compounds present in liquid smoke. Solichin et al. (2005) stated that liquid smoke contains about 67 types of compounds that can function as anti-bacterial (Karseno et al., 2001), antioxidants, giving chocolate color, and a distinctive smoke odor. Vachlepi (2019) stated that soaking natural rubber with acetaldehyde does not significantly affect SVI of natural rubber. Results of this analysis indicate that application of acetaldehyde using soaking method has not been effective in preventing cross-linking of natural rubber particles, which are characterized by a high SVI value.

**Ash Content**

Ash content is one of quality parameters required in SNI 06-1903-2000 concerning SIR. This parameter is analyzed to determine effect of hanging wet crepe on potential for adhering contaminants and is calculated as ash content of TPC product. As shown in Figure 6, it is known that all the ash content of TPC products meet the maximum requirements of SNI 06-1903-2000. The ash content of TPC products ranged from 0.18 to 0.27%. Natural rubber product SIR 20 must have a maximum ash content of 1%. These results prove that drying in drying chamber based on solar energy is clean and free from burning ash. Results of the analysis of LoV rubber ash content using various additives are shown in Figure 6. Usage of additives affects resulting LoV ash content. LoV rubber ash content with additives in form of ammonium sulfate, ammonium chloride, ammonium dihydrogen phosphate and HNS (control additive) meets export quality requirements based on SNI No.1903-2011. Quality requirements for ash content parameters listed in SNI as LoV rubber are a maximum of 0.5%. Low viscosity rubber ash content with ammonium sulfate, ammonium chloride, ammonium dihydrogen phosphate and HNS as stabilizer were 0.50%, 0.49%, 0.49%, and 0.48%, respectively. Natural rubber that uses ammonium acetate and ammonium nitrate additives does not have same export quality requirements as rubber without additives (blank). This could be performed where the graph of the ash content of ammonium acetate (0.98%), ammonium nitrate (0.90%), and blank (0.53%) is above maximum quality requirements for LoV rubber. Thus, it means that additives in form of ammonium acetate and ammonium nitrate cannot

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**Figure 5.** Mooney viscosity value of TPC products with various dosages of whitening ingredients mixed with mixed clone.

**Figure 6.** The value of the ash content of TPC products with various dosages of bleaching agents with mixed clones.
Crepe quality with different dosage of additive...........(Hanifarianty et al.)

be used to produce LoV rubber. High ash content in raw rubber will affect dynamic properties of nature, such as heat buildup and flex cracking resistance (Ministry of Trade and Cooperatives, 1981). Therefore, quality natural rubber must have low ash content according to SNI 1903:2011. Vachlepi (2018) stated that additives used in the form of hydrazine hydrate and ammonium sulfate are not included in inorganic compound that could increase ash in natural rubber. Results of analysis showed that the highest ash content obtained by A1B1 treatment was 0.50%. Meanwhile, the lowest ash content was found in natural rubber A2B2 treatment of 0.30%. For ash content of all treatments came from natural rubber latex. George and Jacob (2000) stated that total concentration of inorganic compounds or ions in fresh latex is about 0.50%. Results of this analysis indicate that natural rubber produced using a mixture of hydrazine hydrate and ammonium sulfate additives meets quality requirements of SNI 06-1903-2000 as SIR 20CV natural rubber where ash content of natural rubber produced is not more than 1%.

In another study, Vachlepi (2019) also stated that ash content of natural rubber produced was not significantly affected by soaking treatment in acetaldehyde. This is because acetaldehyde is not included in inorganic compounds which will be analyzed as ash in natural rubber. Meanwhile, natural rubber which is coagulated with sulfuric acid coagulant tends to have a higher ash content than formic acid coagulant and liquid smoke. For ash content of natural rubber coagulated with sulfuric acid ranges from 0.54-0.58%. Meanwhile, formic acid and liquid smoke coagulants are included in the group of organic compounds which are not counted as ash content so that they only have an ash content of around 0.21-0.42%. Vachlepi and Purbaya (2019) reported that usage of maleic anhydride additives had no effect on ash content of natural rubber produced, which was around 0.51-0.72%, while natural rubber controlled ash content of 0.60%.

### Volatile Substances

Volatile substances in rubber are mostly water vapor and rest are other substances such as serum which is volatile at 100 °C. Presence of volatile substances in rubber, apart from causing a foul odor, also facilitates growth of mold which can cause difficulties when mixing chemicals into rubber when making compound, especially for mixing carbon black at low temperatures (BSN, 2000). In SNI 06-1903-2000, one of quality requirements that must be considered so that natural rubber could be exported to various destination countries is content of volatile substance. For maximum requirement of volatile content in natural rubber is 0.80% (w/w). For value of volatile substance content of all TPC products produced was quite good, which was in range of 0.53-0.67. Based on Figure 7, it is known that value of volatile substance content of all TPC products meets quality standard for type of SIR 20 product.

Vachlepi (2018) stated that levels of natural rubber volatile substances are generally not affected by usage of low additives, namely a mixture of hydrazine hydrate-ammonium sulfate and HNS, which ranges from 0.05% -0.20%. Volatile substances in rubber are mostly water vapor and rest are other substances such as serum which is volatile at 100 °C. In another study, Vachlepi and Purbaya (2019) reported that use of maleic anhydride additives could reduce levels of natural rubber volatile substances produced. Content of natural rubber volatile agent tends to get lower along with higher dose of maleic anhydride additive. Natural rubber that has been modified with maleic anhydride has a volatile substance content of about 0.23-0.47%. This figure is still lower than control natural rubber (without the addition of additives) which has a volatile substance content of 0.75%. This phenomenon occurs more due to change in the functional group of natural rubber polymer which was previously non-polar, now it has changed to a polar which is easier to bind water and even dissolves in water.
CONCLUSIONS

The conclusion of this study is the use of sodium metabisulfite additive consisting of 2.5% (w/w) and 5.0% (w/w) produces TPC that fulfill requirement of SNI 1903-2000. For Po and PRI values were 53.5-54.0 and 76.0-78.0, respectively. Meanwhile, Mooney viscosity value is 77-78. And then, for ash content is 0.26-0.27, while content of volatile substance is 0.63-0.65. For mixed clone could possibly produce TPC product with a bright yellow color. Further research is needed a variety of clones to obtain clones that are more specific for TPC production.

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