

Oleoresin Yield and Gum Rosin Properties from Sulawesi and Sumedang Sub-lines of *Pinus merkusii* Wood

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Abstract

Progeny testing can be performed to reach high oleoresin production from *Pinus merkusii*. This study aimed to determine and compare the oleoresin yields and rosin properties of 15-year-old *Pinus merkusii* wood of the Sumedang and Sulawesi sub-lines from the KPH Banyumas Barat plantation. Tree diameter and oleoresin yield were observed in 20 individual trees of each sub-line. Tree tappings were conducted by the drilling method for three days, which was replicated for five times over a 15-day observation period. Gum rosin samples were obtained from all trees by the hydro-distillation method. The results showed that the tree diameters at breast height and oleoresin yields of the Sumedang sub-line fell in the ranges 22.0–34.2 cm and 10.8–51.2 g/tree/3 days, respectively, whereas those of the Sulawesi sub-line fell within the ranges 19.4–31.2 cm and 11.6–50.4 g/tree/3 days, respectively. Based on t-test results, no significant difference was found between the two sub-lines. The gum rosin yields were 52–79% and 55–78% for the Sumedang and Sulawesi sub-lines, respectively. With regard to rosin properties, the softening point, acid number, saponification value, toluene-insoluble content, and volatile content were $77.80 \pm 4.08^\circ\text{C}$, 189.51 ± 10.51 , 216.54 ± 10.51 , $0.08 \pm 0.11\%$, and $3.78 \pm 3.56\%$, respectively, for the Sumedang sub-line and $66.35 \pm 6.55^\circ\text{C}$, 78.15 ± 2.92 , 189.86 ± 10.68 , 208.02 ± 10.50 , $0.06 \pm 0.11\%$, and $9.82 \pm 2.95\%$, respectively, for the Sulawesi sub-line. Significant effects of sub-line were only observed in terms of saponification value and volatile content, where the rosin from the Sumedang sub-line had better properties than the rosin from the Sulawesi sub-line. These differences indicate different compositions of chemical components of oleoresin or rosin between sub-lines.

Keywords: pine resin, gum rosin, resin distillation, yield, tree diameter

Introduction

Rosin and turpentine oils from the distillation of *Pinus merkusii* Jungh et de Vriese oleoresin are the major commodities of Perum Perhutani in its non-timber forest products (NTFPs) division. For this reason, oleoresin tapping serves as a highly important supply-chain component in supporting the sustainability of its processing industry. Perum Perhutani has made various innovations, including the selection of superior trees, improvement of tapping methods, and improvement of the efficiency of the distribution chain to the mill.

One way of selecting superior trees is by conducting *P. merkusii* progeny tests for oleoresin production, for example one which has been done in 2007 at the West Banyumas KPH plantation. In addition to producing trees with good growth characteristics (relatively large diameters and heights), relatively straight trunks, and resistance to attacks by pests and diseases, the progeny test is also designed for the main purpose of producing high oleoresin yield with a sub-line system based on the origin of the parent tree (Muslimin 2017).

Oleoresin production in several pines is affected by genetic factor (Tadesse *et al.* 2001; Siregar and Hatemer 2004; López-Álvarez 2023) as well as environmental factors (Gajšek *et al.* 2018; Sharma *et al.* 2018; Lukmandaru *et al.* 2020; 2021). The genetic effect on the oleoresin productivity of several sub-lines of *Pinus merkusii* progeny test was

observed by Muslimin (2013) and Nugrahanto *et al.* (2020), who proved the significant effect of the sub-lines. Research on oleoresin production and turpentine and rosin properties showed a significant effect of provenance differences (Sukarno *et al.* 2015; 2018). Genetic differences in pine trees, in addition, are thought to affect the quality of the rosin produced because it can cause variation in the chemical components contained in the rosin.

The first *P. merkusii* introduced to Java was narrow in its genetic base. Therefore, the genetic base of the species was expanded by introducing new genetic materials from varied sources. Sub-lines are genetic materials produced through a selection process that requires a number of tests before they can be released as varieties. This study used the Sumedang and Sulawesi sub-lines of *Pinus merkusii* whose seedlings were taken from the Sumedang and Sulawesi seedling seed orchards (SSO). This study is expected to provide more information with regard to rosin properties from tree breeding programs.

Materials and method

Sampling Site

Sampling was carried out in compartment 37C, Sumedang 2 Block IV for the Sumedang sub-line and in Sulawesi 2 Block VIII for the Sulawesi sub-line in October–December 2022. It took place in RPH Samudra, BKPH

Lumbir, KPH Banyumas Barat (07°20'00" S and 108°55'00" E, 300–500 m above sea level). The average daily temperature was 26.3°C, with a minimum temperature of 24.4°C and a maximum temperature of 30.9°C. With an average rainfall of 3500 mm/year, the site was classified as having B climate according to the Schmidt-Fergusson climate classification. The site was of a hilly topographic condition with a slope of 30–40°, and the soil was of the mediterranean soil (Alfisol) type from limestone with marl parent material (Muslimin et al. 2013).

Oleoresin Tapping

A total of 20 selected healthy and straight trees for each sub-line were tapped using the drilling method. This tapping method was selected in order to produce cleaner oleoresin from the dirt. The selection of trees was based on the

uniform conditions for obtaining sunlight and nutrients. Oleoresin was harvested using the trunk drilling method at a height of ±50 cm above the ground, with a slope of 30–45° toward the trunk axis upward. The drilling depth was ±2–3 cm (10 mm of drill bit) from the outermost wood surface to the sapwood part. Pipes and plastic bags for collecting sap were installed to hold clear oleoresin of premium quality (Figure 1). After three days, the oleoresin collected was taken. Drilling was then carried out again in a different direction, and the pipes and plastic bags were reinstalled. All the oleoresin collected was taken and weighed using a digital scale. Drilling was performed with a total of five replications (over 15 days), and the average weight of oleoresin was calculated. The accumulated production was also calculated for a total of 15 days of observation. The oleoresin was held in a plastic bag, which was then stored at room temperature for six months.



Figure 1. Oleoresin tapping by drilling method

Oleoresin Distillation

The weighed oleoresin was extracted for rosin through a hydro-distillation process (Figure 2). Due to the oleoresin's premium quality, the cooking process was carried out directly by putting the oleoresin (±100 g) into a two-necked flask, and then distilled water was added. The weight ratio of oleoresin to distilled water was 1:1.5. Heating was carried out at a temperature of 140°C for 1.5 hours. Once finished, the liquid-state rosin produced was poured into a cast and then maintained until it hardened (Figure 3). The gum rosin yield was calculated from the weight of rosin based on the initial oleoresin weight.



Figure 2. Hydro-distillation of *Pinus merkusii* oleoresin



Figure 3. Rosin obtained by hydro-distillation method

Physico-chemical Properties of Gum Rosin

The softening point (ring and ball apparatus), acid number (acid-base titration), as well as solubility in toluene and ash content (gravimetry) were measured and compared according to the SNI 7636:2020 standard. These measurements were carried out with three replications

Data Analysis

The obtained data was assayed for normality (Shapiro-Wilk test). The effects of sub-line on tree diameter, resin production, and physico-chemical properties of rosin were analyzed using an independent t-test (normal data distribution) and a Mann-Whitney test (non-normal data distribution). Statistically significant differences were set at a 95% confidence level. All statistical calculations were conducted using SPSS-Win 18.0.

Results and Discussion

Oleoresin Yield

Pine tree tapping was conducted by the drilling method to produce premium-quality oleoresin with maximum yield. In this method, oleoresin was flowed from a drill hole through a pipe that was inserted into the hole and immediately collected into a closed plastic bag to prevent direct contact with dirt and

water during tapping. Oleoresin collection was done with five replications once every three days. The results of measurements of tree diameter, three days' average oleoresin yield, and 15 days' accumulated oleoresin yield are presented in Table 1.

The average oleoresin yield of the Sumedang sub-line was higher (30.69 g/tree/3 days) than that of the Sulawesi sub-line (26.97 g/tree/3 days). Previous research obtained yield values of 4.58 ± 0.27 g/tree/3 days and 3.50 ± 0.18 g/tree/3 days for the Sumedang and Sulawesi sub-lines of 4-year-old *Pinus merkusii*, respectively (Muslimin 2013). The oleoresin yield from *Pinus merkusii* was moderate to high under genetic control (Soekarno *et al.* 2015; Nugrahanto *et al.* 2022). Another study was conducted on three sub-lines of *Pinus merkusii* progeny test (11 years) in the KPH Banyumas Barat plantation (Nugrahanto *et al.* 2020). It was found that the SSO of the Sumedang sub-line gave the highest results with an average oleoresin yield of 17.43 ± 0.38 g/tree/3 days, while the East Java sub-line had an average oleoresin yield of 12.53 ± 0.34 g/tree/3 days. The difference in value is thought to be due to the age of the tree at the time of tapping. Tree age is a factor that greatly affects pine oleoresin yield (Lateka *et al.* 2019), where the older the tree, the more pine oleoresin it produces.

The accumulated results from 15 days of observation showed average yield values of 153.45 g and 134.85 g for the Sumedang and Sulawesi sub-lines, respectively. These values are equivalent to oleoresin yields of 10.23 g/day and 8.99 g/day, respectively. As a comparison, an earlier work using the drilling method on 29-year-old *Pinus merkusii* stands (class age VI) in Tana Toraja obtained a value of 6.4 g/tree/day without stimulant treatment (Lempang 2017). From different provenances of *Pinus merkusii* in a Jember plantation, Soekarno *et al.* (2015) observed average oleoresin yields of 12.2–21.1 g/hole/tree/day. The average daily values were obtained at 4.15 g per tree (class age IV) and 9.10 g per tree (class age VII) per day in the pine stands of KPH Banyumas Timur using the bark chipping method for eight days of observation without wound renewal (Lukmandaru *et al.* 2018).

Table 1. Tree diameter and oleoresin yield from Sumedang and Sulawesi sub-lines

Trees number	Diameter (cm)		Oleoresin yield of tree/3 days (g)		Oleoresin yield of tree/15 days (g)	
	Sumedang	Sulawesi	Sumedang	Sulawesi	Sumedang	Sulawesi
1	34.2	30.6	33.8	31.2	169	156
2	31.8	26.9	36.8	31.8	184	159
3	29.8	26.4	23.2	20.6	116	103
4	25.9	26.1	34.8	22.2	174	111
5	29.3	24.9	27.0	14.8	135	74
6	23.6	20.5	30.0	11.6	150	58
7	28.2	28.9	22.4	26.6	112	133
8	24.3	20.8	40.8	15.2	204	76
9	26.4	27.0	29.8	19.8	149	99

10	23.3	32.7	20.0	20.8	100	104
11	25.0	28.5	34.2	19.8	171	99
12	24.2	24.0	10.8	41.8	54	209
13	22.0	22.1	28.8	39.0	144	195
14	30.5	21.9	32.2	34.4	161	172
15	31.5	24.6	28.4	39.2	142	196
16	32.2	29.4	37.4	44.8	187	224
17	27.7	19.4	27.0	11.6	135	58
18	29.0	26.5	40.8	26.0	204	130
19	25.8	31.2	51.2	17.8	256	89
20	29.6	29.6	24.4	50.4	122	252
Average	27.72	26.10	30.69	26.97	153.45	134.85
St. Dev	3.42	3.80	8.77	11.53	43.83	57.65
Coeff. var. (%)	12.34	14.56	28.56	42.75	28.56	42.75
t-test (prob)	0.16		0.25		0.25	

Yield and Properties of Gum Rosin

The average yield of rosin for the Sumedang sub-line was 64.98%, while for the Sulawesi sub-line the average yield of rosin was 66.24%. Through different distillation processes on a laboratory scale, the rosin yields from *Pinus merkusi* grown in West Sumatra in previous studies ranged from 58% to 63% (Wiyono *et al.* 2003). It was also mentioned that the yield on a factory scale ranged from 60% to 69%. For comparison, the rosin yields of *P. oocarpa* and *P. patula* were 75.0% and 74.5%, respectively (Sarria-Villa *et al.* 2021).

The acid numbers of rosin of the Sumedang and Sulawesi sub-lines ranged from 168.3 to 203.36 and from 161.29 to 199.86, respectively. The average acid numbers for the Sumedang and Sulawesi sub-lines were 189.51 and 189.86, respectively. These ranges were still within the SNI requirements (160–200) and comparable to the acid numbers of trees from West Sumatra (Wiyono *et al.* 2003) but higher than acid numbers of trees of the Aceh provenance and Jember landrace (Soekarno *et al.* 2018). The saponification values of rosin were 216.55 for the Sumedang sub-line and 208.03 for the Sulawesi sub-line. Based on the average values, the saponification values obtained were still in accordance with the SNI standards but lower than those observed by Wiyono *et al.* (2003) and higher than those observed by Soekarno *et al.* (2018).

The comparatively high acid numbers in this study indicate that the concentrations of free resin acid in the rosin were quite large. This is thought to be because the oleoresin in this study had been degraded by the breakdown of unsaturated resin acid through the oxidation or hydrolysis process due to the storage time being too long, namely six months, hence generating high acid numbers. In addition, this study did not experiment with oleoresin dilution with turpentine, causing the free resin acid to increase. The quality of rosin produced for food grade purposes will decrease if the acid number is higher (Hidayat *et al.* 2021).

The toluene-insoluble content in rosin was 0.08% and 0.06% for the Sumedang and Sulawesi sub-lines, respectively, falling within the range of 0–0.4%. These values

met the SNI 7 636:2020 standard as they remained below the 0.1% threshold. The rosin from the Sumedang sub-line was of the third quality class (N), while the rosin of the Sulawesi sub-line was of the second quality class (WG), suggesting that the rosin from the Sumedang sub-line had a higher level of impurities than the rosin from the Sulawesi sub-line.

The permissible value of softening point of rosin based on SNI 7636:2020 is $\geq 74^{\circ}\text{C}$. Wiyono *et al.* (2003) observed values of 76–80 $^{\circ}\text{C}$, and almost uniform values (76.0–76.5 $^{\circ}\text{C}$) were obtained by Soekarno *et al.* (2018) in previous works. The softening points of rosin of the Sumedang and Sulawesi sub-lines fell within the ranges 69–83 $^{\circ}\text{C}$ and 72–84 $^{\circ}\text{C}$, respectively. Thus, the rosin from the Sumedang sub-line could be classified in the second quality class with an average softening point of 77.8 $^{\circ}\text{C}$, while the rosin of the Sulawesi sub-line was in the super quality class with an average softening point of 78.15 $^{\circ}\text{C}$. Based on these results, it can be concluded that the rosin from the Sulawesi sub-line had lower dirt/impurity content than the rosin of the Sumedang sub-line. The dirt content in rosin usually takes the form of turpentine or short-chain fatty acid organic compounds.

The volatile content of rosin from the Sumedang sub-line ranged from 0.4% to 10.4%, while the rosin from the Sulawesi sub-line had volatile content from 3.6% to 15.2%. Based on the volatile content, several rosin samples from the Sumedang sub-line belonged to the quality classes X to N (Table 2), while all rosin samples from the Sulawesi sub-line failed to meet SNI standards. An imperfect cooking process can prevent turpentines in oleoresin from evaporating completely, hence resulting in a decrease in softening point (Hidayat *et al.* 2021). Values in the range 2.1–2.3% were previously obtained by Soekarno *et al.* (2018). Meanwhile, the rosin obtained from the Sindang Wangi mill in West Java through the steam distillation process had softening points of 90 $^{\circ}\text{C}$ for WW quality rosin and 88 $^{\circ}\text{C}$ for X quality rosin (Khadafi *et al.* 2014).

Compared to the steam distillation process, the research direct contact (boiling) applied in this experiment is thought to cause a low softening point and a high level of evaporated parts due to the presence of turpentine. At

atmospheric pressure, turpentine has a boiling point of around 180°C and an operating time 3–4 times longer (Abdulgani 2002). However, the application of a high temperature will reduce the yield of rosin due to the quality degradation of the resin compounds.

Sub-line Effect

In an earlier study, a significant genetic control on the secondary plant chemistry of *Pinus contorta* var. *latifolia* was observed (Ott et al. 2011). Based on independent t-test results, sub-line factor was found to have no significant effect on both tree diameter and oleoresin yield. It is thought that those properties are weakly genetically controlled. This indicates a strong environmental influence, with the two blocks observed here sharing similar hilly topographic conditions (slope of 30–40°). In addition, the trees of both sub-lines were the same in terms of age (15 years), treatment (no thinning), and tapping method. This trend was not observed by Muslimin (2013), who investigated five different sub-lines, including the Sulawesi and Sumedang sub-lines, and Nugrahanto et al. (2020), who also studied five different sub-lines.

Based on Mann-Whitney test and independent t-test results (Table 2), the difference in sub-lines only gave significant effects on saponification value and volatile content. It is assumed that the similar moisture and impurity content in both sub-lines would give similar yield and softening point. Saponification value is often associated with acid number. The high saponification value of the Sumedang sub-line indicates the presence of a higher proportion of short-chain carbon of resin acids with a low molecular weight in this sub-line than in the Sulawesi sub-line. The higher the saponification value, the lower the fatty acid content and the better the quality of the rosin. Conversely, the lower the saponification value, the higher the fatty acid content and the lower the quality of the rosin (Wijayanti et al. 2012). The rosin from the Sumedang sub-line had significantly lower volatile content than the rosin from the Sulawesi sub-line. It might be due to the lower monoterpene portions in the rosin of the Sumedang sub-line. The volatile part from monoterpene group such as pinene, carene, and camphene have been detected in oleoresin of *Pinus merkusii* (Sukarno et al. 2015). Further investigation with spectroscopic work is needed to find out more about the chemical composition of rosin.

Table 2. Gum rosin properties from Sumedang and Sulawesi Sub-line

Rosin properties	Sub-line Sumedang (n = 20)				Sub-line Sulawesi (n = 20)				t-test/ Mann-Whitney (prob.)	Indonesia National Standard (SNI 7636:2020)
	Min	Max	Average (Sd)	CV (%)	Min	Max	Average (Sd)	CV (%)		
Yield (%)	52	79	65.05 (7.48)	11.4	55	78	66.35 (6.55)	9.87	0.56	-
Softening point (C°)	69	83	77.80 (4.08)	5.2	72	84	78.15 (2.92)	3.73	0.52	XB: ≥78; X: ≥78; WW: ≥78; WG: ≥76; N: ≥74
Acid number	168.30	203.36	189.51(10.51)	5.5	161.29	199.86	189.86(10.68)	5.6	0.91	160-200
Saponification number	184.43	230.01	216.54(10.51)	4.8	185.83	224.40	208.02(10.50)	5.0	0.01*	170-220
Solubility in toluene (%)	0	0.40	0.08 (0.11)	137.5	0	0.40	0.06 (0.11)	183.3	0.62	XB: ≤0,02; X: ≤0,02; WW: ≤0,05; WG: ≤0,07; N: ≤0,1
Volatile content (%)	0.40	10.40	3.78 (3.56)	94.1	3.6	15.2	9.82 (2.95)	30.0	0.01>*	XB: ≤2; X: ≤2; WW: ≤2, WG: ≤2,5; N: ≤3

Remark: * significant at 95% confidence level in Mann-Whitney test. CV = coefficient of variation, Sd = standard of deviation, min = minimum, max = maximum

Conclusions

There were no significant differences in tree diameter and oleoresin yield between the Sulawesi and Sumedang sub-lines. With regard to rosin properties, significant differences were observed in saponification value and volatile content, where the Sumedang sub-line provided better quality. Although the oleoresin studied here was of premium quality, some test samples did not meet SNI standards. The use of the hydro-distillation method in this experiment might have caused a decrease in softening point and high volatile content of rosin.

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