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Full Length Research Article

Enhancing Pine Resin Productivity through Sulfuric Acid–Modified ETRAT Stimulants under Operational Conditions in *Pinus merkusii* Stands

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ABSTRACT

Pine resin is an important non-timber forest product in Indonesia; however, the productivity of *Pinus merkusii* tapping remains relatively low and has not yet met industrial demand. This study evaluated the effect of a sulfuric acid–modified ETRAT stimulant on pine resin productivity under operational tapping conditions in Perum Perhutani forests in Central Java, Indonesia. Resin tapping was conducted on 160 trees distributed across four age classes (AC III–VI) using a randomized block design with four stimulant treatments: ETRAT, ETRAT + 5% H_2SO_4, ETRAT + 10% H_2SO_4, and a control without stimulant. Resin yield was monitored over 10 consecutive tapping periods, with intervals of 3 days. Resin productivity exhibited temporal variation across tapping periods, with generally lower yields during the initial tapping periods, increasing toward the intermediate periods, and declining slightly thereafter; however, differences among tapping periods were not statistically significant. Stimulant treatment significantly affected resin productivity. The highest productivity was achieved with ETRAT + 5% H_2SO_4 (11.29 g/quarre/day), representing an approximately 78% increase compared to the control (6.34 g/quarre/day). The addition of 10% H_2SO_4 did not further improve productivity, suggesting potential physiological stress associated with excessive acidity. Productivity also differed among age-class blocks, with the highest values observed in AC V and AC VI, although these differences likely reflected combined stand-level effects, including stand density and site conditions. These findings demonstrate that low-concentration sulfuric acid modification can enhance the operational performance of organic stimulants by improving ethylene-mediated resin flow while maintaining tree physiological tolerance. These results provide practical implications for improving pine resin productivity in tropical production forests under operational management conditions.

1. Introduction

Perum Perhutani operates the largest pine resin tapping program in Indonesia, employing a range of tapping techniques, including both manual and semi-mechanical methods. (Yovi and Fauzi 2021). Generally, tapping is done manually using the quarre method with the use of stimulants. From 2016 to 2020, the average pine resin production by Perum Perhutani was 86,588 tons/year, while production from outside Java and non-Perhutani areas was 67,972 tons/year, bringing the total national potential to 154,560 tons/year. However, the total industrial demand stands at 281,892 tons/year, which comprises a demand of 138,692 tons/year from Perhutani, 59,800 tons/year from non-Perhutani in Java, and 83,400 tons/year from non-Perhutani outside Java (Nugroho et al. 2021). This significant gap between potential resin supply and industrial processing capacity highlights the need to increase tapping productivity.

In Indonesia, pine resin tapping by Perum Perhutani is mainly conducted on *Pinus merkusii*. The average productivity is approximately 433.35 kg/ha/year, which is relatively low compared with pine tapping productivity in China, which reaches 2,000–2,500 kg/ha/year, particularly for species such as *P. massoniana* managed under intensive tapping practices involving stimulant application (Santosa 2023). Pine resin production is influenced by genetic variation, climate, physiography, edaphic factors, dendrometric properties, anatomical characteristics, fire, pests, and extraction methods. Production can be increased through the application of effective tapping methods and stimulant treatments. (López-Álvarez et al. 2023a; 2023b). Perum Perhutani has established the use of organic stimulants for tapping locations at elevations < 800 m above sea level (a.s.l.) and inorganic stimulants for elevations > 800 m a.s.l. (Perum Perhutani 2022).

Perum Perhutani has been using the organic stimulant ETRAT since 2015, which is a mixture of ethephon ($(HO)_2P(O)CH_2CH_2Cl$) and citric acid ($C_6H_8O_7$). Ethephon is a plant growth regulator containing the active ingredient ethylene (C_2H_4) (Lukmandaru et al. 2021). Ethylene is a plant hormone that exists as a gas and regulates all physiological processes throughout the plant's life cycle. It is produced in stem tissues, and increased ethylene signaling in the cambial region is associated with active secondary xylem (tracheids) (Yu et al. 2023). Ethephon, as an ethylene-releasing compound, enhances secondary metabolism and oleoresin flow in pines. At the same time, citric acid-based stimulant pastes also affect the permeability of the resin channel membranes, allowing the resin to flow smoothly through the tree trunk (Rubio-Pérez et al. 2025). The use of ethylene in tapping pine resin has been carried out using a stimulant composed of an ethylene precursor, such as CEPA (López-Álvarez et al. 2023a), to increase pine resin production in several resin-producing regions, particularly in Southern Europe (Spain and Portugal), South America (Brazil), and parts of Asia (Turkey and China). In addition to its use in pine tapping, stimulants with ethylene as the active ingredient are also used in rubber tapping activities, including for younger or developing rubber trees in Indonesia (Budiasih et al. 2020).

When resin emerges from the resin canal opening and comes into contact with air, it coagulates, potentially clogging the opening and hindering resin flow. The drawback of ETRAT (a commercial stimulant containing 1.25% ethephon and 4% citric acid) is that it does not produce a heating effect; consequently, resin quickly solidifies and blocks the opening. Therefore, organic stimulants are allocated to forest areas with relatively warm ambient temperatures at altitudes < 800 m a.s.l. Low temperatures accelerate resin coagulation, requiring additional heat to maintain resin fluidity. Sulfuric acid can act as a heating agent and has been reported to increase the productivity of tapped pine resin compared to the ETRAT stimulant (Lukmandaru et al. 2021). However, sulfuric acid is a strong acid that can damage xylem, with negative implications for tree health (Rubio-Pérez et al. 2025).

Combining the ETRAT stimulant, which influences resin formation within the tree, and sulfuric acid, which modifies resin flow and physicochemical properties as it exits the trunk, is expected to enhance pine resin production. Previous studies have investigated the use of ethylene precursors such as ethephon, including in combination with sulfuric acid, to improve resin yield

(López-Álvarez et al. 2023a; Lukmandaru et al. 2021; Rodríguez-García et al. 2015). These studies have also documented temporal variation in resin production under repeated tapping, driven by physiological responses and environmental factors. Nevertheless, existing studies have predominantly relied on experimental formulations evaluated under controlled or non-operational conditions, thereby limiting their direct applicability to field-scale resin-tapping practices. In particular, evidence on the performance of commercially available stimulant formulations under operational conditions remains limited. Therefore, this research aimed to examine productivity variation across tapping periods and to evaluate the effect of sulfuric acid addition to an organic stimulant (ETRAT) on pine resin productivity across different age classes under operational Perhutani conditions in *Pinus merkusii* stands.

2. Materials and Methods

2.1. Research Time and Location

The research was conducted from January to March 2024. The study took place in stands of *Pinus merkusii* of age classes III, IV, V, and VI located in BKPH Paninggaran, KPH Pekalongan Timur, Perum Perhutani, Central Java, Indonesia (**Fig. 1**).

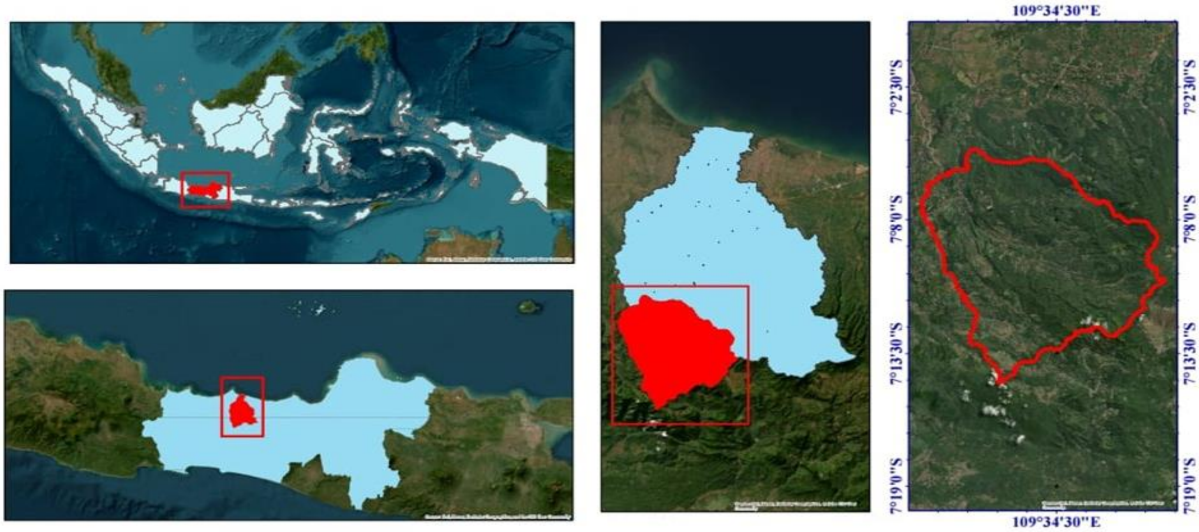

Fig. 1. Multi-scale map of the study area: (a) national-level map showing the location within Indonesia; (b) island-level view; (c) regional context of the sampling site; and (d) detailed boundary of the study area with geographic coordinates.

2.2. Tools and Materials

The equipment used in this research included a diameter tape (phiband), a traditional tapping tool (kadukul), a 1 L hand sprayer, a digital scale, a tapping channel, and a glass measuring cup. In addition, work area maps from KPH Pekalongan Timur and BPKH Paninggaran were used to provide spatial information on the research location. The materials used were ETRAT (a commercial organic stimulant containing ethephon and citric acid, supplied by Perum Perhutani, Indonesia) and sulfuric acid (H_2SO_4), technical grade, approximately 96 to 98%, purchased from a chemical supplier in Bogor, Indonesia.

2.3. Experimental Design and Treatments

This study was conducted on 160 healthy *Pinus merkusii* trees (diameter > 20 cm), distributed across four age classes (AC III, IV, V, and VI), with 40 trees per class. The age classes were defined as follows: AC III (11 to 15 years), AC IV (16 to 20 years), AC V (21 to 25 years), and AC VI (26 to 30 years).

The stimulants used were the organic stimulant ETRAT and a mixture of ETRAT and H_2SO_4. The sulfuric acid concentrations were prepared on a volume-to-volume (v/v) basis. The treatments were A1 (100% ETRAT), A2 (95% ETRAT + 5% H_2SO_4), A3 (90% ETRAT + 10% H_2SO_4), and A4 (control, without stimulant). Each treatment was applied to 10 trees within each age class.

2.4. Resin Tapping Procedure and Data Collection

Initially, a 20 cm × 70 cm area of bark located 20 cm above the ground was cleaned until the grooves were even. The initial wound (initial quarre) was then made at the base of the trunk, measuring 6 cm × 10 cm with a depth of 2 cm into the wood, using a traditional tapping tool (*kadukul*) (**Fig. 2a**). A tapping gutter was installed at the base of the wound through a thin incision made with a machete. A plastic container was placed beneath it to collect the resin. Subsequently, approximately 1 mL of stimulant per tree was applied to the wound using a hand sprayer to ensure even distribution.

After three days, the harvested resin was collected, visually inspected to remove any rainwater, and immediately weighed using a digital scale to ensure accurate measurement. The wound was then renewed by making a new 5 mm cut just above the existing wound using the *kadukul* (Yovi and Amanda 2020), followed by the application of the stimulant. This cycle of resin harvesting, weighing, wound renewal, and stimulant application was repeated every three days for a total of 10 tapping periods. The application volume was standardized at approximately 1 mL/quarre/tapping period. The tapping tools and field activities are illustrated in **Fig. 2**.

Fig. 2. Resin tapping in the study area: (a) traditional tool (*kadukul*); (b) tapping activity; (c) tapped pine stem with resin collection.

2.5. Data Processing and Statistical Analysis

The experiment was arranged in a randomized block design, with treatments A1, A2, A3, and A4, while age classes (AC III to VI) served as blocks. The blocks were located in separate stands within the same rainfall catchment area, with elevations ranging from 620 to 760 m above sea level (m.a.s.l.). Although climatic conditions (such as temperature and rainfall) were generally comparable, variations in stand characteristics, particularly stand density, existed among blocks due to differences in age and forest management practices. Forest management followed Perhutani standards, including thinning during the rotation cycle, with initial spacing of 3 m × 2 m (1,600 trees/ha) decreasing to 300–400 trees/ha in older stands (AC VI). Therefore, the randomized block design was used to account for environmental and stand heterogeneity, and the age class was treated as a blocking factor rather than an independent explanatory variable.

Each combination of treatment and block consisted of 10 sample trees, which served as independent replicates (n = 10). Each sample tree was tapped 10 times at three-day intervals. Resin yield per tapping was recorded, and daily productivity (g/quarre/day) was calculated by dividing the resin weight obtained in each three-day collection period by three. To avoid pseudo-replication from repeated measurements on the same trees, resin yield values from the 10 tapping periods were averaged per tree, and the mean value was used as the experimental unit in the analysis of variance (ANOVA).

Data were analyzed using ANOVA for a randomized block design. If the treatments had a significant effect on pine resin productivity, further testing was conducted using the Tukey test Minitab 18 software (Pennsylvania, USA) to identify significant differences among treatment groups. A significance level of $p = 0.05$ was applied for all statistical tests.

3. Results and Discussion

3.1. Condition of the Sample Plots at the Research Site

The experiment was conducted from January to March 2024 in the Pekalongan region, Central Java, Indonesia. During this period, the study area experienced typical wet-season conditions. Daily climatic data were not recorded on-site; therefore, regional climatological data from BMKG (2024) were used to describe general conditions during the study period. Monthly precipitation ranged from approximately 100 to 300 mm, indicating moderate to high rainfall intensity. Air temperatures ranged from approximately 21 to 32°C, reflecting warm, humid tropical conditions. Such environmental conditions may influence resin viscosity, flow rate, and the likelihood of resin clogging, as reported in previous studies (Lukmandaru et al. 2021).

The research was conducted in pine stands at altitudes of 650–760 m a.s.l. (**Table 1**). The site altitude for these pine stands is close to the optimum growth elevation for pines with the highest resin productivity, both with and without stimulants (Lukmandaru et al. 2021). These findings are consistent with Haryani et al. (2023), who reported that the highest average pine resin productivity in KPH Kedu, Central Java, Indonesia, was 0.226 kg/quarre/month at an elevation of 700–999 m and in age class V. Moreover, Perum Perhutani allocates pine-growing areas at < 800 m a.s.l. for the use of organic stimulants (Perum Perhutani 2022). The forest area is divided into four age classes: AC III, AC IV, AC V, and AC VI. Information for each research location is presented in **Table 1**. An overview of the sample plot conditions is shown in **Fig. 3**. The stands were established on latosol soils and exhibited typical spacing and density conditions for Perhutani-managed pine forests.

All sample trees were subjected to newly established tapping wounds created specifically for this study to ensure uniform initial conditions and eliminate the influence of previous tapping history. The general tree condition was relatively uniform, with trees in productive condition across all age classes and suitable for resin tapping.

Table 1. Stand characteristics of the study plots across age classes

Ages class	Block	Elevation (m.a.s.l)	Planting year	Stand density (trees/ ha)	Soil type
AC III	57A-2	650	2011	800	Latosol
AC IV	58C-3	750	2007	925	Latosol
AC V	60A	620	1999	450	Latosol
AC VI	55L	760	1993	256	Latosol

Notes: Each age class represents a different stand with varying density and site characteristics. The effect of age class should be interpreted with caution.

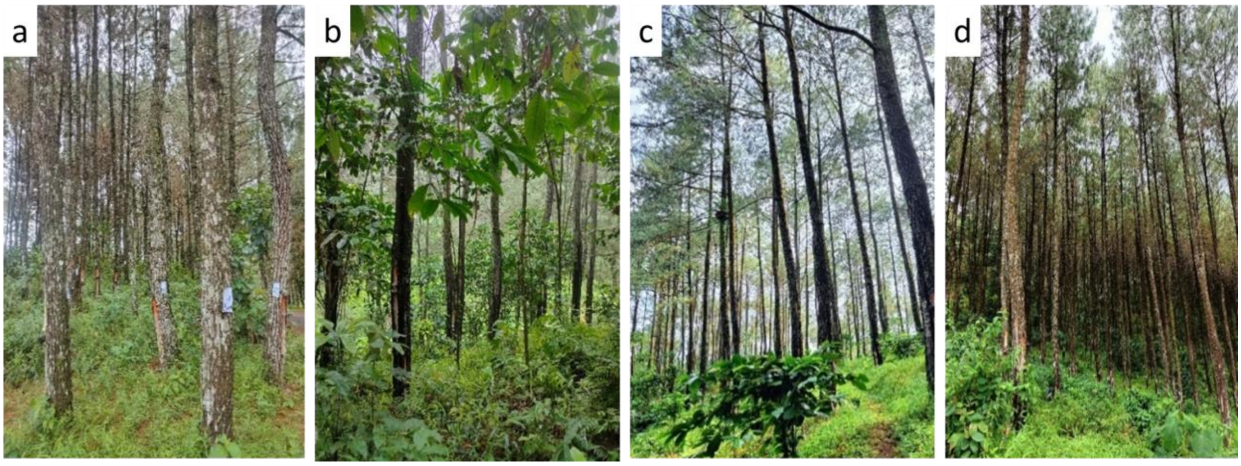

Fig. 3. Condition of research location: (a) AC III, (b) AC IV, (c) AC V, and (d) AC VI.

3.2. Productivity of Pine Resin Tapping

3.2.1. Variation across tapping periods

Resin tapping was conducted over 10 tapping periods, each lasting three days. Resin productivity showed temporal variation across tapping periods, reflecting the dynamic physiological response of the pine trees to repeated wounding (López-Villamor et al. 2021). This variability is primarily influenced by internal physiological processes and, to a lesser extent, potentially by environmental conditions around the tapping site (López-Álvarez et al. 2023a; Rodríguez-García et al. 2015).

Productivity was relatively low during the initial tapping periods, increased toward the middle periods, and declined slightly in the later periods (**Fig. 4**), indicating a dynamic temporal pattern. The increase in resin production during the early to mid-tapping periods is primarily associated with wound-induced physiological responses, including enhanced ethylene signaling and the formation of traumatic resin ducts (TRDs), which enhance resin flow (López-Álvarez et al. 2023a; López-Villamor et al. 2021). In addition to these internal mechanisms, environmental factors during the experimental period, particularly rainfall, humidity, and temperature, may have influenced resin productivity and contributed to the observed variability. Elevated temperatures and lower humidity promote resin fluidity and flow, whereas rainfall may reduce apparent yield by increasing moisture content or interfering with resin collection (López-Álvarez et al. 2023a; Rodríguez-García et al. 2015). During the study period (January–March 2024), the site experienced typical wet-season conditions, with monthly precipitation ranging from approximately 100 to > 300 mm and temperatures between 21°C and 32°C (BMKG 2024). However, meteorological variables were not recorded at a temporal resolution corresponding to each tapping period; therefore, these effects are interpreted as general contributing factors rather than directly quantified drivers.

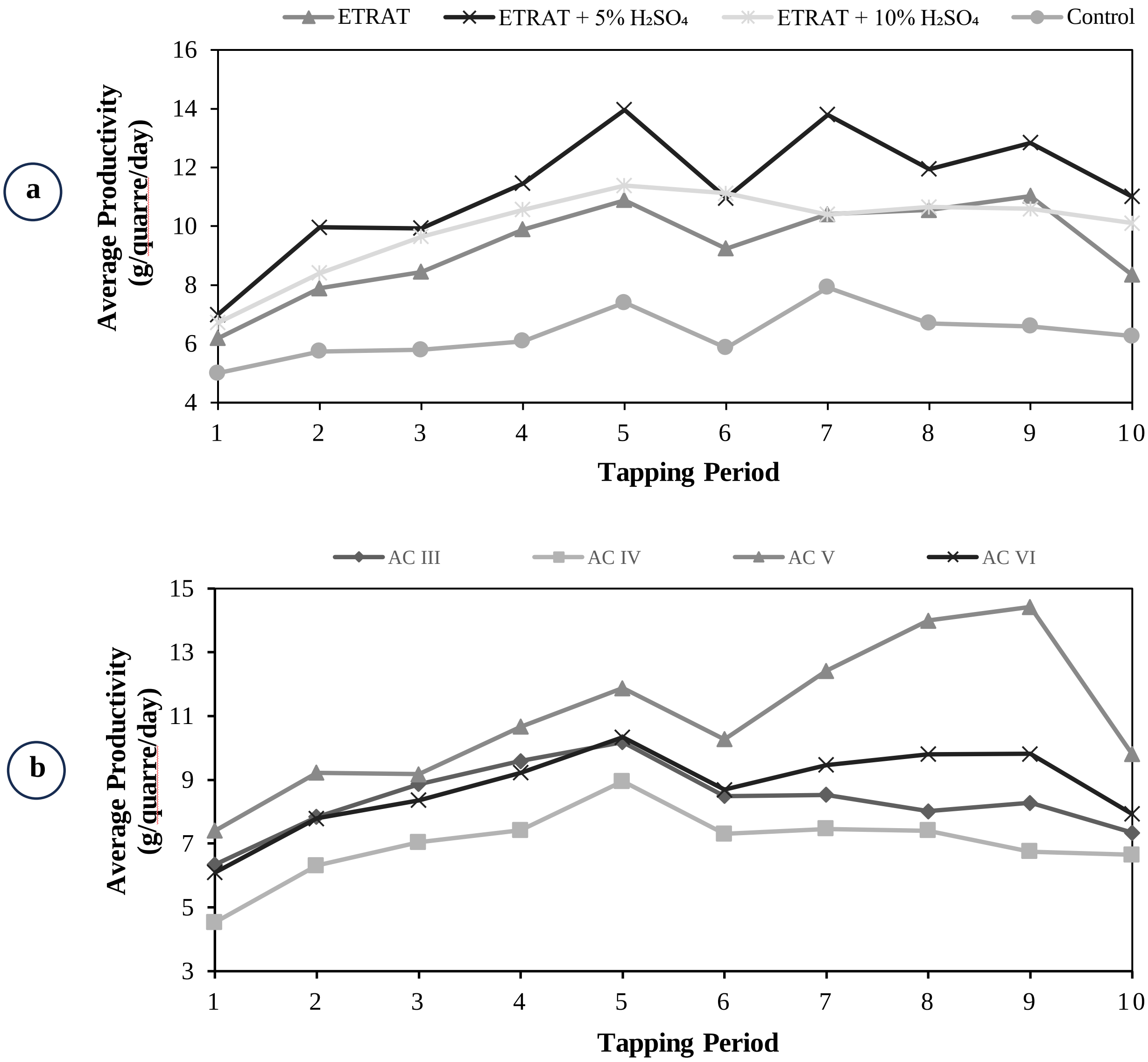

Fig. 4. Average productivity for each tapping period resulting from treatments with various stimulants (a) and across various age classes (b).

Fig. 4a illustrates the differences in stimulant effects on pine resin productivity across the tapping periods. However, the one-way ANOVA indicated no significant difference in average productivity across tapping periods by stimulant type (p = 0.160; **Table 2**). A similar pattern was observed across age classes.

Table 2. Results of the one-way ANOVA test among tapping periods on various types of stimulants

Source	Sum of Squares	Df	Mean Square	F	Sig.
Tapping periods	66.55	9	7.39	1.60	0.16
Error	138.51	30	4.62		
Total	205.07	39			

Although **Fig. 4b** shows variation in productivity among age classes across tapping periods, the one-way ANOVA also indicated no significant difference ($p = 0.152$; **Table 3**). Therefore, because the variation between tapping periods was not statistically significant, tapping productivity for each combination of stimulant treatment and age class was determined by averaging across 10 tapping periods.

Table 3. Results of the one-way ANOVA test among tapping periods across various age classes

Source	Sum of Squares	Df	Mean Square	F	Sig.
Tapping periods	70.69	9	7.85	1.63	0.15
Error	144.83	30	4.83		
Total	215.52	39			

3.2.2. Influence of stimulants and age classes on resin tapping productivity

The experimental design used to determine the effect of stimulants on various age classes was a randomized block design. The results of the analysis of variance (**Table 4**) indicate that stimulant treatment has a significant effect on pine tapping productivity and on age class blocks ($p < 0.05$). However, the effect attributed to age class should be interpreted with caution, as it is confounded with stand-level conditions, because each age class is represented by different forest stands (**Table 1**); therefore, the observed differences reflect a combination of stand age and site-specific factors (e.g., stand density and environmental conditions), rather than the effect of stand age alone.

Table 4. Analysis of variance of the influence of stimulants across various age classes

Source	DF	Adj SS	Adj MS	F-Value	*P*-Value
Block (Age classes)	3	425.01	141.67	12.17	< 0.001
Stimulant	3	525.33	175.11	15.04	< 0.001
Error	153	1781.31	11.64		
Total	159	2731.65			

3.2.2.1. Influence of stimulants on resin tapping productivity

The ANOVA results (**Table 4**) showed that the type of stimulant significantly affected the productivity of tapped pine resin. The Tukey post-hoc test on the effect of stimulants revealed three groupings, indicated by different lowercase letters (**Fig. 5**). The first group utilized the stimulant ETRAT + 5% H_2SO_4, which resulted in the highest productivity. The second group included the use of ETRAT + 10% H_2SO_4 and ETRAT, both of which yielded the same productivity level. The addition of 10% H_2SO_4 to ETRAT did not significantly alter productivity compared to ETRAT alone. The third group, which received no stimulant (control), had the lowest productivity.

Fig. 5 shows that the application of the ETRAT stimulant can increase pine resin tapping productivity by approximately 47% compared to the control, increasing from 6.34 g/quarre/day (without stimulant) to 9.29 g/quarre/day (with ETRAT). Research by Lukmandaru et al. (2021) demonstrated that stimulants such as ETRAT can boost tapping yields by 130–150% compared to controls, depending on environmental conditions and tapping frequency.

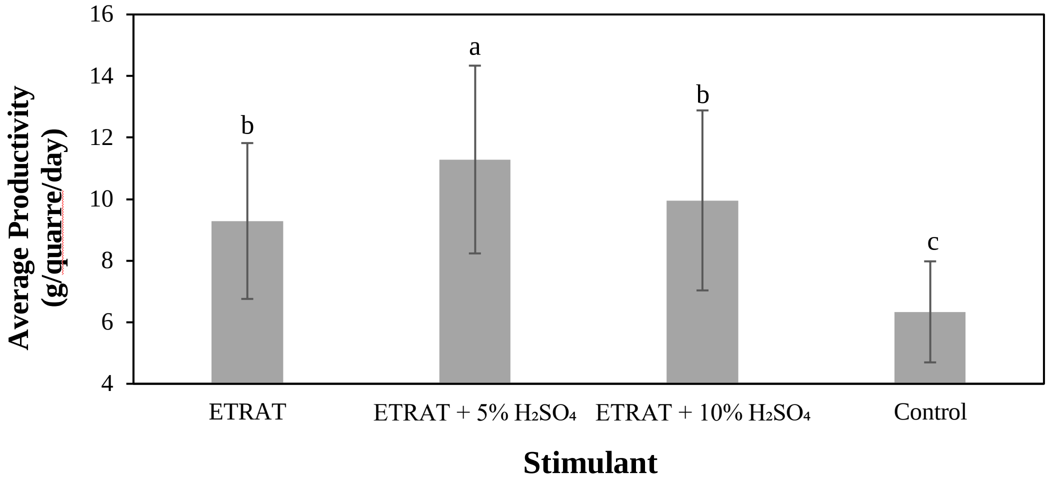

Fig. 5. Average pine resin tapping productivity resulting from various types of stimulant treatments over 10 harvesting periods. Different letters above the bars indicate significant differences ($p < 0.05$).

ETRAT is a stimulant with active ingredients ethephon and citric acid. Ethephon is a plant growth regulator (PGR) that plays an important role in secondary metabolic reactions during latex formation. Ethephon, with the chemical formula ($(HO)_2P(O)CH_2CH_2Cl$) or known as (2-chloroethyl)phosphonic acid, when hydrolyzed, forms ethylene (C_2H_4), phosphoric acid (H_3PO_4), and hydrochloric acid (HCl). This reaction requires acidic conditions. In ETRAT (ethephon 1.25% + citric acid 4%), citric acid ($C_6H_8O_7$) donates H^+ ions to protonate the oxygen atom in the ethephon phosphonate group. This protonation increases the positive charge around the phosphonate group, thereby weakening the C–Cl bond and making it more prone to cleavage. Water molecules act as nucleophiles (attackers) toward the carbon bonded to chlorine (C–Cl). The Cl^- ion is released and combines with H^+ to form HCl. An unstable intermediate compound with an –OH group is also formed. This intermediate undergoes dehydration, resulting in ethylene (C_2H_4) and phosphoric acid (H_3PO_4). The citrate ion (H_2Cit^-) acts as a buffer, stabilizing excess H^+ and preventing the pH from dropping too low (Zhang et al. 2010). The chemical reactions that occur in the ETRAT system are illustrated below.

$$(HO)_2P(O)CH_2CH_2Cl + H_2O \xrightarrow{C_6H_8O_7} C_2H_4 + H_3PO_4 + HCL$$

Ethylene is a hormone that acts as a chemical messenger, triggering secondary metabolism in trees. In the ethephon system, the stability of ethylene release is strongly influenced by solution pH. The presence of weak acids can act as buffers, stabilizing pH and maintaining continuous ethylene release. The more ethylene that is formed, the higher the rate of secondary metabolism, making ETRAT effective in increasing resin production by releasing ethylene that stimulates the activity of resin canals and terpene biosynthesis enzymes (Lukmandaru et al. 2021; Zhang et al. 2010).

Adding sulfuric acid to ETRAT increases pine resin tapping productivity compared with using ETRAT alone. The application of ETRAT stimulant, combined with an additional 5% sulfuric acid, increases pine resin tapping productivity by 78%. For ETRAT stimulant with 10% H_2SO_4, productivity increases by 57% compared to the control. In comparison, the application of ETRAT stimulant alone increases pine resin tapping productivity by 47% (**Fig. 5**). Resin tapping productivity in the ETRAT treatment is lower than in the ETRAT plus sulfuric acid treatments because the hydrolysis rate of ethephon proceeds more slowly under weakly acidic conditions. Citric acid, a weak triprotic acid with a high buffering capacity but low proton donation ability, causes only a small portion of ethephon molecules to break down into ethylene, phosphoric acid, and hydrochloric acid (Zhang et al. 2010).

Because sulfuric acid is a strong acid, its addition greatly increases the concentration of H^+ far beyond what is provided by citric acid. As a result, the medium becomes highly acidic, which greatly facilitates the protonation of the phosphonate group in ethephon and accelerates its hydrolysis to ethylene (Lukmandaru et al. 2021). Therefore, when 5% sulfuric acid is added to ETRAT, the pH of the solution decreases to reach the optimum pH for ethephon hydrolysis. The H^+ ions from sulfuric acid accelerate the controlled release of ethylene without causing tissue damage, thereby significantly increasing the activity of terpene synthase enzymes and resin flow. SO_4^{2-} (from H_2SO_4) remains as a dissolved anion that does not react directly in the C–Cl cleavage mechanism but increases the ionic strength of the solution. H_2Cit^- (citrate residue) can act as a buffer until its capacity is saturated. The presence of citric acid, a weak triprotic acid in ETRAT, with a high buffering capacity, can help resist extreme pH changes, allowing ethylene release to remain stable and continuous (Zhang et al. 2010). The mechanism of ethephon hydrolysis in ETRAT with added sulfuric acid is illustrated below.

$$(HO)_2P(O)CH_2CH_2Cl + H_2O \xrightarrow[C_6H_8O_7]{H_2SO_4} C_2H_4 + H_3PO_4 + HCL$$

The addition of sulfuric acid will facilitate ethylene formation, thereby improving the resin-forming metabolic process. However, excessive addition of sulfuric acid can cause phytotoxicity in plant tissues, trigger excessive ethylene release resulting in physiological stress (leaf drop and premature aging), and increase total acidity due to the formation of HCl, which can worsen damage to tree tissues. **Fig. 5** shows that the highest resin productivity was observed in trees treated with ETRAT + 5% sulfuric acid.

The addition of 10% H_2SO_4 lowers the solution pH to a very low level, causing hydrolysis of ethephon to proceed too quickly. Sudden and excessive ethylene release can trigger physiological stress, including premature aging (senescence), stomatal closure, and damage to secretory cells (Goldental-Cohen et al. 2017), thereby reducing the tree's ability to produce pine resin. In addition, the hydrolysis byproduct HCl can worsen tissue phytotoxicity, damage cell membranes, and interfere with the activity of terpene biosynthetic enzymes required for resin production (Zhang et al. 2010). As a result, although initial stimulation is high, the resin-producing tissues are quickly damaged, and resin production decreases. A similar phenomenon was also reported by Lukmandaru et al. (2021), who found that the success of ethephon stimulation depends on a balance between solution acidity and the plant tissue's ability to tolerate low pH. Thus, the addition of 5% H_2SO_4 provides optimal conditions for ethephon decomposition and physiological stimulation without causing damage, whereas a 10% concentration induces toxic effects that reduce production efficiency.

3.2.2.2.The influence of age class (block) on pine resin tapping productivity

The results of the variance analysis (**Table 4**) show that pine tree age class affects the productivity of pine resin tapping. **Fig. 6** shows three groups: the first is the productivity of pine resin tapping in stands of age class V, which is the same as age class VI. The productivity of pine resin tapping in stands of age class III is lower and significantly different from classes V and VI, but higher compared to age class IV. The lowest productivity is found in age class IV, unlike the other classes.

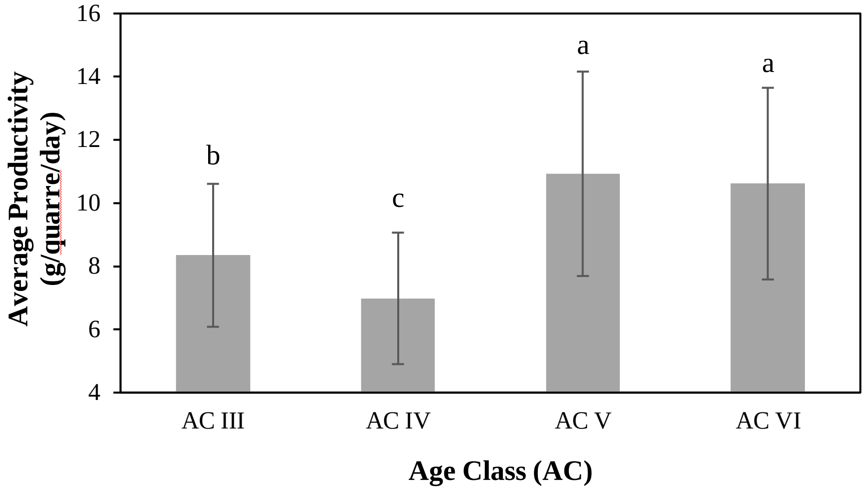

Fig. 6. Average pine resin tapping productivity resulting from various types of age class treatments over 10 harvesting periods. Different letters above the bars indicate significant differences (p < 0.05).

Fig. 6 shows a tendency for pine resin tapping productivity to increase with tree age. This pattern is consistent with previous studies indicating that older trees tend to have a greater abundance of resin ducts, which are positively correlated with resin production (Zas et al. 2020). In this study, pine resin tapping productivity in AC V (10.93 g/quarre/day) was among the highest and not significantly different from AC VI, indicating comparable performance between these age classes. Although differences in resin productivity were observed among age classes, these should be interpreted with caution. Each age class represents a distinct stand with varying structural and characteristic features, particularly stand density and site conditions. Such differences may act as confounding factors influencing productivity. For example, AC IV showed lower tapping productivity (6.98 g/quarre/day) than AC III (8.35 g/quarre/day), as shown in **Fig. 6**, despite being older, which may be related to its higher stand density. AC IV had a higher stand density (925 trees/ha) than AC III (800 trees/ha), as presented in **Table 1**. Increased stand density can intensify competition for light and resources, potentially reducing resin production, as reported in previous studies (López-Álvarez et al. 2023a; Wang et al. 2017). Therefore, the observed variation likely reflects the combined influence of stand conditions and age rather than age alone.

Higher stand density increases competition for growing space and reduces the crown size of individual trees. Pine resin production has been reported to be influenced by tree diameter, crown ratio, and competition index. Crown closure affects the growth of pine trees (Wang et al. 2017). Reduced crown size limits light interception and decreases photosynthetic capacity, thereby reducing the availability of energy for growth and resin biosynthesis. In addition, high tree density will lead to smaller tree diameters. This finding is consistent with López-Álvarez et al. (2023a), who stated that tree diameter and crown closure are key factors influencing pine resin production, with larger diameters associated with higher resin yield. Therefore, stand density indirectly influences resin productivity through its effects on crown development and tree growth.

The sample plots used as observation sites still implement a silvicultural system aimed at producing pine wood. At the time of planting, a spacing of 3 m × 2 m was applied. As the trees age, thinning is carried out to provide growing space for the pine trees. Thinning is gradually carried out until the stand reaches an optimal tree density of approximately 400 trees per hectare. Silvicultural actions, such as thinning, not carried out in accordance with regulations, have resulted in the AC IV sample plot having a higher tree density than the AC III plot. The increase in pine resin production can, in part, be attributed to the formation of traumatic resin canals. Recent studies have demonstrated that stem wounding and fungal inoculation induce TRD formation in conifers as part of the defense response, thereby enhancing oleoresin production (Mercado et al. 2023). Induction of traumatic resin canals in pine trees can also be achieved using methyl jasmonate (López-Villamor et al. 2021).

4. Conclusions

Resin productivity did not differ significantly across tapping periods, indicating that the average of repeated measurements provides a reliable estimate of treatment performance. Adding sulfuric acid to the ETRAT stimulant increased pine resin productivity under the tested conditions. The application of ETRAT with 5% sulfuric acid resulted in the highest productivity (11.29 g/quarre/day), representing an increase of approximately 78% compared to the control. Resin productivity also varied among age classes, with the highest values observed in age class V (10.93 g/quarre/day), which was not significantly different from that of age class VI. However, these differences should be interpreted as block effects associated with stand-level conditions rather than purely age-driven responses. Overall, the results suggest that adding sulfuric acid at low concentrations can improve the performance of organic stimulants in pine resin tapping under operational conditions. Further studies are needed to better account for the hierarchical data structure and to evaluate long-term effects on tree health.

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Author Contributions

G.S.: Conceptualization, Methodology, Software, Validation, Formal Analysis, Investigation, Resources, Writing – Original Draft Preparation, Writing – Review and Editing, Visualization, Supervision; J.M.R.: Conceptualization, Methodology, Writing – Original Draft Preparation, Writing – Review and Editing; R.D.: Data Analysis and Software; D.A.: Data Analysis and Software; R.K.S.: Conceptualization, Methodology, Writing – Original Draft Preparation, Writing – Review and Editing.

Conflict of Interest

The authors declare no conflict of interest.

Declaration of Generative AI and AI-Assisted Technologies in the Manuscript Preparation

During the preparation of this work, the authors used Turnitin to provide a more comprehensive and organized discussion. After using this tool/service, the authors reviewed and edited the content as needed and take full responsibility for the content of the publication.

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