International Journal of Research Publication and Reviews, Vol 6, no 2, pp 4212-4218 February 2025

International Journal of Research Publication and Reviews

Journal homepage: www.ijrpr.com ISSN 2582-7421

An Extraction Process of Tannin from Guava Leaves (Psidium Guajava L.) As a Thermal Insulator and Flame Inhibitor for Wood Material Protection

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ABSTRACT

This study explores the extraction of tannins from guava leaves (*Psidium guajava L.*) as a natural flame retardant and thermal insulator for wood protection. Tannin-infused wood samples underwent flame burning and hygrometric tests, showing a significantly lower weight loss (0.34g) compared to untreated wood (0.98g). An independent t-test confirmed a statistically significant difference ($t = -39.147$, $p < 0.001$), demonstrating the tannin's efficacy in enhancing fire resistance. These results support the potential of guava leaf tannins as an eco-friendly alternative to conventional fire retardants in construction materials.

Keywords: Tannins, Psidium Guajava L., Natural Fire Retardant, Hygrometric Analysis, Flame Burning Test, Flame Inhibitor, Thermal Insulator

I. Introduction

Fire is a very versatile disaster, and the world has faced countless fire incidents for decades, if not centuries. Fire incidents have occurred frequently in the Philippines for the past years. Some accidents are relatively low, with few to no injuries and ruins, while others are disastrous, drastically altering people's lives for the worse. A recent report from the Bureau of Fire Protection (BFP) stated that the primary cause of fires is smoking, followed by unsupervised open flames from cooking and electronic ignitions. BFP also stated from the same report that from January 1 to March 1 of the year 2024, 3,044 fire incidents were reported, compared to 2,424 incidents in the same period the previous year (Caliwan, 2024). An evident increase is observed, therefore an intervention that would effectively mitigate the spreading of fire is necessary.

Tannin is a polyphenol molecular structure found in vascular plants. Its molecular weights range from 500 to 3000 kDa (Govindarajan et al. 2016). A high molecular weight indicates stronger thermal stability, which suggests that tannin can endure higher temperatures without breaking down. Moreover, a high molecular weight also indicates a more stable formation of char layer which is also a great insulating barrier. An experimental study conducted by Sebestyén et al. (2019) proved that Condensed tannins had the highest char production, compared to hydrolyzable tannins. Tannins exhibit low thermal conductivity and strong chemical and thermal stability above 300 °C because of their distinctive aromatic structure (Júnior et al. 2024). It is a type of naturally occurring plant polyphenols with a broad source and abundant reserves. Additionally, a study by Lisperguer et al. (2016) applying tannins obtained from Acacia dealbata bark showed significant thermal resistance via Thermogravimetric Analysis. In recent years, TA based on textile, steel, and other natural and synthetic polymeric materials has shown great potential for flame retardancy (Basak et al., 2012b). Furthermore, Wang et. al., (2022) also proved tannin's benefits to the flame-retardant industry by stating that it has a low combustion. Moreover, Pizzi et al. (2024) stated that common sources of tannins include wood, leaves, buds, stems, fruits, seeds, roots, and plant galls. Tannin coating modified the color of the wood and hindered the spread of fire; its presence also significantly shortened the duration of the flames while preserving the structural integrity of the wood (Júnior et al., 2024b).

Guava leaves (Psidium guajava L.) are abundant in tropical countries like the Philippines. In accordance with the extraction process executed by Faradilla and Rizal (2023), it contains a variety of chemical compounds, such as tannins, flavonoids, and saponin. According to a different study conducted by Mailoa et al. (2014), guava leaves (Psidium guajava) consist of 9% tannin. These qualities of Guava leaves (Psidium guajava) enable them to be an efficient fire-retardant material.

For generations, people who are eager to seek a solution for this issue continuously do research about this matter. One of the potential answers for flame retardancy is tannin. As per reported documents, the past few years have witnessed great flame retardancy potential of tannin and tannin-based plant bio-macromolecules on textile, steel and other natural and synthetic polymeric materials. However, till date, no critical review has been registered on the compilation of the flame-retardant performance of tannin (Basak et al., 202b). Additionally, although tannin is considered to have great potential for flame retardancy, the potential for utilizing tannin as a fire retardant has been limited by its poor thermal stability (Xia et al., 2018). Depending on the method used and the addition of flame retardants to a flame-retardant system based on tannin, various flame retardation modes of action and fire-resistance performances are achieved (Wang et al., 2018). These previous findings regarding this topic suggest we implement a more thorough process in doing a comprehensive and in-depth study of tannin's chemical composition.

If the tannin acid proves to act as a viable alternative to commercial flame retardants, then it could substantially benefit the civil construction industry as well as families residing in the slums. Tannins, known for their renewable nature and having low toxicity to human health, hold promise for enhancing the thermal properties of wood. Implementing this improves the fire resistance of wood thus increasing its durability and safety as a construction resource. This paper aims to assess the efficacy of tannins as flame retardants on materials such as wood and compare which tannin-producing plant is more potent. In this study, Guava Leaves (Psidium Guajava L.) will be the source of tannin as it is abundant in the Philippines and rich in tannin at the same time. The wood will undergo immersion under the extracted tannin solution for 24 hours. The results will later then be evaluated through a Flame Burning Test and a Hygrometric Analysis. The findings of this study are anticipated to support tannins as alternatives to commercial flame retardants in the market.

II. Results and Discussion

A. Flame Burning Test

In this aspect of the paper, the flame resistance of the samples was measured by the burning test. The samples were of the same size and the duration of burning was of also the same time.

Table 1. Shows the mean of the weight loss and standard deviation (SD) of tannin-coated treatments compared to controlled treatments

	Tannin-coated	**controlled**
Valid	9	9
Missing	0	0
Mean	0.344	0.979
SD	0.011	0.047

Table 1 presents a comparison of mean weight loss between tannin-coated treatments and control treatments, highlighting significant differences. Both groups consist of 9 valid samples, with the tannin-coated treatments exhibiting a mean weight loss of 0.344, which is substantially lower than the control group's mean of 0.979. Furthermore, the standard deviation for the tannin-coated treatments is 0.011, indicating greater consistency in the results compared to the control group's standard deviation of 0.047. This suggests that the tannin coating effectively mitigates weight loss, likely contributing to enhanced thermal stability and durability.

Table 2. Shows the efficacy of the treatment using independent t-test compared to controlled treatment.

Independent Samples T-Test

	t	df	p
weight loss	−39.147	16	< .001

Note. For all tests, the alternative hypothesis specifies that group *tannin-coated* is less than group *controlled* .
Note. Student's t-test.

Table 2 illustrates that the independent samples t-test reveals a highly significant difference in weight loss between the tannin-coated and control groups ($t = -39.147$, $p < 0.001$), with the negative t-value indicating that the tannin-coated group lost significantly less weight. The p-value below 0.001 provides strong evidence to reject the null hypothesis that there is no difference in weight loss of the two treatments, supporting the alternative hypothesis that tannin-coating reduces weight loss more effectively.

B. Hygrometric Data Analysis

In this section, it was tested whether or not tannin-treated samples can act as a thermal insulator. To do this, the temperature and humidity was constantly monitored using a digital hygrometer by calculating both air pressure and temperature changes in an environment. The data were recorded at 15-minute intervals with the whole testing lasting one hour and thirty minutes.

Table 3. Shows the mean and standard deviation of the first trial of the Hygrometric Data Analysis

Descriptive Statistics

	2:00PM		2:15PM		2:30PM		2:45PM		3:00PM		3:15PM	
	Experimental	Controlled	Experimental	Controlled	Experimental	Controlled	Experimental	Controlled	Experimental	Controlled	Experimental	Controlled
Mean	35.733	36.733	35.467	36.267	35.467	36.267	34.200	34.887	32.900	35.267	33.267	33.533
Std Deviation	0.153	0.589	0.153	0.404	0.153	0.404	0.173	0.306	0.794	1.155	0.058	0.058

Table 3 showcased the hygrometer readings at 15-minute intervals in a span of an hour and 15 minutes with a total of 6 stages starting from 2:00 PM until 3:15 PM. The temperature of tannin treated samples (Experimental Treatment) were consistently decreasing each passing stage. The same goes with the tannin-free samples (Controlled Treatment) however, the temperature recorded from experimental treatment were significantly lower indicating that there is a steady and constant drop in temperature over the course of the whole time period despite having two stages namely, stage 4 (2:45 PM) and stage 6 (3:15 PM) with results close to each other. Stage 5 (3:00 PM) had the biggest difference in temperature drop with a mean difference of 2.37°C. The trend of the experimental treatment suggests that tannins indeed acted as thermal insulators as it retained heat over time. The results in temperature confirmed that tannin-infused wood samples are effective in thermal stability. There is a statistically significant difference and measurable effects between tannin treated samples and the controlled samples. Across all time intervals, the P values are below the significance level of 0.05, indicating that the results are significant and stating that tannins are effective in heat retention, supporting the alternative hypothesis. Experimental treatment trend likely has better heat retention and insulating properties than controlled treatment which explains why despite having the two treatment's temperature going down across each stage, the data recorded from the experimental treatment still has a significantly lower temperature drop.

III. Conclusions

This paper examines the possibility of infusing flame retardancy using tannins extracted from Psidium Guajava leaves for wood material protection. The utilization of tannins as a fire retardant has made significant changes in the wood's thermal stability and combustion behavior. The data analyzed showed that the presence of tannin reduced the spread of fire in burning conditions with short exposure to flame and heightened its heat retention making it a suitable thermal insulator. The study demonstrated that the tannin compound has the ability to modify the properties of wood making it effective against heat and fire. These characteristics allowed the development of an efficient fireproof protective coating and shows that the unique psychochemical properties of tannin can be utilized effectively in the field of construction in the industry and ultimately expand the tannin potential application.

For the succeeding researchers, this study strongly suggests utilizing various different types of wood that is bigger in size and investigate further its ability to reduce heat consumption and fire risks through a more detailed analysis such as the Thermogravimetric Analysis.

IV. Methods

A. Preparation and Collection of Materials

Guava leaves (psidium guajava L.) were collected at the Jumawan Residences' backyard. The collected guava leaves (psidium guajava L.) were dried for a week until they were completely crunchy. Afterwards, the dried leaves were crushed using mortar and pestle. Additionally, other materials such as ethanol, beaker, brushes, and containers were also prepared by the researchers. Popsicle sticks were utilized to undergo flame burning tests and hygrometric analysis measuring 15.0 cm in length, 1.8cm in width and 0.16cm in thickness. This was bought from a local bookstore in town. Chipboards that are 22.5x 28 inches in size were bought in the same store as material for miniature houses production necessary for the hygrometric analysis. Digital Hygrometers were also acquired and prepared for the purpose of testing. For safety and precautions, the researchers ensured the implementation of protective protocols during the preparation and collecting process. Accordingly, the researchers ensured that the chosen materials were approved by the professionals through consultation.

Figure 1: Picking of Guava Leaves

Figure 2: Preparation of Tools

B. Extraction of tannins from Guava Leaves

The extraction process was aligned with the paper of Mailoa et al., (2013). The process of extraction was by maceration. The powdered leaves were soaked in 95% ethanol solution for one week. 150 mL of Ethanol was used as the solvent because of its ability to dissolve polar and even non-polar compounds. The ratio is approximately 3.73 ml/g. For every 150 ml of ethanol, 40.22g of leaves were mixed. Subsequently, the mixture was filtered to acquire the filtrate - tannins. To confirm its concentration, the solution underwent the process of rotary evaporation to vaporize the solvent and ensure the pureness of the filtrate.

Figure 3: Crushing of Guava Leaves

Figure 4: Soaking to Ethanol

Figure 5: Receipt of Rotary Evaporation

C. Application of the Tannin Solution

The application process was aligned with the methods of Júnior et al. (2024). The popsicle sticks were soaked in tannin for 24 hours to ensure that the tannin content will tightly hold on to the wood material. The tannin was extracted from one source only and a total of one treatment has been done. With these, a total of 18 samples were used including the controlled samples that did not contain tannin. Another 12 samples were used for another testing including the controlled samples. In the immersion procedure, it was ensured that all surfaces of the popsicle sticks were in contact with the solution by occasionally stirring the solution or moving the container.

Figure 6: Brush coating of Tannins

Figure 7: Immersion of Popsicles

D. Analytical Characterization

To evaluate the thermal stability and combustion behavior of the tannin-treated woods, all samples underwent flame burning test and hygrometric test for effective results.

E. Flame Burning Test

In this process of testing the efficiency of tannin, the weight loss of the woods was measured to determine the thermal deterioration profile. The popsicle sticks measured 15.0cm in length, 1.8cm in width, and 0.16 in thickness. Primarily, the popsicles with zero tannin content were weighed and the same goes with the tannin-immersed popsicles. During the burning test, popsicles with and without tannins were burned for two minutes straight using an alcohol lamp. Each of the samples were replicated nine times - giving the researchers a total of eighteen samples - to gauge the precision and accuracy of the result. The two-minute duration of burning was set considering the size of the popsicle stick. After two minutes, the popsicles were weighed again to determine the difference between the initial and final weights. Afterwards, the popsicles were weighed again to determine the difference between the initial and final weights.

Figure 8: Burning of Popsicle without Tannins

Figure 9: Burning of Popsicle with Tannins

Figure 10: After burning results

F. Hygrometric Test

The hygrometric test was conducted to measure the humidity of houses made of tannin roofs. Initially, the researchers formed six miniature houses using chipboards as the enclosing walls and popsicles for the roof. The house was made using four equal boards each measuring 6.5x6.5 inches each as the walls. The roof was made of popsicle sticks, with both sides measuring 16.2 cm in width and angled to resemble a traditional triangular roof. The base was made out of cardboard to support the whole construction of the house. A small hole was poked in one of the walls of each house to insert the digital hygrometer to initiate testing. Consequently, three roofs were brush coated with tannins and three were not. The roofs without the tannins were called the controlled variables. Afterwards, hygrometric testing was conducted to determine the change in humidity and temperature for every 15 minutes in a span of 1 hour and 30 minutes. This test underwent two trials at one day interval to see whether there would be significant difference in the results or not.

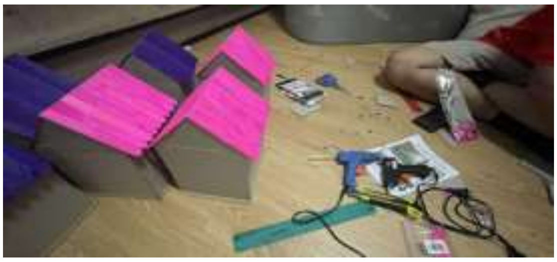

Figure 11: Making of MIniature Houses

Figure 12: 1st Test at our Adviser's Place

Figure 13: 2nd Testing at SHS Quadrangle

Risk and Safety

To establish the promised precautionary actions, the researchers ensured the proper implementation of safety measures. During the initial procedure of extracting the tannins, the researchers wore lab coats and properly disposed of the wastes. Additionally, in the process of rotary evaporation, the students were guided by the professionals. Ultimately, during the flame burning test, the researchers used a tong to hold the samples and avoid the risk of being burned and scalded.

Data Analysis

The researchers conducted a comprehensive analysis of the data utilizing descriptive statistics, specifically focusing on the mean and standard deviation. These statistical measures facilitated the assessment of weight loss variations among the popsicle sticks exposed to different treatments. To evaluate the significance of the observed differences in treatment outcomes, an Independent T-test was employed. In addition, the same statistical techniques were applied to analyze the temperature differences derived from the hygrometric analysis, ensuring a thorough examination of the data across both experimental contexts.

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VI. Appendices

Table 1. Shows the initial weight, final weight, weight loss, and weight loss percentage of the experimental treatments (with tannins).

Initial Weight	Final Weight	Weight Loss	Weight Loss Percentage
1.17g	0.83g	0.34g	29.1%
0.89g	0.53g	0.36g	40.4%
0.92g	0.58g	0.34g	37.0%
1.15g	0.80g	0.35g	30.4%
1.15g	0.79g	0.36g	31.3&
0.96g	0.63g	0.33g	34.4%
1.13g	0.80g	0.33g	29.2%
1.17g	0.83g	0.34g	29.1%
0.94g	0.59g	0.35g	37.2%
AVERAGE: 1.05g	**0.71g**	**0.34g**	**33.35%**

Table 2. Shows the initial weight, final weight, weight loss, and weight loss percentage of the controlled treatments (without tannins).

Initial Weight	Final Weight	Weight Loss	Weight Loss Percentage
1.16g	0.16g	1g	86.2%
1.17g	0.18g	0.99g	84.6%
1.13g	0.15g	0.98g	86.7%
0.98g	0.12g	0.86g	87.8%
1.16g	0.14g	1.02g	88.0%
1.15g	0.15g	1g	87.0%
0.89g	0.12g	1.01g	86.5%
1.16g	0.18g	0.98g	84.5%
1.14g	0.17g	0.97g	85.1%
AVERAGE: 1.10g	**0.15g**	**0.98g**	**86.27%**