Biochar-Assisted Purification of Wood Vinegar Derived from Coconut Shell Pyrolysis

Praveen Dhananja Lakshan, Anjalika Sewwandi Manoharan, and Duleeka Sandamali Gunarathne

1. Introduction

Wood vinegar, also known as pyroligneous acid, is a liquid byproduct obtained from the process of slow pyrolysis of biomass [1]. Slow pyrolysis involves the thermal decomposition of organic materials at temperatures typically ranging between 300°C and 800°C in the absence of oxygen, which results in the production of biochar, syngas and wood vinegar [2]. The primary feedstocks for wood vinegar production include various types of biomasses such as hardwood, softwood and agricultural residues [3].

During slow pyrolysis, biomass undergoes a series of complex chemical reactions, leading to the formation of condensable vapors and non-condensable gases. The condensable vapors, when cooled, form a liquid mixture comprising water, acetic acid, methanol, acetone, and a variety of phenolic compounds [2]. This liquid mixture is known as wood vinegar. The composition of wood vinegar can vary significantly depending on the type of feedstock, pyrolysis temperature, and pyrolysis duration [4].

Wood vinegar has garnered significant interest due to its diverse range of potential applications in agriculture, animal husbandry, and environmental management. For instance, it can be used as a natural pesticide, soil conditioner, and growth promoter in agricultural practices [5]. Moreover, it has been shown to possess antimicrobial properties, making it useful in animal husbandry for improving the health and hygiene of livestock [6].

The production of wood vinegar from slow pyrolysis offers a sustainable approach to valorizing biomass waste, contributing to waste management and the development of bio-based products. As research progresses, the optimization of pyrolysis conditions and the refinement of wood vinegar composition continue to enhance its applicability and effectiveness in various domains [4].

Wood vinegar can be purified using various methods to enhance its quality for use in agriculture, food preservation, and other applications [7]. Common purification techniques include adsorption processes employing materials such as activated carbon or zeolites, which effectively remove tar, particulates, and other impurities. Other techniques include distillation to separate and concentrate desired compounds, settling and decantation for natural separation of heavier tar particles, and advanced methods like membrane filtration (including ultrafiltration and nanofiltration) that target molecular size for more precise purification [7]. Acid-base neutralization and solvent extraction are also employed to adjust the chemical properties and extract specific components, respectively, further refining the quality of wood vinegar for its varied uses [8]. These methods have been extensively researched in studies that analyze their efficiency, cost-effectiveness, and suitability for scaling up to industrial applications. Among the available purification methods, adsorption is considered a promising approach for wood vinegar purification.

Since activated carbon is typically two to five times more expensive than biochar, biochar is an economical alternative to commercial activated carbon in adsorption-related application [9]. Further, life-cycle analyses show that biochar production can have much lower energy demand (~6 MJ/kg) compared to activated carbon (~97 MJ/kg), and even negative net greenhouse-gas emissions due to carbon sequestration [10]. Biochar can sequester carbon stably over long periods, helping mitigate CO_2 emissions [11]. Therefore, biochar stands out as an economical, sustainable, and eco-friendly adsorbent compared to commercial activated carbon. This study is a preliminary investigation on the use of coconut-shell-derived biochar produced at low pyrolysis temperatures for wood vinegar purification. Most reported adsorbents are prepared at high temperatures and feasibility and performance of low-temperature biochar for the purification of wood vinegar, also produced from coconut shells via slow pyrolysis is limited. The study aims to assess the adsorption capability of such biochar in removing impurities while refining wood vinegar to meet agricultural standards, thereby supporting safer and more sustainable agricultural practices.

2. Methodology

2.1 Materials

In the preparation of wood vinegar and biochar for the study of adsorption processes, coconut shells were selected as the primary raw material due to their abundant availability and carbon-rich composition. Initially, the coconut shells were thoroughly cleaned to eliminate any surface impurities that could affect the purity of the final products. Following cleaning, the shells were mechanically crushed into smaller pieces, approximately 0.1 cm^2 in size, to enhance the surface area available for chemical reactions and heat transfer during the production process. Finally, these crushed pieces were oven-dried at 105 °C for one hour to remove residual moisture, ensuring consistent quality and reactivity in the subsequent preparation stages.

2.2 Methods

2.2.1 Preparation of Biochar and Wood Vinegar

Biochar and wood vinegar were produced using a slow pyrolysis reactor operated at 340 °C for 90 minutes, where the temperature was selected based on TGA data of coconut shells reported in literature [12], and the residence time ensured complete devolatilization corresponding to selected temperature [13]. The gases released during pyrolysis were passed through tubing into a water-filled bottle placed in a cold-water bath to promote condensation. This condensed liquid, termed wood vinegar, was then collected. After production, all samples of wood vinegar were stored in sealed glass bottles and shielded from sunlight to preserve their chemical integrity and prevent any degradation [14]. The experiment was replicated twice, and each replicate was subsequently divided into four sets for further analysis. Since direct condensation produces a dilute mixture of wood vinegar and water, the actual wood vinegar concentration was calculated, resulting in a concentration of 10.6 ± 0.2%. Resulting biochar from each experiment was stored for further pretreatment.

1.1 2.2.2 Pretreatment of Biochar

To enhance the adsorption properties and purity of biochar, several pretreatments were performed. The biochar was first ground into a fine powder to increase surface area, then washed 5–8 times with distilled water until the runoff was clear. It was next soaked in 10% (v/v) acetic acid for 24 hours to remove impurities and modify its chemical properties. After this acid treatment, the biochar was washed three times with distilled water to remove any residual acid. Then treated with 0.1 M NaOH for 24 hours to neutralize residual acidity and enhance surface activation. [15]. Post-neutralization, the biochar was washed until the pH of the wash water reached neutral, ensuring the removal of any residual alkalinity. Finally, it was oven-dried at 105 °C for 90 minutes to eliminate moisture, yielding purified, activated biochar ready for experimentation. (see **Figure 1**)

Figure 1. Pre-treated Biochar.

2.2.3 Wood Vinegar Purification

To evaluate the purification efficiency of biochar, eight samples were prepared by varying the residence time (1, 2, and 3 days) and the mixing ratios of wood vinegar to biochar. These parameters were selected because adsorption performance is mainly influenced by adsorbent dose and contact duration [16]. By altering these two factors, the study aimed to determine their effect on the removal of tar and other impurities, and to compare the treated samples with raw wood vinegar. Mixing ratios and conditions are summarized in **Table 1**. Adsorption was carried out at 8 °C to enhance adsorption performance, as lower temperatures generally improve the uptake of impurities by biochar. After treatment, the mixtures were filtered using F1001 grade filter papers to separate the purified wood vinegar from biochar residues.

Table 1. Description of Samples with Conditions

Set	Sample	Description
Set 1 (At 8 °C, Wood vinegar: Biochar 1:1)	WV_{Raw1}	Raw wood vinegar of sample set 1
	WV_{1day}	Wood vinegar treated with biochar for 1 day
	WV_{2days}	Wood vinegar treated with biochar for 2 days
	WV_{3days}	Wood vinegar treated with biochar for 3 days
Set 2 (At 8 °C, Wood vinegar treated with biochar for 3 days)	WV_{Raw2}	Raw wood vinegar of sample set 2
	$WV_{4:1}$	Wood vinegar: Biochar mixing ratio is 4:1
	$WV_{2:1}$	Wood vinegar: Biochar mixing ratio is 2:1
	$WV_{1:1}$	Wood vinegar: Biochar mixing ratio is 1:1

2.3 Materials Characterization

A series of seven analytical tests were performed to measure key properties indicative of purity of wood vinegar, including Fourier-transform infrared spectroscopy (FTIR), spectrophotometry, pH, density, refractive index, and Brix value.

3. Results and Discussion

3.1 Product Yield

Figure 2 shows the product distribution from coconut shells pyrolysis at 340 °C for 90 minutes. Showing a similar trend, literature reported a yield of 42 wt% charcoal and 53 wt% wood vinegar for coconut shells pyrolyzed at 300 °C for 5 hours [17].

3.2 FTIR Analysis

Fourier Transform Infrared (FTIR) spectroscopy was utilized to evaluate the effects of contact time between wood vinegar and biochar on the purification of wood vinegar derived from coconut shells. The primary objective was to explore how increasing the contact time between wood vinegar and biochar influences the removal of tar compounds, which are predominantly phenolic in nature. The study considered a raw sample of wood vinegar and three samples treated with biochar for 1, 2, and 3 days, respectively. The FTIR results of wood vinegar samples subjected to purification using different residence times of wood vinegar and coconut shell biochar are shown in **Figure 3.**

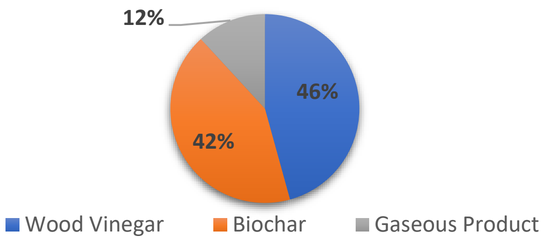

Figure 2. Pyrolysis Product Distribution from 35.39g Coconut Shell.

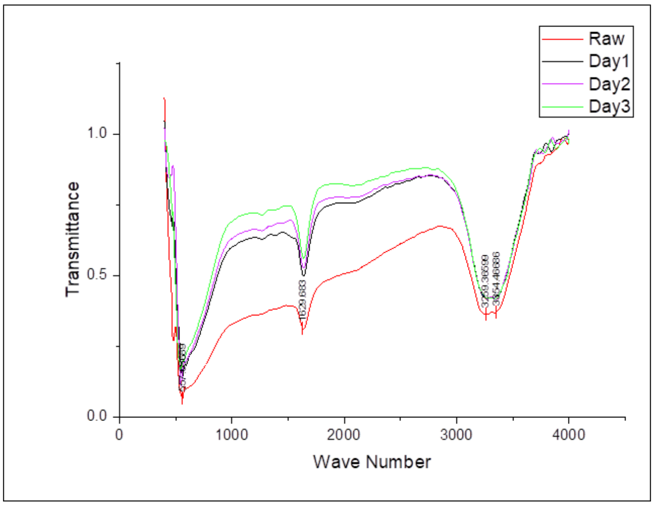

Figure 3. FTIR Results of Sample Set 1.

The FTIR analysis shows clear differences between the raw wood vinegar and the samples treated with biochar. The broad peak at 3200–3550 cm^{-1} (–OH groups) increases in transmittance after treatment, indicating the removal of phenolic and alcoholic compounds. This reduction is most prominent on Day 1, suggesting rapid adsorption, potentially through hydrogen bonding and surface complexation with oxygenated functional groups on biochar.

The peak at 1566–1650 cm^{-1}, associated with aromatic rings, also shows increased transmittance in purified samples, reflecting a decrease in aromatic compounds. This removal is consistent with π–π interactions between biochar's graphitic surfaces and aromatic tars, supported by hydrophobic interactions that promote adsorption of non-polar species. The diminishing changes over time may result from the gradual saturation of active sites.

The Fourier-transform infrared spectroscopy (FTIR) of wood vinegar samples subjected to purification using different mass ratios of wood vinegar to coconut shell biochar is shown in the **Figure 4**. The ratios examined were 4:1, 2:1, and 1:1, alongside a control sample (raw wood vinegar). This analysis aimed to determine the efficacy of biochar in removing tar compounds by observing changes in specific functional groups.

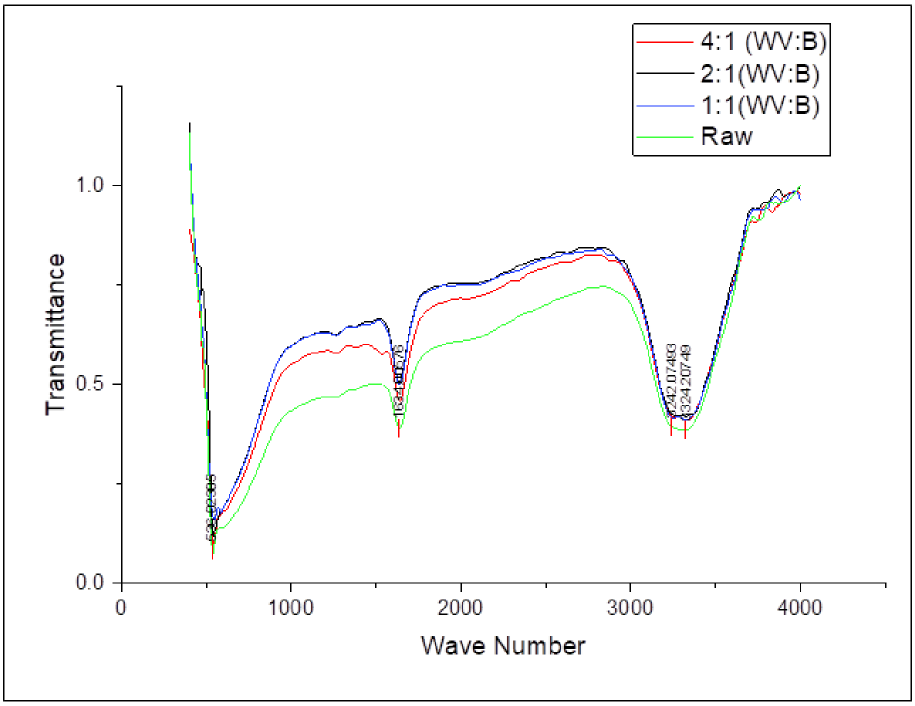

Figure 4. FTIR Results of Sample Set 2.

The broad peak at 3200–3550 cm^{-1}, associated with –OH groups from phenols and alcohols, shows a clear increase in transmittance in all biochar-treated samples, indicating their effective removal. This reduction suggests adsorption driven by hydrogen bonding and surface complexation between these oxygenated compounds and functional groups on the biochar surface.

Similarly, the peaks at 1566–1650 cm^{-1}, corresponding to aromatic rings, show higher transmittance for the 4:1 and 2:1 ratios, reflecting the removal of aromatic tars. This is consistent with π–π interactions between aromatic compounds and the graphitic domains of biochar, supported by hydrophobic interactions that favor adsorption of non-polar species. The limited difference between the 1:1 and 2:1 ratios indicates that increasing biochar beyond a certain level does not significantly improve adsorption.

3.3 Properties of Wood Vinegar

The set 1 focused on the effect of different biochar ratios in wood vinegar, maintaining a constant contact time of 3 days. The samples were treated with wood vinegar to biochar ratios of 4:1, 2:1, and 1:1. The values of the four samples under the following parameters are given in **Table 2**.

Table 2. Set 1 Data for Relevant Parameters.

Set 1 (8 °C, 3 days)				
Parameter	**Raw WV**	**WV:Biochar (4:1)**	**WV:Biochar (2:1)**	**WV:Biochar (1:1)**
pH Value	3.45	3.47	3.5	3.52
Absorbance	3.099	2.946	2.399	2.306
Refractive Index	1.338	1.3375	1.337	1.3365
Brix Value (°Bx)	0.35	0.34	0.3	0.25
Density (kgm^{-3})	1007.36	1006.97	1006.94	1006.86

The set 2 focused on the contact time of biochar in wood vinegar at a constant 1:1 ratio, examining samples treated for 1, 2, and 3 days. The values of the four samples under the following parameters are given in the **Table 3**.

Table 3. Set 2 Data for Relevant Parameters.

Set 2 (8 °C, WV:Biochar 1:1)				
Parameter	**Raw WV**	WV+Biochar (1 day)	WV+Biochar (2 days)	WV+Biochar (3 days)
pH Value	3.17	3.19	3.19	3.21
Absorbance	3.927	3.528	3.023	2.736
Refractive Index	1.338	1.3375	1.3372	1.337
Brix Value (°Bx)	0.35	0.32	0.3	0.27
Density (kgm^{-3})	1006.38	1006.198	1004.188	1003.911

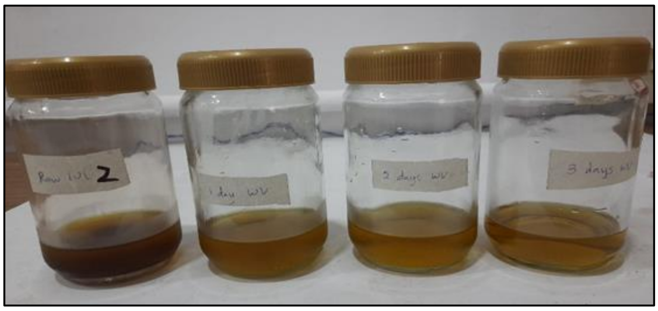

Figure 5. Color Change of Sample Set 2

3.3.1 pH

An acidic pH in the range of 3.17–3.52 was observed in the wood vinegar samples. According to the literature, wood vinegar produced from coconut shells at 300–400 °C typically exhibits a pH of 2.3–2.9 [17], [18]. The slightly higher pH values obtained in the present study are reasonable, given that the wood vinegar is diluted with water during condensation. Table 2 and Table 3 show a slight pH increase across samples, indicating a minor reduction in acidic components of wood vinegar. The maximum pH increases of only about 2% indicates no significant change in acidity. Although the high-water content (89.4%) resulting from condensation in a water medium can influence pH, even the hypothetical 100% wood vinegar sample shows minimal variation. This stability suggests that the biochar primarily adsorbs neutral and semi-volatile organic compounds, such as tar formed from polymerized phenolics, aldehydes, and ketones, rather than removing acidic constituents. Thus, the purification process selectively targets non-acidic impurities while leaving the acidic profile of wood vinegar largely unchanged.

3.3.2 Absorbance

The observed reduction in absorbance and shift from darker to lighter shades in the treated wood vinegar indicate effective removal of chromophoric (color causing) substances, such as phenolic compounds, aldehydes, and ketones, which are typically rich in tar and strongly absorb light. Table 2 and Table 3 show that increasing the biochar ratio enhances adsorption of these color-causing substances, producing a clearer vinegar. Extending biochar contact time further decreases absorbance, highlighting the combined effect of biochar proportion and residence time in adsorbing tars and aromatics. Maximum change of absorbance of 30% is a significant achievement. This optimization is essential for achieving higher purity and clarity in the final product. This finding is further evidenced by visual appearance of wood vinegar in **Figure 5**.

3.3.3 Brix Values

The observed decreases in Brix values of above Table 2 and Table 3 such as increasing the ratio of biochar and extending the contact time with wood vinegar suggests that biochar is effective at adsorbing dissolved solids, which are likely composed of high-molecular-weight tar compounds in the wood vinegar samples. Up to 20% change of brix value was observed which is a significant improvement. A direct correlation has been reported between the Brix value and the tar content of wood vinegar, indicating that higher tar concentrations correspond to increased Brix values [19]. Further, around 40% drop of yield during crude wood vinegar refining with charcoal has been reported due to removal of tarry components [20].

3.3.4 Other Properties

No significant change was observed in the refractive index and the density values across the samples. In literature, almost no difference in density and the refractive index has been reported after wood vinegar refinement with activated carbon. Reported values are very close to present study with a density of 1 g/cm^3 and refractive index of 1.3 [21].

4. Conclusion

This study demonstrated that coconut shell biochar is an efficient and cost-effective adsorbent for purifying wood vinegar. Both materials were produced via slow pyrolysis of coconut shells, ensuring economic viability through effective by-product utilization. The pretreated biochar enhanced adsorption efficiency, and analyses including FTIR, spectrophotometry, and measurements of pH, density, refractive index, and Brix values were performed. Results showed significant improvements in the chemical and physical properties of treated wood vinegar, with FTIR confirming the removal of undesirable organic compounds. Overall, coconut shell biochar proved to be a sustainable and effective medium for enhancing wood vinegar purity and quality. In future work, detailed characterization of both wood vinegar and biochar is expected to understand the adsorption mechanisms that can support optimization of the refining process. Additionally, techno-economic and environmental analysis of an integrated biochar production and wood vinegar purification process is suggested as important directions for future research.

Declaration of Competing Interest

The authors declare no competing interests.

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Keywords

Wood vinegar, Biochar, Adsorption, Purification, Pyrolysis, Coconut shell

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