ISSN 3030-783X

Comparative Study of One-Step and Two-Step Slow Pyrolysis of Bamboo in Inert and Oxidative Media

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1 Introduction

Global energy demand continues to rely heavily on conventional fossil fuels, despite growing environmental and sustainability concerns. Renewable alternatives, such as biomass, have emerged as a promising solution due to their abundance, carbon neutrality, and potential for energy and value-added product generation. Biomass encompasses organic materials derived from plant and animal residues, which can be converted into energy and chemicals through various thermochemical processes. Among these, pyrolysis, a thermal decomposition process conducted in the absence (or limited presence) of oxygen has gained significant attention for its ability to transform biomass into biochar, pyroligneous acid (wood vinegar), and syngas, each with distinct industrial and environmental applications [1], [2].

Slow pyrolysis, characterized by moderate temperatures (300–500°C) and prolonged residence times, favors biochar production, while fast and flash pyrolysis maximize liquid and gaseous outputs [3]. Recent studies have explored stepwise pyrolysis as a means to enhance product selectivity by employing intermediate isothermal stages. For instance, two-step slow pyrolysis has been shown to marginally alter yields compared to continuous methods [4], while combining slow and fast pyrolysis stages increased char yield in eucalyptus wood [5]. Further, stepwise collection of wood vinegar would yield two distinct fractions: one enriched with oxygenated compounds and the other with aromatic compounds, enhancing the versatility of its applications. Despite these advances, low temperature stepwise pyrolysis remains underexplored, particularly for specific feedstocks like bamboo.

Bamboo, a fast-growing grass with high biomass productivity, low ash content, and porous biochar characteristics, is an ideal candidate for pyrolysis [6], [7]. Its decomposition occurs between 200–500 °C, with hemicellulose degrading at 200–360°C and lignin at 360– 500 °C [6]. Most existing studies focus on pyrolysis in inert atmospheres (e.g., N_2) [7], [8], [9] leaving a gap in understanding oxidative pyrolysis, where controlled oxygen exposure (e.g., via flue gas) could reduce energy costs and enable industrial integration. Flue gas often contains trace oxygen and may influence pyrolysis kinetics through oxidation reactions. Successful oxidative pyrolysis could promote industrial symbiosis by repurposing waste heat in industrial flue gases, though its effects on product quality.

This study investigates stepwise slow pyrolysis of bamboo under inert (N_2) and oxidative (simulated flue gas) conditions, comparing one-step and two-step approaches. The research evaluates the impact of these parameters on biochar and wood vinegar yields and properties, while exploring potential applications and aims to advance sustainable biomass valorization strategies.

2 Experimental Section

2.1 Materials

For this study, Yellow Bamboo (Bambusa Vulgaris) was selected as it is one of the most common types of bamboo found in Sri Lanka. The bamboo stems were sourced from Kegalle area in Sri Lanka and approximately 24 months old at the time of harvesting. The Bamboo were processed and crushed into 5 mm – 10 mm pieces in size. The relatively larger sizes not only simulate a practically achievable size range in a commercial application, but also reduces the chance of combustion when performing oxidative pyrolysis. Before the experiment, samples were dried in an oven at a temperature of 105 °C for 24 hours to remove moisture.

To create the required inert experimental conditions inside the reactor, nitrogen gas is used. To create the simulated flue gas conditions, nitrogen with 3% vol oxygen concentration was used. It is not expected of a flue gas to contain a high amount of oxidative compounds. Also, 2-4% is an ideal concentration of oxygen to form sufficient amount of micropores [11].

2.2 Methods

The pyrolysis reactor used in this study (**Figure 1**) was a horizontal cylindrical reactor. The reactor consisted

of a nickel-chrome heating coil covered by a porcelain insulation. The heating chamber was insulated with K-wool and a temperature controlling system was available to regulate the process temperature effectively. Both the gas inlet and the reactor outlet were comprised of needle valves. The inside of the reactor consisted of platform that can house a tray where the biomass was placed on. The reactor had two openings in each side for gas inlet and outlet of which the outlet can be removed along with the connected pipe. The sample tray was removed and inserted through this opening as required. The set-up also consisted of a condensation unit. This unit featured an ice bath and a bottle with openings for condensable gas inlet and exhaust gas outlet. To successfully condensate pyroligneous acid, the bottle was filled with 200 ml of water in which the diluted pyroligneous acid would be condensed.

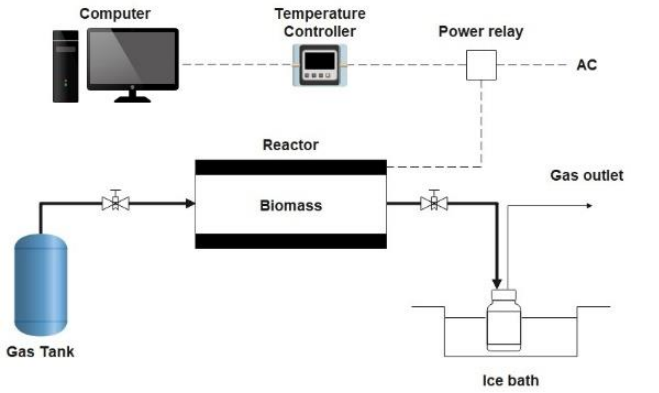

Figure 1. Reactor Setup.

2.3 Materials Characterization

Step temperatures and step residence time was determined for the steps wise and continuous pyrolysis processes. To identify the step temperatures, thermogravimetric analysis (TGA) data was used. It provides the thermal behavior of biomass by monitoring the percentage mass loss over time with increasing temperatures. According to Fabricio et al. 2022 [12] the differential thermogram (DTG) curves obtained from the TGA experiments for Bambusa Vulgaris, holocellulose (hemicellulose + cellulose) peaks can be observed at temperatures of 320.8 °C and 360.0 °C. These two temperatures were selected for the step temperature values as peaks in DTG curves represents the temperatures at which the mass reduction of the sample is at the highest. It is assumed that these temperatures would yield the most distinctive results within the temperature range considered. For the residence time during isothermal conditions, duration of 30 minutes was selected as it offers a balance between sufficient time for pyrolysis of the samples and practical applicability for potential commercial-scale processes.

2.4 Experiment Procedure

The crushed bamboo is weighed using a scale along with the sample tray. Approximately 10 g of bamboo is used per experiment. In this study, the experiment is started by heating the empty reactor to 160 °C at a rate of approximately 15 °C/min. Upon reaching 90 °C, nitrogen gas was introduced into the reactor at a flow rate of 100 mL/min by opening the gas valve, thereby establishing the desired reaction environment and purging the reactor of air. Once the reactor temperature reached 160 °C, the system was briefly opened to insert the sample tray. Simultaneously, the condenser setup was connected to the reactor. The temperature was then adjusted to the target pyrolysis temperature, as determined through thermogravimetric analysis (TGA). For single step pyrolysis, the set temperature was 360 °C. Once the temperature is reached, the reactor was kept at isothermal conditions for a residence time of 30 minutes. For two step-pyrolysis, the sample was kept under isothermal conditions for 15 minutes each at a first set temperature of 320 °C and a second temperature was 360 °C respectively. After the heating process is over, heating to the reactor was turned off, and the system was allowed to cool while maintaining nitrogen gas flow to prevent oxidative reactions. The biomass sample and the pyroligneous acid sample, at approximately 140 °C, was withdrawn, cooled, and weighed for analysis. Additionally, the mass and pH value of the acid sample were recorded.

2.5 Product Characterization

The yields of biochar (y_c) and wood vinegar (y_w) were determined based on the initial biomass weight using standard mass balance equations, while the yield of non-condensable gases (y_g) was calculated by difference.

$$y_c(wt\%) = \left(\frac{W_c}{W_b}\right) \times 100\% \qquad Eq(1)$$

$$y_w(wt\%) = \left(\frac{W_w}{W_b}\right) \times 100\% \qquad Eq(2)$$

$$y_g(wt\%) = 100 - y_c - y_w \qquad Eq(3)$$

Where W_c and W_w are measured weights of biochar and wood vinegar samples, respectively, and W_b is the initial weight of the bamboo sample.

Fourier Transform Infrared (FTIR) spectroscopy was performed over the range of 4000 – 500 cm^{-1} employing the KBr-tableting method for biochar and the ATR method for wood vinegar, to identify key functional groups and chemical characteristics of the produced materials.

3 Results and Discussion

3.1 Product Yields

The yields of the primary pyrolysis products (biochar, wood vinegar and pyrolysis gas) are summarized illustrated in **Table 1** and **Figure 2**. The results demonstrate that two-step pyrolysis produced almost equal biochar yield, higher wood vinegar yield and lower gas yield compared to one-step pyrolysis, under both inert (N_2) and oxidative (simulated flue gas) conditions. This observation aligns with prior studies on stepwise pyrolysis of various biomass feedstocks, which reported similar results [4], [9]. Notably, biochar yields were lower in oxidative pyrolysis than in inert pyrolysis across all tested scenarios. This reduction can be attributed to the presence of oxygen, which promotes partial combustion and oxidative degradation of the biomass. Under oxidative conditions, the thermal decomposition of lignocellulosic components (cellulose, hemicellulose, and lignin) is accelerated, leading to higher volatilization rates and consequently lower solid residue (biochar) formation.

Table 1. Product Yields.

Method		Yield		
		Biochar	Wood vinegar	Pyrolysis gas
Inert	One step	38.40%	33.59%	28.01%
	Two step	38.86%	36.34%	25.00%
Oxidative	One step	35.84%	32.99%	31.17%
	Two step	35.96%	37.61%	26.43%

3.2 Product Analysis

FTIR spectra of biochar and wood vinegar revealed peaks around 1600 cm^{-1} (C=C aromatic) and 3400 cm^{-1} (O–H stretching), suggesting the presence of hydroxyl-rich surfaces and aromatic structures [10]. These features of biochar enhance adsorption capacity for heavy metals (Pb, Cu, Ni, As) in water treatment [11] and ensure carbon stability in soils. Wood vinegar's alcohol and phenolic content also support biological herbicide potential [12].

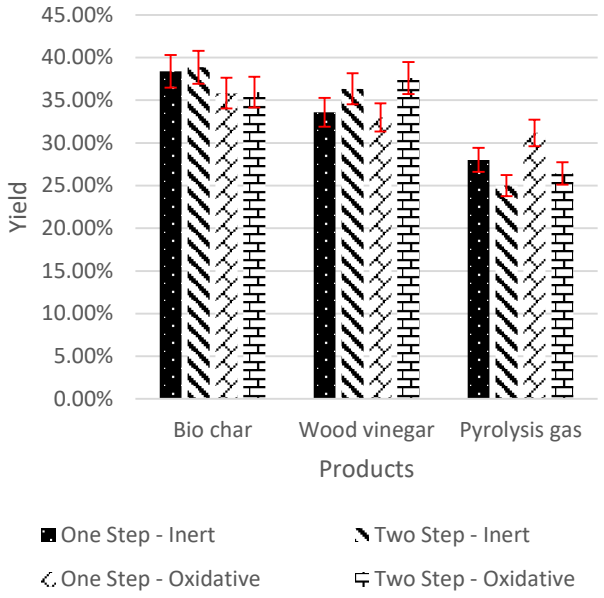

Figure 2. Comparison of Product Yields.

3.3 Energy Analysis

Energy analysis indicated that the two – step process utilize less energy compared to the conventional continuous process as the sample is kept at 320 °C and 360 °C for 15 minutes respectively, instead of full 30 minutes at 360 °C. This reduces the amount of energy utilized for biomass heating without compromising yield or quality, demonstrating energy-efficient scalability. It has been reported up to 33% saving in energy consumption due to two stage slow pyrolysis within the temperature range of 250-400 °C. The saving is greater when temperatures of two stages are far apart [13].

Furthermore, flue gas containing an approximately similar oxygen concentration can substitute the requirement for a dedicated inert gas such as N_2, while simultaneously supplying the necessary heat for the process with less energy requirement. This concept is further illustrated in the sample calculation provided below.

List of constants

- Biomass mass: m_b
- Temperature change: $T_f - T_0 = \Delta T$
- Biomass specific heat capacity: $c_{p,b}$
- Gas flow: Q

- Heating rate: r
- Gas production energy/m³: e_{N_2}
- Stoichiometric coefficient: v_i
- Standard formation enthalpy: $\Delta H_{f,i}^{\circ}$
- Density of purge gas: ρ

Compute heat-up time and total run time

Heat up time:

$$t_{\text{heat}} = \frac{\Delta T}{r} \qquad Eq(4)$$

Total run time (heat up time + isothermal step time):

$$t_{\text{total}} = t_{\text{heat}} + 30 \qquad Eq(5)$$

Sensible heating energy

Total gas usage:

$$V_{\text{gas,total}} = Q \times t_{\text{total}} \qquad Eq(6)$$

Sensible heating energy of purge gas:

$$E_{\text{gas-heat}} = V\rho C_p \Delta T \qquad Eq(7)$$

Sensible heating energy of the biomass:

$$E_{\text{biomass}} = m_b\, C_{p,b}\, \Delta T \qquad Eq(8)$$

Heat of reaction

$$\Delta H_{rxn} = \sum v_{i,products} \times \Delta H_{f,i}^{\circ} - \sum v_{i,reactants} \times \Delta H_{f,i}^{\circ} \qquad Eq(9)$$

Since the produced biochar and wood vinegar do not exhibit significant differences in their properties between the inert and oxidative studies, a substantial variation in reaction energy between the two cases is not expected. Therefore, the heat of reaction can be considered as a constant value for both conditions.

Energy requirement for gas production

$$E_{\text{gas}} = V_{\text{gas,total}} \times e_{N_2} \qquad Eq(10)$$

$$E_{\text{ox}} = V_{\text{gas,total}} \times e_{ox} \qquad Eq(11)$$

The energy required for gas production in both cases N_2 and simulated flue gas was found to be within a similar range (0.5-1.5 kWh/kg). Therefore, the gas supply energy does not introduce a significant difference in the experimental setup.

Total energy requirement

N_2 case:

$$E_{Total,N_2} = E_{\text{biomass}} + E_{\text{gas-heat}} + E_{\text{gas}} \pm \Delta H_{rxn} \qquad Eq(12)$$

Simulated flue gas case:

$$E_{\text{Total,ox}} = E_{\text{biomass}} + E_{\text{ox-heat}} + E_{\text{ox}} \pm \Delta H_{rxn} \qquad Eq(13)$$

Flue gas case:

$E_{\text{ox}} \rightarrow 0$: The gas is not produced for pyrolysis purpose. It is a waste from another activity.

$T_{0,Flue\ gas} > T_0$: Therefore, the sensible energy requirement for gas heating is reduced since the $\cdot(T_f - T_0)$ is being reduced.

Consequently,

$$E_{\text{Total, }N_2} > E_{\text{Total, flue gas}} \qquad Eq(14)$$

A significant difference in energy demand is not expected when using simulated flue gas in place of N_2. However, the overall energy requirement is expected to be lower when an actual industrial flue gas stream is utilized.

4 Conclusions

Stepwise oxidative slow pyrolysis of bamboo provides comparable or improved yields while reducing energy consumption relative to single-step process. The approach enhances product functionality and supports sustainable biomass valorization. The combination of mild oxidative conditions and two-step temperature control offers a cost-effective route for producing biochar and wood vinegar suitable for environmental remediation. This study demonstrates the feasibility of stepwise oxidative slow pyrolysis using simplified energy considerations. As the next step, the authors intend to perform detailed energy analysis.

Declaration of Competing Interest

The authors declare no competing interests.

Acknowledgements

Authors would like to acknowledge the Department of Materials Science and Engineering of the University of Moratuwa for providing FTIR analysis of biochar and wood vinegar.

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Y.B.P. Ranasinghe: Experimentation, Data Analysis, Visualization, Writing – Review & Editing.

Duleeka Sandamali Gunarathne: Conceptualization, Supervision, Writing – Review & Editing.

Keywords

slow pyrolysis, stepwise pyrolysis, oxidative pyrolysis, bamboo, biochar, wood vinegar

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