

Original Research Article

Development and Performance of Pyrolyzer for Production of Biochar and Wood Vinegar from Domestic Agriculture Farm Waste

Mohd Fazly Mail.^{1*}, Mohammad Hariz Abdul Rahman.², Nur Adliza Baharom³, Mohammad Shahid
Shahrin⁴, Mohd Ridzuan Mohd Daud⁵

¹Farm Mechanisation Program, Engineering Research Centre, Malaysian Agricultural Research and Development Institute (MARDI), Serdang, Selangor, Malaysia, fazlym@mardi.gov.my

²Climate Change Program, Agro-Biodiversity & Environment Research Centre, Malaysian Agricultural Research and Development Institute (MARDI), Serdang, Selangor, Malaysia, hariz@mardi.gov.my

³Pest and Disease Management Program, Horticulture Research Centre, Malaysian Agricultural Research and Development Institute (MARDI), Serdang, Selangor, Malaysia, nuradliza@mardi.gov.my

⁴Soil Science and Water Program, Soil and Fertiliser Research Centre, Malaysian Agricultural Research and Development Institute (MARDI), Serdang, Selangor, Malaysia, shahid@mardi.gov.my

⁵Agronomy & Production System Program, Horticulture Research Centre, Malaysian Agricultural Research and Development Institute (MARDI), Serdang, Selangor, Malaysia, ridzone@mardi.gov.my

*Corresponding author: Mohd Fazly Mail, Farm Mechanisation Program, Engineering Research Centre, Malaysian Agricultural Research and Development Institute (MARDI), Serdang, Selangor, Malaysia; fazlym@mardi.gov.my

Abstract: This invention describes the development and performance of a pyrolyzer to produce biochar and wood vinegar from domestic agriculture farm waste. Two pyrolyzers (fix and portable) were designed and developed in MARDI. The system consists of a kiln with ignition area and feeder, cyclone area connected with a 7.5 cm diameter pipe, two vinegar collectors and temperature data logger. The system is capable of producing two main products, biochar and wood vinegar. The main heating materials are coconut shells and other pruning wastes such as mango and carambola, burned in the kiln under limited oxygen condition. Wood vinegar is a by-product of charcoal production. The design concept is to force the gases produced into the liquid through condensation. Theoretically, the smoke from the burned pruning wastes would rise. The gases will then enter the 7.5 cm pipe. At this stage, the pipe forces the gases to hit each other and become liquid. There is a cyclonic system to optimise the conversion of gas into a liquid. At the bottom of the system, there is one connected pipe to collect wood vinegar. An additional line is connected to the cyclone system to force the remaining gases through a smaller tube to become liquid. Overall, there are two vessels to collect the vinegar. After the functional test run, the results showed that the prototype gave satisfactory results with biochar products with 60 to 70% carbon with additional wood vinegar products suitable for eventual use. The study recommends pyrolyzation of waste as one of the sustainable approaches to manage abundant agricultural residues.

Keywords: pyrolyzer; biochar; wood vinegar; pruning wastes; limited oxygen

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1. Introduction

The growing awareness about environmental issues has led to the search for new technologies that are green and sustainable (De Sousa Jabbour *et al.*, 2018). Several technologies were proposed, which include the potential use of biomass as promising sources of carbon-neutral and recyclable products (Phitsuwan *et al.*, 2013). Additionally, several possible techniques have also been suggested and put in practice, including biomass conversion into bio-energies (Chisti, 2008), organic composts (Barton *et al.*, 2008) and biochars (Lin & Hwang, 2009).

Recently, increased attention has been focused on the thermochemical conversion of biomass into biochar. Biomass thermochemical conversion is a process such as pyrolysis where materials are burned under limited or zero-oxygen conditions (Sohi *et al.*, 2010). This conversion technique is unique as it produces biochar (charcoal) in addition to biofuel (Inyang *et al.*, 2010).

Agricultural residues with solid characteristics, such as fruit stones and nutshells, are very good precursors for biochar and bio-oils production (Demirbas *et al.*, 2006). In Malaysia, coconut shells, a residue from the coconut processing industry, are highly abundant and suitable for biochar production. Conventionally, biochar from coconut shells was produced using a standard size drum pyrolysis (Hariz *et al.*, 2015). However, advanced methods could be developed and improved mechanically to improve efficiency.

Biochar represents a potential route to achieving both sustainable environment development and optimising product effectiveness. The products (biochar) are mainly used for agricultural activities, such as mixing media for fruits and vegetable cultivations. The compound is highly adsorbent and may increase its ability to retain water, nutrients and agricultural chemicals, preventing water contamination and soil erosion (Mullen *et al.*, 2010). In addition to this, an efficient design for biochar production could also produce wood vinegar as secondary products from trapping and condensation of the gas (smoke) as a by-product (Baharom *et al.*, 2020). Wood vinegar was reported to exhibit a high degree of antimicrobial activity against various microorganisms. However, its chemical composition and bioactivity intrinsically depend on which plant material is charred (Baharom *et al.*, 2020; Yang *et al.*, 2016).

The present study attempted to design and develop a pyrolyzer to produce biochar and wood vinegar from domestic agriculture waste. These include the design and preliminary evaluation of the efficiencies of the system.

2. Materials and Methods

2.1 Design Requirement

The prototype machine has to fulfil several requirements elaborated below. Of these, two requirements, dual-process carbonisation: endothermic and exothermic and pyrolysis treatment system that chemically decomposes organic materials by heat in the absence of oxygen, were given prominence in the machine's design consideration.

2.1.1 Construction

A pyrolyzer is like a kiln for thermal decomposition of carbonaceous material in the absence of or under limited oxygen supply. The purpose is to construct both fixed and portable type of pyrolyzer, in which both prototypes share desirable design concept of a simple yet robust built, capable to withstand up to 800 °C heating temperature, should weight no more than 100 kg and requires only a single operator to run the machine.

2.1.2 Function

The pyrolyzer should be capable of running a pyrolysis of dual carbonisation, which are endothermic and exothermic, to produce a high-quality charcoal from coconut shell, carambola and mango wood as well as wood vinegar. These prototypes also should consist of a cyclonic system in order to produce more wood vinegar and also some additional functions that allow the wood vinegar to be collected at the end of the system.

2.1.3 Costs

The pyrolyzer should not cost more than RM 7,500 for portable type and RM 15,000 for a fix type unit ex-factory, inclusive of 30% profit for the fabricators.

2.2 Configuration

The pyrolyzer design concept (Figure 1) followed a self-heating process, earlier studied by Hariz *et al.* (2019). Energy from initial burnings and flame is taken in by endothermic reactions followed by an exothermic reaction. Exothermic reactions transfer

power to the adjacent biomasses, and the temperature increases. Its causes a chain reaction which converts the biomasses into charred (biochar) materials.

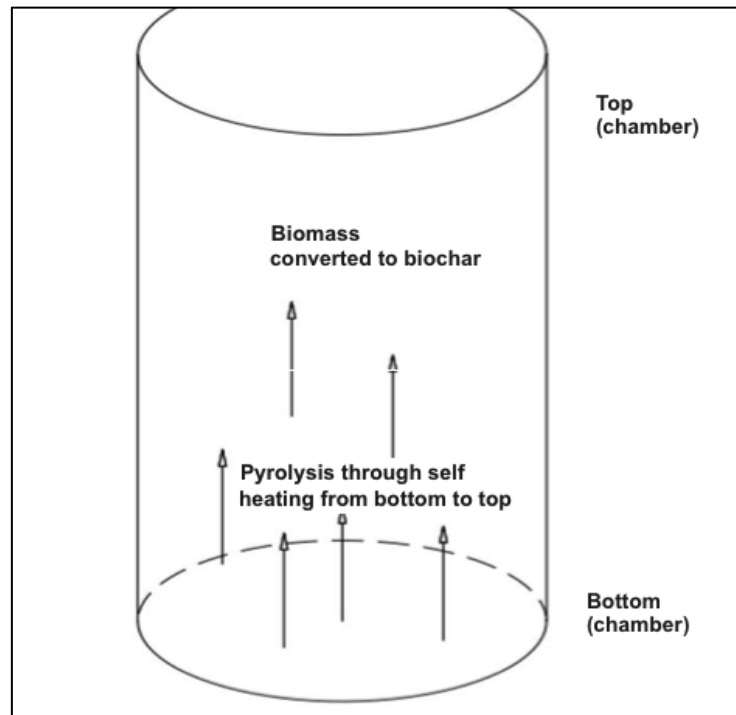
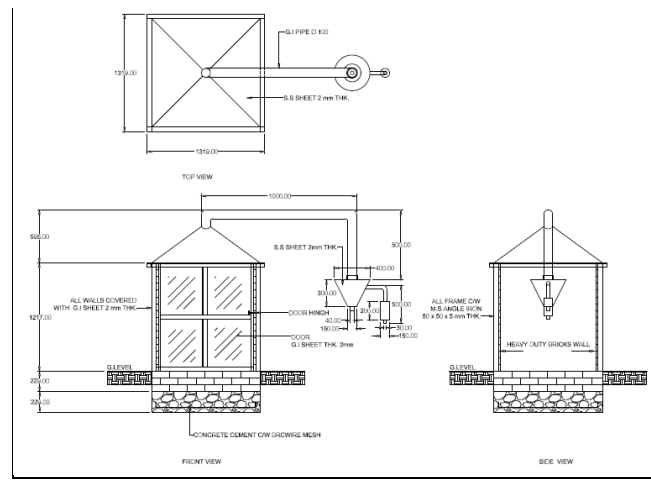


Figure 1. The design concept of the pyrolyzer based on Hariz *et al.*, (2019).

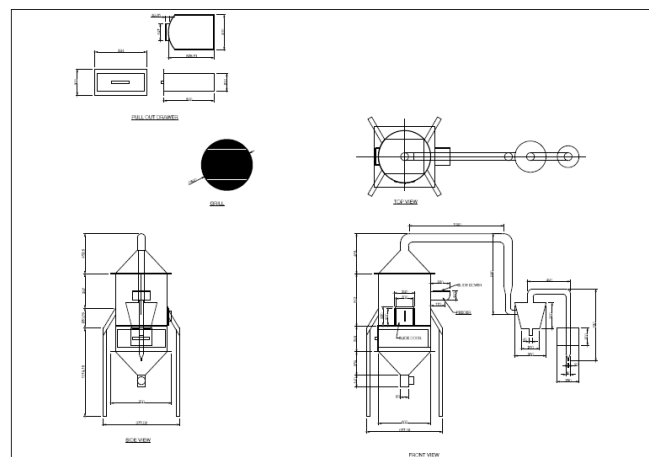
Conventionally, the drum pyrolyzation of biochar from coconut shells was used to produce biochar. However, the smoke resulting from combustion was released into the air. That was a huge loss as the free smoke can be trapped into a good wood vinegar for agricultural purposes. The gas is light and is known to rise upwards. Therefore, a conical rooftop needs to be designed and ensure that the combustion smoke is forced through a small cylinder. When the smoke is forced through the cylinder, the gas accumulates and will produce a liquid. Using this principle, the size of cylinders was reduced from one point to another until the wood vinegar outlet was collected.

This invention describes the design and performance of a kiln to produce biochar and wood vinegar from domestic farm waste. Two kilns that are fix and portable (Figure 2) were designed and developed in MARDI. It consists of a kiln with an ignition area and a feeder, a cyclone area connected with a 7.5 cm diameter pipe, two vinegar collectors and a temperature data logger. The system is capable of producing two main products; biochar and wood vinegar. The main heating materials are the farm and pruning wastes such as coconut shell, mango and carambola burned in the kiln under limited oxygen. Wood vinegar is a by-product of charcoal production.

The fixed pyrolyzer type required a square 1319 mm x 1319 mm x 229 mm thickness concrete cement with reinforcement steel mesh and bricks wall for ground, 1319 mm x 1319 mm x 1217 mm height heavy duty bricks wall and 2 mm thick galvanised iron sheet for covering an outer wall. All frames including the door with hinges were made from mild steel angle iron, 2 mm thickness stainless 1319 mm x 1319 mm x 595 mm height steel rooftop, galvanised iron pipe for smoke channel 100 mm diameter, a cyclonic system for smoke and vinegar recovery. Stainless steel material was selected as the main body structural for the portable pyrolyzer type. The mechanical properties in specific temperature ranges at which the melting points were between 1375 to 1530 degrees Celsius and corrosion resistance, and product cost can all influence stainless steel selection (Beddoes, J *et al.*, 1999).



(a)



(b)

Figure 2. Configuration and main components of the prototype pyrolyzer: (a) Fix type pyrolyzer; (b) Portable type pyrolyzer.

2.3 Data Collection

One kg of coconut shell was placed in the burning tray and lighted. The flaming tray was inducted into the burning chamber. Ten kg of coconut shell were placed in the pyrolysis chamber. There is a window feeder at the pyrolysis chamber. Four temperature reading points at the burning chamber and pyrolysis chamber were taken around the kiln every five to 20 minutes. A temperature data logger (thermocouple) was built to record the temperature (Figure 3).

The developed temperature data loggers used a microprocessor, an internal memory for data storage, and a sensor to collect data. The logger was a small and battery-powered device. The data logger interfaces with a computer to view and analyse the collected data. Using an electronic data logger was much more effective, accurate and reliable than taking periodic manual readings.

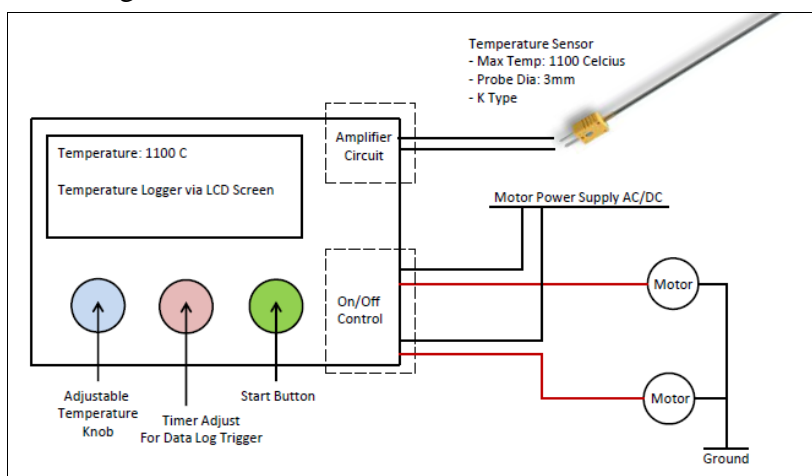


Figure 3. A temperature data logger.

2.4 Characterisations of Biochar

Characterisations of biochar were carried out to determine the chemical contents of the biochar. The analysis was carried out using Elemental Macro Cube. The elements analysed were carbon (C), hydrogen (H), nitrogen (N) and sulphur (S). Prior to the analysis, the biochar (charred) samples were first crushed, grounded and prepared in an appropriate amount in triplicate samples before continuing with elemental analysis procedures.

2.5 Characterisation of wood vinegar

The chemical compound of wood vinegar was analysed using GC-MS (PerkinElmer Clarus 600, USA) using the headspace technique. An amount of 10 mL of wood vinegar sample (coconut shell, carambola and mango) were placed in a 30 mL glass vial

(5cmID×10.0cmH). A 250µL gastight microsyringe (Hamilton® GASTIGHT® syringe 1825) was rinsed at least five times with acetone and thereafter with another five more times with air. The needle tip was later inserted into the vial, followed by the plunger's repeated drawing five times. The final volume was at 250 µL and was quickly inserted into the injector port. The samples were injected into a single taper liner with an internal volume of 1200 µL. The samples were then analysed using a 1 mL/min constant flow DB-5ms capillary column (Agilent Technologies, NEW Castle, DE, U.S.) (30 m x 0.25 mm, 0.25 µm film thickness) with an inlet temperature of 200 °C, and the temperature of the transition line mass spectrometry of 300 °C and M.S. source temperature of 200 °C. (Yang *et al.*, 2016).

2.6 Performance Of Wood Vinegar Against Selected Phytopathogens

2.6.1 In vitro screening

Agar well diffusion technique was used to screen for the antifungal behaviour of wood vinegar in a combination coconut shell, mango and carambola against *Colletotrichum gloeosporioides*, *Fusarium oxysporum*, *Pestalotiopsis microspora* and *Pythium aphanidermatum*. Three treatments were assessed in this experiment: Sterile distilled water (T1), wood vinegar (T2) and commercial fungicide (T3). In the centre, each PDA plate was inoculated with a 6 mm disc and 7-day-old isolates. Four wells with a diameter of 4 mm were produced, 1 cm apart, surrounding the fungal disc. Then, 50 µL of respective treatments were added to each well. The plates were then left undisturbed to allow diffusion of the wood vinegar into the agar and incubated at 25 °C until the control fungal growth reached the plate edge. The experiment was arranged in a completely randomised design with triplicates. The fungal colony's mean diameter was measured in mm at 7 and 20 days after incubation as proposed by Jackson *et al.* (1991) with a slight modification. The percentage of inhibition reduction was measured using the following equation (1):

$Rr = \frac{[(R1 - R2)]}{2} \times 100$	(1)
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Where;

Rr = the percentage of inhibition reduction in colony diameter,

R1= the colony diameter on the untreated medium (mm) as a negative control, and

R2= the colony diameter on the treated medium (mm).

Data collected were subjected to analysis of variance (ANOVA) by Statistical Analysis System (SAS 9.0). Means were compared using Duncan Multiple Range Test (DMRT) at $p \leq 0.05$.

2.6.2 *In vivo* screening

Chili fruits (Kulai 461, SAKATA) were sprayed with wood vinegar with 10^6 cfu/mL spore suspension of *C. gloeosporioides* before inoculation as described by Dilani *et al.* (2019). The tested wood vinegar was compared with commercial wood vinegar for positive control and sterile distilled water as a negative control. Disease assessments were carried out at 3 and 9 days after inoculation, respectively, using disease score rating developed by Montri *et al.* (2009). Disease severity was calculated using the following equation (2):

$$\text{Disease severity (\%)} = \left[\frac{\sum (r \times Nr)}{R \times n} \right] \times 100\% \quad (2)$$

Where;

r = the rating value,

Nr = the number of infected leaves with a rating of r , and

R = the value of the most serious disease severity, and n is the total number of tested plants.

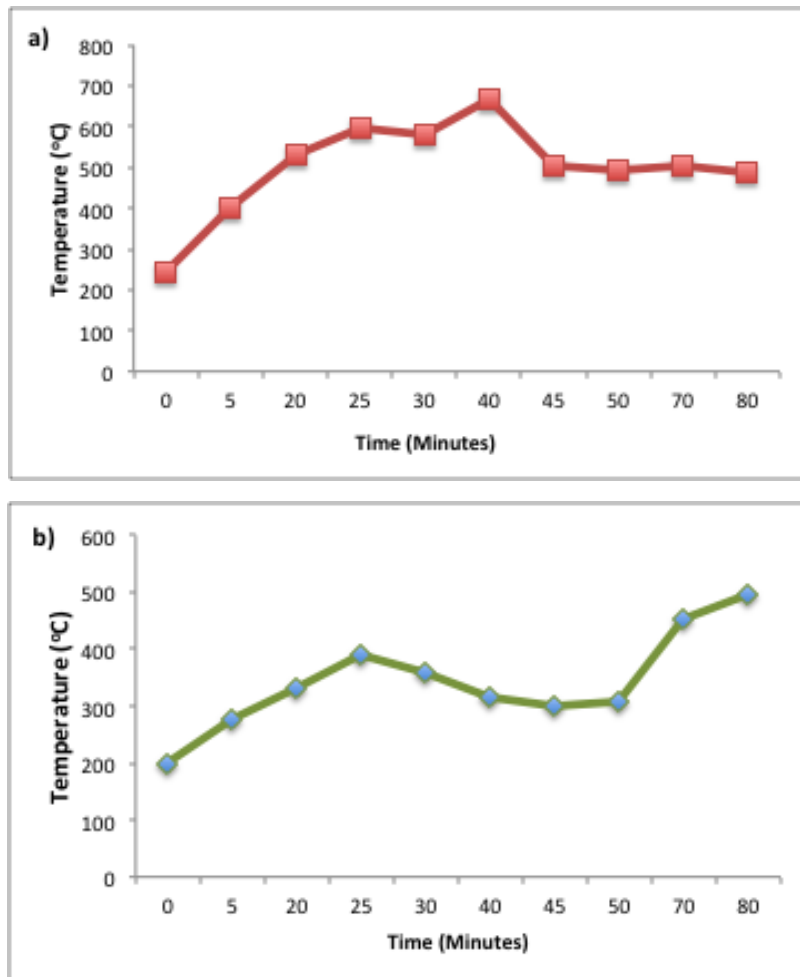
3. Results and Discussions

3.1. Functional Performance

The two types of pyrolyzer constructed in MARDI Jelebu (fixed) and MARDI Serdang (portable) as shown in Figure 4. Data collected for temperature profiles throughout the pyrolysis process were depicted in Figure 5 (a-c) using samples from coconut shells. The sampling points consisted of the lower part, middle part and upper part of the kiln. A complete pyrolysis process for coconut shells requires approximately 80 minutes to complete a cycle. The white smoke in the condensation area's sudden disappearance occurred with a sudden rise in temperature within the chamber. In order to terminate the process, water will be sprinkled inside the chamber to end the pyrolysis process and thus abruptly decreased the temperature. This situation can be observed in Figure 5c. At the end of the process (80 minutes), the pyrolysis temperature was high at 476 °C before the sudden observation of white smoke's disappearance. It was an indicator that within a short moment, the chamber should be extinguished by sprinkling water. Otherwise, the charred materials will continue to slowly burn themselves (under limited oxygen supply) and reduces the biochar's quality.



Figure 4. The constructed prototype of pyrolyzer: (a) Fix type pyrolyzer; (b) Portable type pyrolyzer.



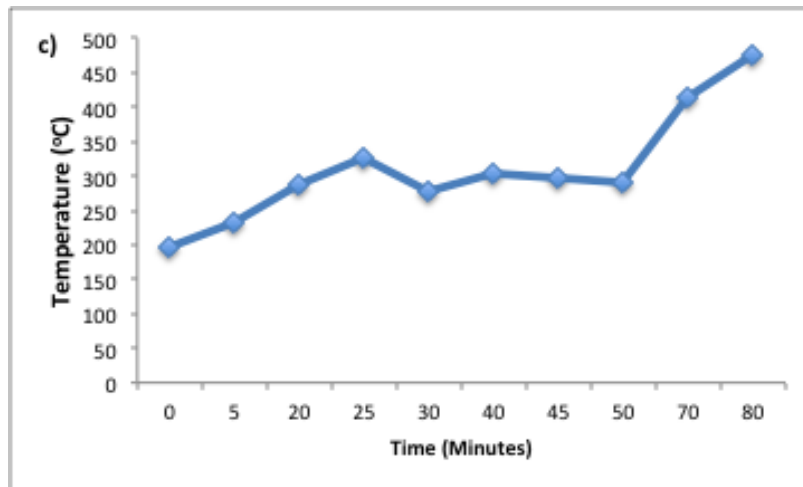


Figure 5. Temperature distribution around the kiln: (a) lower part of the kiln, (b) middle part of the kiln (c) upper part of the kiln.

Table 1 shows the temperature distribution at the lower part, middle part and upper part of the kiln. The maximum temperature at the lower part was 667 °C after 40 minutes. In contrast with the middle kiln, the maximum temperature was 495 °C after 80 minutes (at the end of the process). At the upper kiln, the maximum temperature was 476 °C. However, it was found that the highest temperature was at the middle of the process (40 minutes) followed by the middle part, which increased in temperature towards the end. The increasing temperature at the central part indicated that the pyrolyzation process was ongoing. The system was able to withstand and maintained high temperatures continuously.

Table 1. Distribution of temperature at the lower, middle and upper part of the kiln.

Time (Minutes)	Temperature Distribution Temperature (°C)		
	Bottom	Middle	Top
0	242	200	195
5	400	275	233
20	530	331	288
25	595	392	325
30	580	357	278
40	667	317	305
45	502	301	298
50	495	306	291
70	505	453	414
80	489	495	476

The cost to fabricate one unit of the both portable and fix pyrolyzer prototype was estimated (Table 2). The kiln was expected to be retailed at an estimated ex-factory price of

RM xxxx/unit which was inclusive of the manufacturer's overhead cost and 30% profit. The estimates were based on 2018 costs.

Table 2. Estimated costs of manufacturing one unit of the prototype pyrolyzer.

Cost component	Portable Pyrolyzer	Fix Pyrolyzer
	Cost (RM)	
Material and parts	3840	8275
Direct labour (@RM 80/man-day)	560	1120
Overheads (@1 x direct labour cost)	560	1120
Manufacturing cost	4960	10515
Profit (@ 30% of manufacturing cost)	1488	3154.5
Ex-factory retail price	6448	13669.5

3.2. Characterisations of Biochar

Table 3. Characterisation of biochar products.

Biochar Source	Mango pruning	Carambola pruning	Coconut shells
C (%)	74.00±2.99	62.93±4.85	73.81±21.32
H(%)	3.13±1.21	3.26±1.22	3.51±0.82
N (%)	0.70±0.07	0.36±0.06	0.30±0.08
S (%)	0.26±0.17	0.56±0.10	0.37±0.06

Table 3 highlights the elemental analysis results on the samples of biochar produced using the conceptual design. The results fulfilled one of the criteria for biochar characterisations considered to have more than 50% (European Biochar Certificate, 2012). Meanwhile, percentages of hydrogen were higher than nitrogen, while the lowest detected was the sulphur results. Given the low nitrogen rate available in biochar, it is deemed not suitable for direct use as fertiliser but rather suitably evaluated as a soil conditioner.

Biochar can be compared to compost when it comes to its functional use as a soil conditioner. The only difference is that compost has slightly lower in terms of carbon content. Comparison with local compost made up of straw and livestock waste, as studied by Hariz *et al.* (2013), showed that compost contains carbon percentage at approximately 15%. Therefore, it was suggested that biochar could retain more carbons in the final products.

Another benefit of biochar is that it could improve nutrient-holding capacity in soil. Another study by Hariz *et al.* (2015) suggested that coconut shell biochar has a better ability to retain nitrogen. This finding was attributed to its better surface area characteristics. A better adsorption capacity could make biochar as potential materials for mixing with fertilisers.

3.3 Chemical Compounds of Wood Vinegar

The information on wood vinegar's functional chemicals (Table 4) was extracted as a summary from Baharom *et al.* (2020). GC-MS analysis of wood vinegar produced from coconut shells, mango pruning and carambola pruning has identified several similar compounds such as phenol, furfural and acetic acid. In addition, other compounds identified were benzofuran, hexanal, ethanone and formic acid in coconut shells. The carambola compounds were imidazole, 3-pyridinecarboxaldehyde, benzaldehyde, benzofuran, indene, indazole, naphthalene, cyclohexanecarboxylic acid, palmitamide, palmitic acid, heptadecanenitril and sterylamide. The compounds in mango-based vinegar pruning consist of toluene, imidazole, annulene, benzaldehyde, carbamic acid, naphthalene heptadecanenitril and stearylamine. The findings showed that wood vinegar from different plants differs in composition. The variation in the chemical composition of wood vinegar could be affected by temperatures and various parameters, such as wood species, wood humidity and combustion time. (Mohan *et al.*, 2008; Omulo *et al.*, 2017). Therefore, it is important to develop an efficient pyrolyzer prototype and standardise mechanism for a better quality of wood vinegar production.

Table 4. Chemical compounds of wood vinegar analysed by GC-MS (Summary from Baharom *et al.*, 2020).

No.	Retention time(min)	Source		
		Coconut shells	Carambola	Mango
1	3.12	nd	nd	toluene
2	4.04	furfural	furfural	furfural
3	4.10	nd	imidazole	imidazole
4	4.88	nd	nd	annulene
5	4.91	nd	3-Pyridinecarboxaldehyde	nd
6	6.11	nd	benzaldehyde	benzaldehyde
7	6.21	nd	nd	carbamic acid
8	6.22	phenol	phenol	phenol
9	6.58	benzofuran	benzofuran	nd
10	7.34	nd	indene	nd
11	7.48	acetic acid	acetic acid	acetic acid
12	7.59	formic acid	nd	nd
13	7.967	ethanone	nd	nd
14	8.14	hexanal	nd	nd
15	8.25	nd	indazole	nd
16	9.62	nd	naphthalene	naphthalene
17	12.69	nd	cyclohexanecarboxylic acid	nd
18	18.16	nd	palmitic acid	nd
19	20.17	nd	heptadecanenitril	heptadecanenitril
20	20.91	nd	palmitamide	nd
21	23.10	nd	stearylamine	stearylamine

*nd: not detected

3.4 Antimicrobial Effect of Wood Vinegar

Table 5 showed that wood vinegar significantly inhibited *C. gloeosporioides*, *F. oxysporum*, *P. microspora*, *P. aphanidermatum* as compared with other treatments at 100% reduction. *In vivo* screening of wood vinegar against *C. gloeosporioides* also showed promising results where wood vinegar was able to significantly reduced infection severity by 66.00% with the disease score scale of 2.70 (Table 6). This finding actively demonstrated that wood vinegar promotes antifungal properties against plant pathogenic microorganisms. Baharom *et al.* (2020) recently revealed that wood vinegar from coconut shell, mango pruning and carambola pruning has an excellent ability to suppress phytopathogens such as *C. gloeosporioides*, *F. oxysporum*, *P. microspora*, *P. aphanidermatum* and *Ralstonia solanacearum*. Antimicrobial effects showed by wood vinegar were due to its high acidity (pH: 2-3) and the presence of several compounds such as phenol, furfural and acetic acid that have been reported to play major roles in antimicrobial activity (de Souza Araújo *et al.*, 2018). It could also be due to other compounds that later form a synergistic effect in inhibiting various pathogenic microorganisms efficiently. Thus, it is possible to recommend wood vinegar as a cure for disease control.

Table 5. Antimicrobial activity of wood vinegar against selected phytopathogens.

Treatment	Description	Inhibition reduction (%)			
		<i>C. gloeosporioides</i>	<i>P. microspora</i>	<i>F. oxysporum</i>	<i>P. aphanidermatum</i>
T1	Control	0.0 ^e	0.0 ^e	0.0 ^b	0.0 ^b
T2	Wood vinegar	100.0 ^a	100.0 ^a	100.0 ^a	96.4 ^a
T3	Chemical fungicide (a.i:mancozeb)	65.3 ^b	78.6 ^b	nt	nt

*nt: not tested

Table 6: *In vivo* performance of wood vinegar against *C. gloeosporioides*.

Treatment	Disease score scale *	Mean lesion (mm)	Disease severity (%)	Disease reduction (%)
Control	4.00	12.43	1.33	0.00
Wood vinegar (coconut shell, mango and carambola pruning)	2.70	3.27	0.67	66.00
Commercial wood vinegar	3.67	4.46	1.22	8.27

* Average score rating

4. Conclusions

The study recommends pyrolyzation of waste as one of the sustainable approaches to manage abundant agricultural residues. The pyrolyzer prototype had satisfied all design requirements, and objectives set out at the beginning of the study. The prototype's use could

considerably trap the combustion smoke from being released into the air and be converted into good wood vinegar for agricultural purposes besides having the biochar as the main product. The results suggested that biochar could retain as high as 60% to 70% of the final products' carbon contents. There were also co-products in the form of wood vinegar with potential antimicrobial effects. Further study will be carried out to determine the biochar and wood vinegar efficacies for specific agricultural use.

Author Contributions: Mohd Fazly Mail contributed to conceptualisation, methodology, software, data curation, and writing — original draft preparation, review and editing the manuscript. Mohammad Hariz Abdul Rahman was involved in conceptualisation, methodology, data curation, writing, review and editing the manuscript, supervision and project administration. Nur Adliza Baharom and Mohammad Shahid Shahrin supported data curation, formal analysis, writing and editing the manuscript. Mohd Ridzuan Mohd Daud was responsible for resources and data curation.

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