Research and Development of Rice Husk Recycling Technology for Agricultural Utilization in Taiwan

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Abstract

To meet the food demand of increasing human population with the planet's limited resources has been a challenge for globe food supply. With the use of synthetic fertilizers and pesticide, the yield of agriculture has massive grown, giving a hope to food demand problem. However, the application of synthetic fertilizers and pesticide has shown side effect on environment. The sustainable development of agriculture has become an important issue due to food supply challenge.

Circular agriculture, a concept of circular economy applying in agriculture, aims to reduce agriculture waste and make use of it into valuable products. Circular agriculture starts with restoring soil fertility as well as limits the loss of nitrogen and water in soil. Furthermore the most important thing is to minimize the input of chemical fertilizer as well as output of harmful substances and waste.

In Taiwan, rice is Taiwan's most valuable crop. Rice straw and rice husk are the main agriculture wastes of paddy cultivation. Rice straw can be buried in the soil by agricultural machinery. But rice husk, a by-product of rice milling, is used as bedding, growing media and fuel of which has not been effectively used and non-valuable.

Pyrolysis, an ancient technology, is a thermal conversion process of agriculture waste in the absence of air or oxygen leading to the production of biochar and pyroligneous acid (PA). There has been a lot of research on the application of biochar and pyroligneous acid in agriculture due to their potential of replacing chemical fertilizers and pesticides.

In this research, a new technology of biochar reactor has been revealed of which named as Charcoal processing system with internal combustion furnace (CPSICF). Each time the furnace could produce about 14 kg of rice husk biochar (RHB) and 14 kg of rice husk vinegar (RHV) from 40 kg of rice husk. It is confirmed that high-quality RHB and RHV can be produced at gate width 5 and 7.5 mm.

Furthermore, the high-quality RHB and RHV were used as soil amendment and plant promoter, respectively. The efficiency was carried out thorough pot experiment. The ratio of RHB in soil was 0 % at Control; 2, 4 and 6 % at B2, B4 and B6, respectively. RHV was spraying on above-ground part of the crop by non-use at Control, once at V1, twice at V2 and three times at V3. The analysis method for both RHB and RHV

experiment was: 1. Plant biomass amount, 2. Plant nutrition and 3. Sensory test. Soil property was studied for RHB experiment.

In plant biomass amount, the application of RHB revealed a significant effect but RHV on showed effect on some analysis items. In both RHB and RHV experiment, nitrite and vitamin C content was not detected in plant nutrition. Dietary fiber didn't reveal a significant difference with the application of RHB and RHV. Both RHB and RHV could decrease nitrate content which has become an important quality indicator due to the toxicity to human health. In sensory test, there was no significant relationship between Control and the application of RHB and RHV in sensory test either fresh or cooked. RHB revealed a significant effect on soil physical properties. But there was no significant relationship between RHB application ratio and element content in soil. The above results showed RHB as a soil amendment can promote plant growth, decrease nitrate content and change soil physical properties. The application of RHV showed a potential as plant promoter and decrease nitrate content in plant.

Keywords

Sustainable development Circular agriculture Rice husk Pyrolysis Biochar Pyroligneous acid Rice husk biochar Rice husk vinegar

Abbreviation and Nomenclature

- ANOVA: analysis of variance
- COA: Council of Agriculture, Executive Yuan
- CPSICF: Charcoal processing system with internal combustion furnace
- EC: Electrical conductivity
- HPIC: High performance ion chromatograph
- ICP-OES: Inductively coupled plasma atomic emission spectroscopy
- LSD: Fisher's protected least significant difference test
- NPUST: National Pingtung University of Science and Technology
- PA: Pyroligneous acid
- RCBD: Randomized complete block design
- RHB: Rice husk biochar
- RHV: Rice husk vinegar
- SAR: Sodium-Absorption Ratio
- SEM: Scanning electron microscope
- SOM: Soil organic matter
- T&H: Temperature and humidity
- TLUD: Top-lit updraft gasifier
- TOC: total organic carbon

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CHAPTER 1

Introduction

1.1 Research Background

As people are concerned with the quality of life, environmental issues are getting more and more attention. The problems of climate change came to light, leading to the Kyoto Protocol in 1997 and the Paris Agreement in 2015, and the idea of sustainability was born based on three pillars: economy, society, and the environment. Pressure to increase food supply and agricultural production has risen significantly due to population growth. Therefore, food supply chains, energy production and waste management are some of the main challenges today.

In agriculture, soil health is the most important foundation of a healthy farm ecosystem. Due to subtropical climate, soil is severely affected by weathering and leaching; especially short period heavy rainfall in Taiwan. In the past decades, agriculture has heavily relied on the effectiveness of synthetic fertilizer. Excessive fertilizer input in agriculture not only causes waste, but also degrades the soil due to contractual agricultural production.

Additionally, monocropping, an agricultural practice of growing a single crop year after year on the same land, can degrade soil over time. Soil degradation can be defined in the following ways: (1) physical degradation: due to erosion, compaction and crusting; (2) chemical degradation: associated with nutrient mining and acidification; (3) biological degradation: associated with loss of organic matter; and (4) deterioration of drainage conditions causing waterlogging or salinization (Food and Agriculture Organization of the United Nations, 1994).

Taiwan is an island country in East Asia. The main island, known historically as Formosa, makes up 99 % of the area controlled by the Taiwan, measuring 35,808 km² that is approximately equal to Kyushu, Japan. The exact location is $121^{\circ} 59' \text{ E}-120^{\circ} 01'$ E and $21^{\circ} 53' \text{ N}-25^{\circ} 18' \text{ N}$.

Fig. 1-1 is the satellite photo of Taiwan. The main island is a tilted fault block where two thirds of the area consists of rugged mountain in the east and one third of the area consist of flat to gently rolling plains in the west in which most of the Taiwanese population reside. The island of Taiwan lies across the Tropic of Cancer, and the East

Asian Monsoon influences its climate. Northern Taiwan has a humid subtropical climate, with substantial seasonal variation of temperatures, while parts of central and most of southern has a tropical monsoon climate where seasonal temperature variations are less noticeable with temperatures typically varying from warm to hot. The main island is struck by an average of four typhoons yearly. The summer monsoon (from May to October) accounts for 90 % of the annual precipitation in the south, but only 60% in the north. The average rainfall is approximately 2,600 mm per year (The Republic of China Yearbook 2014).

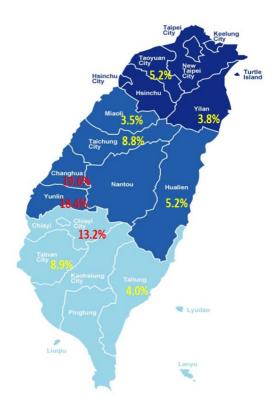
1.2 Purpose of Research

Agriculture is one of the main industries in Taiwan. It contributes to the food security, rural development and conservation of Taiwan. Council of Agriculture, Executive Yuan (COA) is the competent authority on the agricultural, forestry, fishery, animal husbandry and food affairs in Taiwan. Around 24 % of Taiwan's land is used for farming. The total value of agricultural production in 2019 was NT\$ 515.0 billion, of which farming, forestry, fishery, and animal husbandry accounted for 51.0 %, 0.03 %, 17.1 %, and 31.6 % of this total, respectively. In 2019, Taiwan had 790k hectares under cultivation, accounting for 22.1 % of its total land area. Cultivation area of rice, fruits and vegetables were around 272k, 185k and 152k hectares, respectively. The value of production of farm crops was NT\$ 262.5 billion, accounting for 51.0 % of the total value of agricultural production. The main product was fruits with the share of 35.2 %, following by vegetables 29.6 % and rice 15.2 %. The agricultural employment population was 561k accounting 4.9 % of total employment population. In addition, about 76 % of Taiwan's agricultural scale is less than one hectare, and the average farmland area is about 1.1 hectares, which is belonging to small-scale farming (Agriculture and Food Agency, 2018).

Circular agriculture was a new concept of circular economy applying in agriculture (Paraskevopoulou and Vlachos, 2020). It aimed to reduce waste as well as making use of 'wastes' produced by using economically viable processes and procedures to increase their value. In this research, we used the technology, pyrolysis, to turn agricultural waste into economically valuable products: biochar and pyroligneous acid (PA).



Source: Visible Earth website, NASA **Fig. 1-1** Satellite photo of Taiwan



Adapted from MakeWorld.tw Fig. 1-2 Rice production in 2018

District	Production			
District	m.t.	%		
New Taipei City	909	0		
Taipei City	2,270	0.1		
Taoyuan City	101,450	5.2		
Taichung City	171,158	8.8		
Tainan City	174,350	8.9		
Kaohsiung City	38,385	2		
Yilan County	73,470	3.8		
Hsinchu County	58,292	3		
Miaoli County	68,076	3.5		
Changhua County	381,235	19.6		
Nantou County	37,581	1.9		
Yunlin County	359,002	18.4		
Chiayi County	256,662	13.2		
Pingtung County	47,872	2.5		
Taitung County	77,041	4		
Hualien County	102,043	5.2		
Total	1,949,796	100		

 Table 1-1 Rice production in 2018

Source: Agricultural Statistics Annual Report (2018)

 Table 1-2 Production and usage of rice husk in Taiwan 2018

Usage	Bedding	Growing media	Compost	Feedstuff	Fuel	Total
Amount (m.t.)	125,385	71,648	35,824	53,736	71,648	358,242
%	35.0	20.0	10.0	15.0	20.0	100

Source: Green GDP Estimated Agricultural Solid Waste (Statistics office of COA, 2019)

The reactor was called Charcoal Processing System with Internal Combustion Furnace (CPSICF). The agriculture wastes studied in this research was rice husk and the products, biochar and PA, named as rice husk biochar (RHB) and rice husk vinegar (RHV), respectively. The appropriate using method of RHB and RHV was studied. According to the results of the pot experiment, I hope farmers can use RHB and RHV in agriculture and expect to achieve the goal of circular agriculture through the reuse of rice husk.

1.3 Technology for reusing rice husk

1.3.1 Rice Industry in Taiwan

Rice is Taiwan's most valuable crop, with a total yield of more than 1.9 million metric tons from 272k hectares of land for a production value of NT\$ 36.9 billion (about US\$ 1.2 billion) in 2018 (Agriculture and Food Agency, 2018). In the past ten years, the average annual yield of rice per hectare was 6.3 metric tons. Because the temperature from February to November is suitable for rice growth, rice can be harvested twice a year. The first crop contributes 70 % of annual production that is from February to June and the second from July to November.

Table 1-1 and **Fig. 1-2** revealed the rice production of each county in 2018. The top three were Changhua, Yunlin and Chiayi County accounted for 19.6 %, 18.4 %, and 13.2 % of the total, respectively. The main production areas for rice are in the central area and the western half of Taiwan.

Rice straw is an agricultural waste of rice production at harvest. The rice straw to paddy ratio in Taiwan was about 0.9 to 1.2 (Ni Li Fong, 2003). Farmers used to burn rice straw on the field that causes massive air pollution. With the awareness of environmental protection in recent years, burning rice straw on the field is prohibited now. By using combine harvester, the rice straw is separated from the grains. The combine harvester can be attached with a straw cutter, in which rice straw can be chopped and buried into soil during harvesting, in result of increment on organic matters and minerals (Ponnamperuma, 1984).

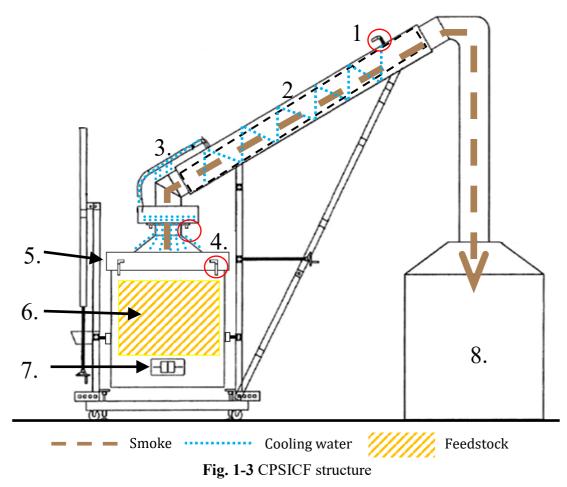
Rice husk is another by-product of paddy cultivation with a paddy ratio 0.2 (Ni Li Fong, 2003) in Taiwan. Most of the rice husk in Taiwan can be collected by rice mill plant, makes it possible for reuse purpose. **Table 1-2** shows the usage of rice husk in Taiwan, most of the rice husk is used as bedding, growing media and fuel.

1.3.2 Introduction of CPSICF

Fig. 1-3 is the structure of CPSICF and **Fig. 1-4** is the photo of CPSICF. Water tank (8) is on the right hand, linking to cooling sprinkler (3). Cooling water can go through two-layer-chimney (2) interlayer and be recycled to the water tank from the upper output port (1), also sprinkle from the cooling sprinkler at the front of the chimney and the top of the lid (5). The cooling water will be collected by the plate above the lid flowing to the cone-shaped lid below and be recycled to the water tank from recycle hole around the cone-shaped lid. There are two PA collected port (4), one is on the top of the lid collecting PA condensed from chimney; another one is at the bottom of the lid collecting PA condensed from lid. The feedstock is placed in furnace (6) which the capacity is about 450 L. Airflow gate (7) is at the bottom of the furnace. Each time the furnace could produce about 14 kg of rice husk biochar (RHB) and 14 kg of rice husk vinegar (RHV) from 40 kg of rice husk.

1.3.3 Biochar and Pyroligneous Acid

Biochar, a porous carbonaceous solid material with a high degree of aromatization and a strong anti-decomposition ability, was produced through decomposition of biomass from plant or animal waste under limited oxygen conditions (Wang, Gao, and Fang 2017). The use of biochar could be an effective tool for sustainable agriculture in the long term, increasing soil C sequestration (abatement strategy), fertility and productivity (soil quality) and reducing greenhouse gas emissions (Jeffery et al., 2015). Pyroligneous acid (PA), a condensed liquid of smoke produced by pyrolysis of plant biomass, was a yellowish brown or dark brown liquid with acidic pH (Mathew and Zakaria, 2015; Mingfenga et al., 2013). PA was also known as wood vinegar, mokusaku, liquid smoke, bio oil, liquid oil, pyroligneous liquid, pyrolysis oil, wood liquid and wood distillate. The application of PA has been widely studied. It exhibits antioxidant and scavenger properties and has been used as antimicrobial agent, insecticide, and for the promotion of seed germination and plant growth (Grewal, Abbey, and Gunupuru, 2018). Mass toxic pesticides have been replaced by PA in organic agriculture (Lu, Jiang, He, Sun, and Sun, 2019).



Legends: 1. Water output port, 2. Chimney, 3. Cooling sprinkler, 4. PA collected port, 5. Lid, 6. Two-layer furnace, 7. Airflow gate, 8. Water tank



Fig. 1-4 Photo of CPSICF

1.4 Structure of the Thesis

The aim of this study is revealed a new pyrolysis reactor and studying the effect of the products, RHB and RHV

In chapter 1, the background of this research is introduced.

In chapter 2, I used CPSICF to produce biochar with rice husk as raw material. In order to study the efficiency of CPSICF, temperature and physical characteristics of RHB were analyzed.

In chapter 3, I studied the characteristics of RHV, another product produced by CPSICF. According to the results, the appropriate operating conditions of CPSICF were confirmed.

In chapter 4, pot experiments were carried to evaluate the efficiency of RHB and RHV which were used as soil amendment and plant promoter, respectively. Plant biomass amount, plant nutrition and sensory test were done for both RHB and RHV experiment. Soil property was only studied in RHB experiment.

In chapter 5, the conclusion of my research and some future plans of CPSICF are given.

CHAPTER 2

Characteristics of RHB Produced by CPSICF

2.1 Research Background

At the 21st Conference of the Parties, the French Minister of Agriculture Stéphane Le Foll set an ambitious international research program, the "4 per mille Soils for Food Security and Climate" of the Lima-Paris Action Agenda. The "4 per 1000" aspired to increase global soil organic matter stocks by 0.4 percent per year as a compensation for the global emissions of greenhouse gases by anthropogenic sources (Minasny et al., 2017). In response to this issue, Cao Qihong, the chairman of the COA, announced the new development direction of agricultural science and technology, "Biochar return to land". He hoped to convert agricultural waste into biochar. By using the property that biochar was not easily decomposed, carbon dioxide in the atmosphere could be sustainably offset into soil. As rice husk production is stable with concentrated production area, it has the potential to be the raw material of biochar.

Biochar could be distinguished from charcoal, used mainly as fuel, as a primary application for a soil amendment with the intention to improve soil function and features the potential to enhance crop productivity and reduce emissions from biomass that would otherwise naturally degrade to greenhouse gases (Blackwell, Riethmuller, and Collins, 2009). The aromatic structure of biochar was responsible for its recalcitrance and potential for long-term C sequestration (Atkinson, Fitzgerald, and Hipps, 2010). When buried biochar in soil, it could act as a long-term soil carbon sequestration remaining for hundreds of years (Ghorbani and Amirahmadi, 2018). Biochar created a recalcitrant soil C-negative pool, using as a withdrawal of atmospheric CO₂ stored in soil C stocks (Glaser, Balashov, Haumaier, Guggenberger and Zech, 2000). Research also showed 50-80 % of nitrous oxide emissions reductions in char-amended soils (Cox, Bezdicek, and Fauci, 2001). Nartey and Zhao (2014) reported that the presence of biochar could decrease the leaching losses of nitrogen and phosphorous in soil and the releases of greenhouse gases (N₂O and CH₄) from soil. Therefore, biochar was under investigation as an approach to carbon sequestration because it had the potential to mitigate climate change.

In addition, biochar not only increased the stable carbon stocks in a soil but also increased nutrient availability beyond a fertilizer effect (Chan and Xu, 2009). Zhang et al. (2017) reported that biochar addition increased soil organic carbon, C/N ratio, and NH₄⁺ and decreased soil bulk density. The application of biochar was a potentially sustainable method for saving irrigation water (Xiao et al., 2018) and soil carbon sequestration(Qi, Niu, Zhou, Jia and Gao, 2018). Because of its porosity and high specific surface area, it could absorb soil nutrients and improve fertilizer utilization. Randolph et al. (2017) observed that incorporation of biochar in soil increased soil pH and improved water retention, electrical conductivity (EC), soil aggregate stability, and micronutrient contents. Biochar chemical alkaline could be used as a material to improve acidic soil (Chintala, Mollinedo, Schumacher, Malo and Julson, 2014); its porous structure could adjust to different soil structures; improve the physical, chemical, and biological functions of soil; and enhance soil fertility (Eilín Walsh, 2012). With potassium, calcium, barium, and other elements, it could directly supple plant nutrients (Tsai Jia Ru and Wu Geng Dong, 2016).

The experimental site was in Cishan District, Kaohsiung City, Taiwan. The experiment date was conducted over April 25 to June 13, 2019. Before the experiment, according to Central Weather Bureau weather forecast, data were only accepted on fair weather days: rainfall probability less than 10% and no thunderstorms in the afternoon. This helped in reducing the influence of humidity changes during the experiment. The CPSICF was settled in a tin house to avoid the inference of external airflow with the intake air volume of the CPSICF opening.

2.2 Purpose of Research

In this chapter, rice husk was used as raw material to produce biochar, called as rice husk biochar (RHB), by CPSICF. The operational conditions were controlled to determine the effect of gate width and cooling water system in the CPSICF. As a preliminary study of CPSICF, the main purpose of this experiment was to effectively improve the productivity and quality of RHB and also reduce production costs. The productivity and quality of CPSICF was revealed.

2.3 Materials and Methods

2.3.1 Rice husk

The material, rice husk, was a hard protective layer of the rice grain and could be used as building material, fertilizer, insulation, or fuel (Padinjakkara, Thankappan, Souza Jr. and Thomas, 2018). Rice husk was a complex lignocellulosic biomass which comprised of high cellulose content (38-50 %), hemicellulose (23-32 %) and lignin (15-25%) (Salimi, Lim, Yusoff and Jamlos, 2017). Rice husk consisted of 60-65 % volatile matter, 10-15% fixed carbon, and 17-23 % ash (Hu et al., 2008; Kwong et al., 2007; Mansaray, and Ghaly, 1997) . Unlike rice straw, rice husk was a by-product of rice milling (Mohamed, Mkhalid, and Barakat, 2015). The bulk density of rice husk lied in the range 90–150 kg/m³ (Bhupinder Singh, 2018). Neither storage nor transportation was ideal due to its high space-taking volume. The rice husk used in this experiment was from the Kaohsiung Cishan Farmer's Association.

2.3.2 The Development of CPSICF

The design of Charcoal processing system with internal combustion furnace (CPSICF) was based on Top-lit updraft gasifier (TLUD). TLUD had the capabilities to produce syngas and biochar simultaneously (Anderson and Reed, 2004). Duo to the high efficiency and low cost of TLUD, most of published researches were about the cooking purposes of this reactor in developing countries (James, Yuan, and Boyette, 2016).

Fig 2-1 was the sketch of TLUD gasifier stove designed for cooking. The feedstock could be dry biomass, wood or agriculture waste. By adjusting air intake, the combustion condition could be controlled and yielded substantial clean heat with charcoal as a co-product.

Fig 2-2 was a biochar kiln based on TLUD. The main structure could be built by 50 gallon drum, a common industrial waste, and covered it by a lid with chimney. At the bottom and around the top the drum, vent holes were punched to supply air during combustion. Usually, the raw material could be waste-wood, wheat straw, rice husk and biomass waste. After ignited, the air could be intake form the bottom vent holes and the raw materials would be burned into biochar in the limited of air. After all raw material turning into biochar, usually took hours to days, removed the lid and sprayed water in stove to extinguish the fire in order to prevent the biochar from burning into ash.

Biochar could be easily produced by using TLUD. However, the yield and quality was not stable and the smoke producing by combustion might cause air pollution. The heat generated by combustion could heat the drum to over 500 °C that might cause fire disaster. Furthermore, if the particle size of raw material was small, such as wheat straw and rice husk, the residue might become ash with a strong alkaline pH over 9.0 instead of biochar due to excessive combustion.

To improve the above disadvantage of TLUD biochar stove, CPSICF was designed as the following features. The furnace was built in two layers, the inner layer was where the raw materials were placed, and the vacuumed interlayer could prevent the temperature rising of the outer layer. This could avoid the risk of fire accident due to the high temperature of the furnace during the production process. The lid could gather the smoke produced during combustion. In addition, it was connected to the chimney to discharge combustion smoke into water tank avoiding air pollution. In order to improve operation convenience, the lid was fixed with iron wire and support making it easily be opened and closed.

The water tank had a pump controller, which connected water tank to cooling sprinkler through a hose. The cooling sprinkler had a sprinkler hole at one end, and the water could be sprinkled on the front end of chimney and the cover. On the other end, it was connected to the chimney, which was also built in two layers with cooling water channel around the interlayer of chimney. The cooling water could pass through the cooling water channel from cooling sprinkler to water output port at the top of chimney.

Besides, the air intake was fully controlled by airflow gate at the bottom of the furnace instead of vent holes. By adjusting the gate at appropriate width, the air could go into the furnace by stack effect. Furthermore, in order to make it easier for users to take out biochar, there were rotating shafts on both sides of the furnace to allow the furnace to rotate and the biochar could be easily poured out.

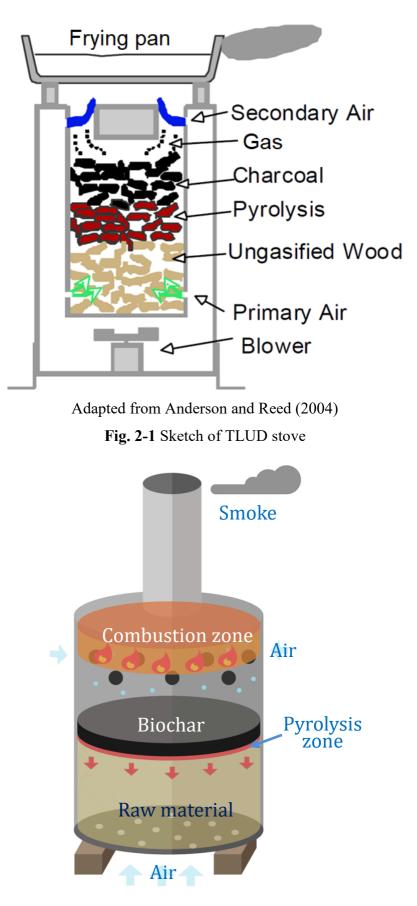


Fig. 2-2 TLUD biochar kiln



Fig. 2-3a Operation photos of CPSICF



Fig. 2-4b Operation photos of CPSICF



Fig. 2-5c Operation photos of CPSICF



Fig. 2-6d Operation photos of CPSICF

2.3.3 Operation procedures of CPSICF

In each trail of experiment, 40 kg of rice husk was used to produce RHB. Then, fired charcoal was placed to ignite rice husk as **Fig. 2-3a**. After rice husk was ignited and the smoke could be observed, the lid was closed, turn on the pump in water tank and adjusted the airflow gate at appropriate width. The cooling water was sprinkling from the cooling sprinkler at the front of the chimney and the top of the lid as **Fig 2-3b**. The combustion process was 21 hours and did not need to pay attention during combustion as **Fig. 2-3c**. After 21 hours, the pump and cooling water was turned off and removed the lid. The furnace was rotated and RHB could be easily taken out like **Fig 2-3d**. The residual material was sprayed with water to extinguish the fire.

The above operation process only requires one person, and there is no need to pay attention on furnace during combustion. Such equipment and simple operation process is very suitable for small-scale agriculture in Taiwan.

The variation factor of the experiment was airflow gate width that was set at 5, 7.5 and 10 mm. Each gate width was studied three times and an additional experiment at 5.0 mm without the cooling water was performed to understand the effect of the cooling water.

2.3.4 Temperature Change in Furnace

The temperature change during burning was recorded using the Portable Data Station (Yokogawa Electric Co., Ltd, Japan; model Yokogawa XL122-D). The temperature measure points were noted in **Fig. 2-4**. Measurement points were set in furnace at top, center and bottom to reveal the actual temperature change during combustion.

2.3.5 Weight and Yield of RHB

After wet RHB was collected, it was spread on tarpaulin and put in tin shelter for airdried. Care was taken to avoid the influence of wind and dust fall. During experiment date, the temperature and average relative humidity was 20-35°C and 74-81%, respectively. After one month, the dried biochar was collected and the weight was measured. The results of the same gate width were averaged for analysis.

The biochar yield was calculated after weight was measured by using the formula, where Yield (%) was the basis yield of biochar, MBiochar (kg) represented the weight, and MRice husk (40 kg) was the weight of rice husk filled in furnace.

$$\text{Yield}(\%) = 100 \times \frac{M_{Biochar}}{M_{Rice\ husk}}$$

2.3.6 Ash Content

After 21 hours of combustion, the inner furnace was sprayed with water. Most of the ash flowed to the bottom of the furnace which could not be collected. Because of the design of CPSICF, there would have a distortion on the real ash content of final product. Therefore the ash content in this experiment was estimated visually by the same experimenter. The ash content was graded after air dried. The scale was from 1-5 with 5 being the highest ash content obtained. The ash content estimated after each experiment would be recorded and compared in the future to see if there was a difference under the same gate width.

2.3.7 pH value

When RHB was removed from the furnace, we collected 20 g of biochar from the top, central, and bottom layers, placed it in a 50-mL beaker, added 20 g water, and continuously stirred the suspension for 5 min. The pH value was measured by pH meter (Thermo Fisher Scientific Inc., America; model Orion Star A329) after the samples were left standing for 30 minutes to allow most of the suspended clay to settle out from the suspension (Agricultural Research Institute, COA, 2013c).

2.3.8 Bulk Density

Bulk density was defined as the dry weight of soil per unit volume of soil. It could represent the biochar weight of per unit volume. The result could also compare with rice husk to understand if there were any changes after burning. Biochar was scattered through sieve freely into the confirmed volume (V0) and weight (M0) vessel until it overflows and carefully scraped the excess powder from the top of the vessel (Quality Assurance and Safety: Medicines (QSM), 2012). The weight of biochar and vessel (M1) was measured by digital scale (A&D Company, Limited Japan; model EK-600i). The bulk density was calculated by using the formula (M1- M0)/V0. Because gate width 5.0, 7.5 and 10.0 mm was done for three times, the biochar sample was taken averagely. The biochar was scattered at the height of 10, 15, and 20 cm. At each height, every sample was done for 10 times and the result was averagely analyzing.

2.3.9 Heat Treatment on RHB

In order to understand the effect of heat treatment, RHB with a complete rice husk shape was randomly selected and the image was obtained by using scanning electron microscope (JEOL Ltd., Japan; model JSM-6510). Then, RHB was put in an oven (CHANNEL BUSINESS CO., Ltd., Taiwan; model DV-902) to dry at 105 °C for 24 hours and obtained images. The experiment was done without any further study in this article. Only the structural differences before and after heat treatment would be determine.

2.4 Results and Discussion

2.4.1 Temperature Change in Furnace

Fig. 2-5 was the temperature record of different gate width at the top, center and bottom in furnace. Temperature change demonstrated that rice husk was burned from the top layer to the center and then reached the bottom. It took about 5 hours to reach the bottom layer and continued burning until the end of the experiment.

In **Fig 2-5a**, when gate width was 5.0 mm, the top point demonstrated a slow upward trend from 32.1 °C to 100.7 °C. By contrast, the center point increased suddenly after 1 hour 20 minutes of burning; indeed, it only took 60 minutes for the temperature to increase from 34.0 °C to 252.1 °C; it then decreased gradually after reaching the highest temperature. The bottom point started rising suddenly after 4 hours 50 minutes, and the temperature was maintained between 350 °C and 370 °C for 2.5 hours before increasing to 505.7 °C after 9 hours. It remained in the range of 505 °C–535 °C for 7 hours 30 minutes and began decreasing after 16 hours 30 minutes.

In **Fig 2-5b**, at gate width of 7.5 mm, the top point showed a slow upward trend from 34.3 °C to 118.2 °C. The center point increased suddenly after 1 hour 50 minutes of burning; it took 45 minutes to rise from 46.4 °C to 135.4 °C and remained in the range of 125 °C–135 °C for 2 hours. The center point started its second stage by increasing to up to 199.8 °C after 6 hours before slowly decreasing. The bottom point started increasing suddenly after 4 hours 35 minutes, after which the temperature was maintained between 200 °C and 235 °C for 2 hours and increased to 523.4 °C after 8 hours 50 minute. It remained at 520 °C–553 °C for 11 hours 20 minutes and started to decrease after 20 hours 10 minutes.

In **Fig 2-5c**, at gate width of 10.0 mm, the top point demonstrated a slow upward trend from 44.4°C to 130.0°C. The center point rose suddenly after 1 hour 40 minutes of burning; after 30 minutes, it increased from 47.6°C to 167.1°C and remained at 130°C– 200°C until the end of the experiment. The bottom point started increasing suddenly after 4 hours 35 minutes and accelerated at 6 hours 15 minutes. After 8 hours, the temperature was maintained in the range of 580 °C–632 °C until 15 hours 15 minutes, and then started to decrease rapidly.

In **Fig 2-5d**, at gate width of 5.0 mm with no cooling water, the top point increased to 206.4 °C after 3 hours 30 minutes. It decreased less than 100 °C at 7 hours 25 minutes and remained in the range of 75 °C–100 °C until the end. The center point increased suddenly after 2 hours 50 minutes from 50.6 °C to 424.3 °C and then began decreasing rapidly but then decreased slowly after 8 hours 30 minutes. The bottom point increased suddenly after 5 hours 10 minutes. It increased from 44.2 °C to 506.0 °C and then remained in the range of 470 °C–545 °C until the end.

With cooling water, the highest temperature of the top layer increased as the gate width increase from 5, 7.5 to 10 mm, and the highest temperatures of each gate width were 100.7 °C, 118.2 °C and 130.0 °C, respectively. Without cooling water, the highest temperature was 206.4 °C that was twice than with cooling water.

At the center layer, the temperature rose for the first six hours and then slowly drops. The temperature change was not related to the gate width. It was estimated that the temperature sensor in the center layer might be affected by combustion, so that the temperature change could not accurately reflect the difference. The presence of cooling water did not have significant different.

At bottom layer, it showed the highest temperature in furnace. With cooling water, the highest temperature of gate width 5, 7.5 to 10 mm was 532.7 °C, 552.9 °C and 631.9 °C, respectively. As the air supply increased during combustion, the heat energy received from the oxidation zone increased. This was characterized by an increase in the temperature of the pyrolysis zone (Dafiqurrohman, Surjosatyo, and Anggriawan, 2018). At gate width 5mm and 7.5 mm, the temperature ranged between 500 °C to 550 °C after nine hours and lasted until the end. However, at gate width 10 mm, it revealed a decrease after 14 hours that showed the combustion process ended and made the temperature have a slow downward trend. There was no significant different with the presence of cooling water at bottom.

2.4.2 Physical Properties of RHB

Table 2-1 presents the relationship between gate width and physical–chemical characteristics of RHB. The results included biochar weight, yield ash content, pH, and bulk density. The temperature would affect the quality and yield of biochar (Jia et al., 2018). It was found that the larger the gate width, the higher was the temperature, and it resulted in the yield decreasing from 36.0 % to 33.8 %. In addition, the ash content increased when the gate width increased, from scale 1 to 3. According to Abrishamkesh et al. (2015), high temperature and severe combustion conditions enhance biomass decomposition, leading to more ash and fine particles and low yield. Saletnik et al. (2018) reported that biochar is a better fertilizer than biomass ash because it has a wider spectrum of activity. At gate width of 5.0 mm with and without cooling water, RHB characteristics did not differ.

The pH of the bottom layer was higher and that of the top layer was lower, comparing with other layers. At gate width of 10.0 mm, the pH was higher than at other gate widths. **Fig. 2-5** and **Table 2-1** revealed that the higher the burning temperature, the higher pH value of the biochar was.

A study found that the pH of biochar typically ranges from 4 to 12 m (Lehmann, 2007) and the results of the present study fell within that range. The formation of carbonates and the contents of inorganic alkalis had a positive relationship with biochar pH value (Ding et al., 2014). In addition, with the rising of pyrolysis temperature, the content of total base cations and carbonates would increase, leading an increase in biochar pH (Yuan, Xu, and Zhang, 2011). The temperature range at which significant changed in the content of acidic and basic substances were observed was 300 °C–500 °C (Abrishamkesh et al., 2015).

The temperature record in furnace revealed temperature increase with gate width increase, especially at the bottom layer. At gate width of 10.0 mm, the bottom temperature increased up to 600 °C after 8 hours that would burn out the acidic substances resulted in the highest pH value. In addition, in **Fig 2-5d**, the temperature of top and central layer was higher than that with cooling water. This resulted in higher pH in the absence of cooling water.

Bulk density obtained at each gate width did not differ much. The bulk density of rice husk was $90-150 \text{ kg/m}^3$ (Bhupinder Singh, 2018). Thus, the bulk density between rice husk and its biochar was not different.

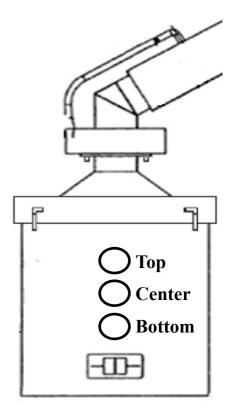
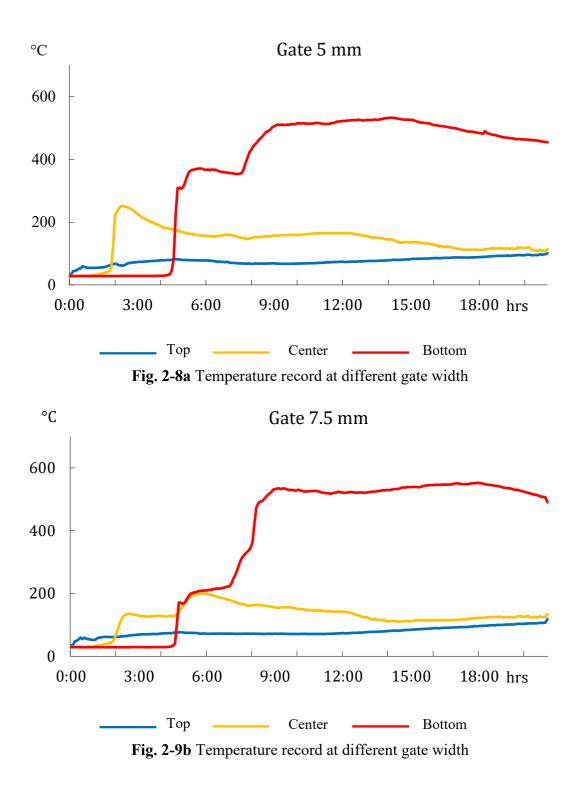
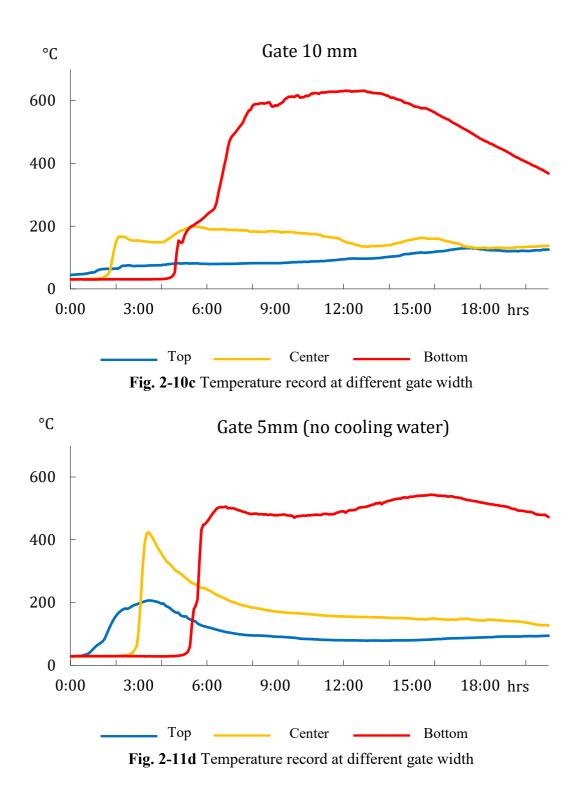


Fig. 2-7 Temperature measurement points in furnace

Gate width (mm)	Weight (kg)	Yield (%)	Ash (Scale)		pH(-)			lk Dens (g/cm ³)	5
()	(8)	()	()	Тор	CTR	BTM	10 cm	15 cm	20 cm
5.0	14.4	36.0	1	4.9	6.0	8.8	0.10	0.10	0.10
7.5	13.8	34.5	2	6.9	7.8	8.9	0.11	0.11	0.11
10.0	13.5	33.8	3	8.6	8.5	9.8	0.10	0.10	0.11
5.0 (no cooling water)	14.6	36.5	1	6.4	7.6	9.1	0.10	0.10	0.10

Table 2-1 Relationship between gate width and biochar characteristics





2.4.3 Heat Treatment on RHB

Fig. 2-6 and **Fig. 2-7** displayed biochar and dried biochar photo taken by scanning electron microscope (SEM) images ($500 \times \text{magnification}$). The multilayer structure and pore space was more obvious after heat treatment in **Fig. 2-7** comparing with **Fig. 2-6**. Furthermore, Intani et al. (2018) reported the phytotoxic potential of biochar before and after heat treatment and reported that it could reduce the levels of volatile organic compounds and polycyclic aromatic hydrocarbons, thus changing the physicochemical properties such as hydrophobicity and hydrophilicity, in the biochar. Thus, dried biochar might have several potential uses and the utility and efficiency should be further study.

2.5 Conclusion

In this research, rice husk was used as raw material and CPSICF to produce biochar. The physical properties of biochar in this study included yield, pH value, and bulk density, which were 33.8 %–36.5 %, 4.9–9.8, and 0.10–0.11 g/cm3, respectively. These results were similar to those of other charcoal processing systems. The result of heat treatment, the efficiency on biochar can be further study and focus on the increase of specific surface area and porosity. Absence of cooling water affected the temperature inside the furnace, but the biochar quality was not affected significantly.

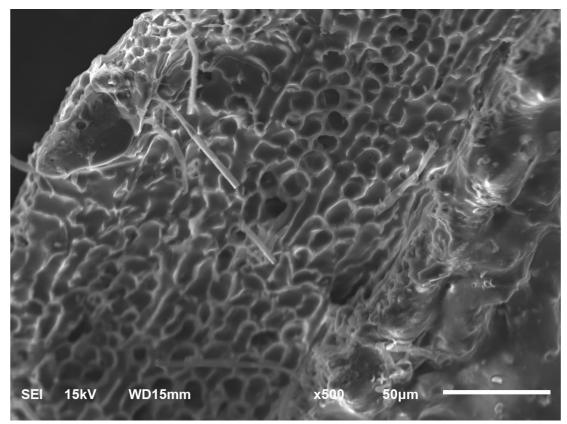


Fig. 2-12 SEM photo of Fresh RHB

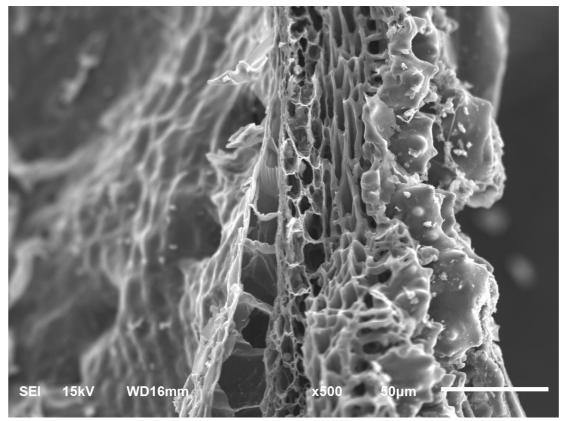


Fig. 2-13 SEM photo of RHB after heat treatment

CHAPTER 3

Characteristics of RHV Produced by CPSICF

3.1 Research Background

Pyrolysis is a thermal conversion process of plant biomass or organic material in the absence of air or oxygen leading to the production of charcoal also known as biochar, smoke, pyroligneous acid (PA) and various gas (Grewal et al., 2018; Tiilikkala, Fagernäs, and Tiilikkala, 2014). Plant biomass pyrolysis involved decomposition, oxidation, polymerization, and condensation of hemicellulose, cellulose and lignin (Risfaheri, Hoerudin, and Syakir, 2018; De Wild, Reith, and Heeres, 2011).

Due to antioxidant and scavenger properties of PA, it was used as antimicrobial agent, insecticide and plant promoter in organic agriculture. The physical properties of PA included a pH 2-4, specific gravity within 1.005–1.016 g/mL, dissolved tar content 0.23–0.89 % wt. and exhibited transparency without suspended solid matter (Theapparat, Chandumpai, and Faroongsarng, 2018). PA was composed of more than 200 water-soluble chemical compounds including acids, alcohols, phenols, esters, carbonyl and furan (Shan, Liu, and Zhang, 2018). Among these, acetic acid accounts for about 50 % in solution (Wei, Ma, and Dong, 2010). The component of PA might change depending on the production method, refining method, storage time and raw materials (Qing, Yong-liang, Zhi-ping, Gao-jian, and Cai-xia, 2019). The component of wood vinegar was complex and unstable. Mingfenga et al. (2013) revealed that chemical reaction would take place and change the components after preservation.

Chang-feng et al. (2019) revealed that PA could effectively inhibit mildew in 48 hours by the ratio of wood vinegar: water = 1:20. PA had an antimicrobial activity against pathogenic microorganisms for humans and animals (Souza, Guimarães, Campos, and Lund, 2018). Mustikawati et al. (2016) found that PA could avoid pest attack of armyworms (*Spodoptera sp.*), pod borer (*Etiella sp.*) and pod sucking (*Nezara viridulla*, *Riptortus linearis*, and *Piezodorus hybneri*) on soybean plants, with a concentration of 15 ml/L of water and applied every two weeks. The efficacy of PA was comparable to synthetic pesticide used in the study. QuanYuan et al. (2009) reported that wood vinegar could act as a foliar fertilizer improving the yield and quality of celery. Bamboo vinegar field experiments conducted in China among three kinds of crop had also demonstrated an increase in plant growth (Mu et al., 2006). Lashari et al. (2013) found that PA could decrease soil pH on leaching soluble salts and improve crop productivity in saline soils.

3.2 Purpose of Research

In previous study, relationship between CPSICF gate width and temperature change in furnace during combustion had been confirmed. In this chapter, the pyrolysis technology was studied first. Then rice husk was used as raw material to produce PA, called as rice husk vinegar (RHV), by CPSICF. The operational conditions were controlled to determine the effect of gate width and cooling water system in the CPSICF. The main purpose of this experiment was to figure out RHV productivity and quality of CPSICF. The experimental site was in Cishan District, Kaohsiung City, Taiwan conducted during April 25 to June 13, 2019.

3.3 Materials and Methods

3.3.1 Pyrolysis Technology

Pyrolysis could be divided into four types in **Table 3-1**: fast, intermediate, carbonization and torrefaction. The yield of three products (gas, liquid and solid) was influenced by mode and the conditions of pyrolysis (Angin, 2013; DeWild et al., 2011). What we studied here was carbonization, also known as slow pyrolysis.

Slow pyrolysis could be carried out in various types of equipment. It could be categorized into three types by heating mode (Garcia-Nunez et al., 2017; Pecha and Garcia-Perez, 2015):

- 1. Partial combustion: The heat of pyrolysis zone came from material combusting. Heat transfer was simple and direct, but efficiency is low (e.g. Earth kiln, Top-lit updraft gasifier).
- Indirect heating: The heat, supplied by an external source, came through outer walls of pyrolysis zone. The external combustion source could be fuel or pyrolysis gases formed during carbonization. This might lose energy but better process control (e.g. Paddle pyrolysis kiln).
- 3. Direct contact with recirculating hot gases: The heating method was by contacted with heat gases inside the reactor. The pyrolysis gases could be recycled and burnt to supply the heat of pyrolysis. The charcoal yield and quality was high. However,

the main disadvantages of this method were high capital costs, attrition problems and the need of external energy (e.g. Lambiotte reactor).

Although direct contact with recirculating hot gases was one of the best slow-pyrolysis technologies, most people could not use this technology due to high capital costs. Even though the quality and yield produced by using partial combustion method would vary due to equipment differences, through the improvement of equipment, the quality and output of yield could be controlled in a certain level.

3.3.2 Production procedures of RHV

The experiment operation procedure and conditions was the same with RHB study. The combustion process was 21 hours with 40 kg of rice husk for each experiment. The airflow gate width was set at 5, 7.5 and 10 mm; each of which was studied three times and one addition without cooling water at 5 mm.

The lid of CPSICF, which was connected to the chimney, could collect the combustion smoke and condense it into PA. The other combustion smoke could be leaded into water tank to purify the exhaust preventing air pollution. **Fig. 3-1** showed that there were two PA collected port. One was at the bottom of the lid which collected RHV condensed on the lid. The sample RHV from this port was mentioned as A. RHV condensed in chimney was collected by another port that was at the top of the lid in front of chimney. The sample RHV from this port was mentioned as B.

3.3.3 Temperature Change on Chimney

Since the production of PA was relying on condensation, the temperature of the chimney was studied. The temperature change during combustion was recorded using the Portable Data Station (Yokogawa Electric Co., Ltd, Japan; model Yokogawa XL122-D). There were two temperature measure points, noted in **Fig. 3-2**. One was at the above of chimney, another one was at the below of chimney.

3.3.4 Physical Properties of RHV

After combustion process, PA collecting from A and B were analyzed separately. The analysis method including weight, pH, total acid content, tar content and suspended solids.

The weight was measured by electronic scale. The pH value was measured using a pH meter (Thermo Fisher Scientific Inc., USA; model Orion Star A329) at a liquid

temperature of 20°C. The total acid content (acidity) was evaluated through titration with 0.1 N sodium hydroxide (NaOH). The content of acetic acid (CH₃COOH), a major organic acid in wood vinegar, was calculated using the titrated sodium hydroxide volume (Mun, Ku, and Park, 2007).

The tar content was measured by boiling 100 mL of the product in a glass beaker; the residue was tar. The amount of total soluble tar indicated the presence of phenolic compounds, which might have antifungal activity and applications as wood preservatives (Theapparat, Chandumpai, Leelasuphakul, Laemsak, and Ponglimanont, 2014).

Suspended solids was measured by sampling 20 g of RHV and filtered through a preweighed standard glass fiber filter. The filter was then dried, and the residue on the filter was retained in a 103 °C–105 °C oven (CHANNEL BUSINESS CO., Ltd Taiwan; model DV-902) for 1 h. The increase in filter weight indicated suspended solids (American Public Health Association, American Water Works Association, 2017).

3.4 Results and Discussion

3.4.1 Temperature Change on Chimney

Fig 3-3 and **Table 3-2** were temperature record on chimney. It revealed the temperature change at above of chimney was similar to room temperature, which ranged from 23 °C to 33 °C. The change of temperature was not significantly different among different conditions. With the addition of the cooling water, the temperature at above and below of chimney did not significantly differ between the gate widths (less than 3 °C).

But without cooling water, temperature at the below of chimney increased at the beginning, reached a peak of 48.2 °C after 4 hours 35 min, and then decreased gradually. The maximum temperature was higher approximately 10 °C than that with the cooling water. The overall difference temperature between at above and below of chimney were approximately 20 °C. This confirmed that cooling by water affected the temperature on the chimney.

		Conditions		Con	Composition (%)		
Mode	Temperature Heating rates		Time	Liquid	G - 1: 1	C	
	(°C)	°C/sec	Time	Liquid	Solid	Gas	
Torrefaction	< 290	> 1	< 30 min	0-5	72-77 solid	23	
Carbonization	400 to 500	> 1	Hrs. to days	32	33 char	35	
Intermediate	400 to 500	1 to 1000	10 to 30 sec	50	25 char	25	
Fast	>500	> 1000	< 1 sec	75	12 char	13	

 Table 3-1 Types of pyrolysis

Adapted from DeWild et al. (2011)

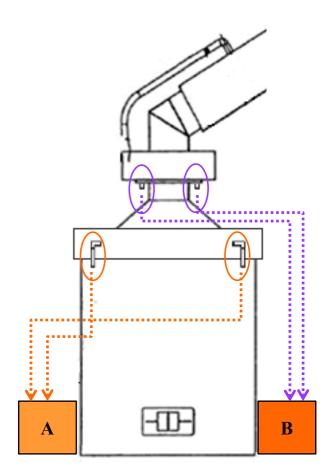


Fig. 3-1 Collection method of PA

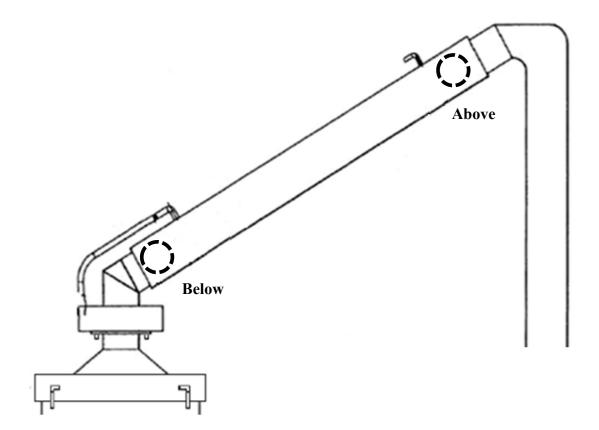


Fig. 3-2 Temperature measurement points on chimney

Cata width (mm)	Abov	e (°C)	Below (°C)	
Gate width (mm)	Min	Max	Min	Max
5.0	29.4	34.1	28.3	34.8
7.5	29.7	34.9	29.0	36.0
10.0	32.6	36.0	31.8	38.9
5.0 (no cooling water)	26.0	31.3	29.7	48.2

 Table 3-2 Temperature record on chimney

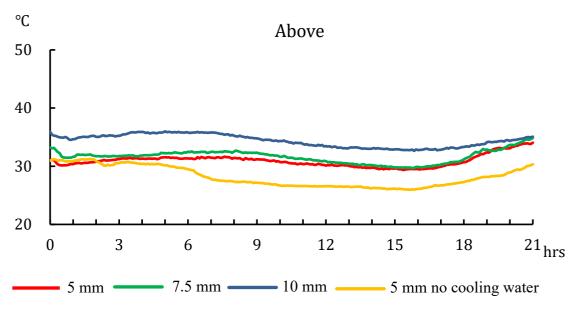


Fig. 3-3a Temperature change of chimney

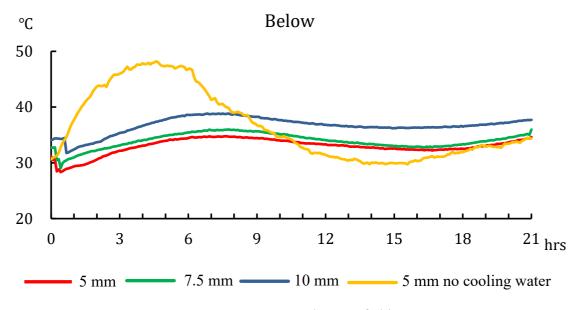


Fig. 3-4b Temperature change of chimney

3.4.2 RHV Characteristic Condensed from the Lid

Table 3-3 showed the relationship between gate width and RHV characteristic which condensed from the lid and collected from the below PA collected port. With cooling water, the highest weight was 11.11 kg at gate width 7.5 mm and the lowest was 10.06 kg at gate width 10 mm. The weight increased first and then decreased with the gate width became larger. Without cooling water, the weight was only 2.09 kg that was far less than others were.

The pH value demonstrated an increase as the gate width became larger. Moreover, the highest pH was observed in the 5 mm gate without cooling water. Comparing temperature change record in previous chapter, pH increased as temperature increased which was confirmed by other research (Hwang, Lee, Ho, Wang, and Yu, 2017).

The acidity demonstrated a decrease with gate width increase under the presence of cooling water. It decreased from 6.3 % at gate width 5 mm to 5.9 % at gate width 10 mm. Without cooling water, acidity increased to up 8.3 %.

As the presence of cooling water, tar content revealed a decrease then increase with the gate width increase. However, the tar content of gate width 5 mm and 7.5 did not differ much, gate width 10 mm was twice than gate width 5 mm and 7.5 mm. Suspended solid also showed the similar situation. In addition, without cooling water the tar content and suspended solid demonstrated significant difference.

3.4.3 RHV Characteristic Condensed from the Chimney

Table 3-4 was the relationship between gate width and RHV characteristic that condensed from the chimney and collected from the above PA collected port. The weight increased with gate width increase. The heaviest was 5.14 kg at the absent of cooling water.

The pH value increased as the gate width became larger, with the highest pH at gate width 5 mm without cooling water. The acidity ranged from 6.2 % to 6.9 %, with a lowest at gate width 10 mm and a highest at gate width 5 mm without cooling water.

With cooling water, the tar content ranged from 3.1 % to 3.7 %. Although it showed an increase with gate width increase from 7.5 mm to 10 mm, the difference was not as significant as the RHV collected from the lid.

Suspended solid showed a significant increase with the gate width increase. The absent of cooling also affected suspended solid increasing by 3.7 mg/L comparing gate width 5 mm with and without cooling water.

3.4.4 Differences of RHV collected from different ports

In the experiments with cooling water, the weight of A, RHV condensed on the lid, was three times more than that of B, RHV condensed in the chimney. The largest total amount was 14.08 kg collected at gate width 7.5 mm. The yield of gate width 5 mm, 13.92 kg, was slightly less. The yield with a 10 mm gate width 13.12 kg was 7 % less than that with a 7.5-mm gate width. Furthermore, at a 5 mm gate width without cooling water, the total yield 7.23 kg was nearly half the amount in other conditions. The weight of A was less than half of that of B, which was different from the condition with cooling water.

Comparing temperature changes in the furnace and chimney with the RHV weight, the results revealed that cooling water affected the temperature also affected the yield of the RHV. Without cooling water, temperature in the top layer was higher, that was revealed in Chapter 2. Higher temperature affected the amount of RHV condensed on the lid. The rest of the RHV was condensed from the chimney.

Higher heating rates tend to favor higher wood vinegar yields (Ratanapisit, Apiraksakul, Rerngnarong, Chungsiriporn, and Bunyakarn, 2009). However, the yield would decrease after certain point. In china, Lu et al. (2018) used China fir sawdust as raw material to produce wood vinegar. It revealed the yiled of wood vinegar increased when temperture increase form 350 °C to 450 °C but decrease after then. Than and Suluksna (2019) reported that increasing primary airflow initially increased each of the process rates but caused a decrease in the rates beyond a certain point.

The pH value was not significantly different between A (3.1–3.6) and B (3.2–3.6). The pH value demonstrated an increase as the gate width became larger. Moreover, the highest pH was observed in the 5 mm gate width and no cooling water condition. Vinegars with lower pH values were preferred because of their higher antibacterial abilities (Budaraga, Arnim, Marlida, and Bulanin, 2016).

The result of acidity in A with cooling water, it showed a decrease as the gate width increased. This result was consistent with the research result of Budaraga et al. (2016) that total acid value decreased as pyrolysis temperatures increased. But there was no

interaction observed between the acidity of B and gate width. In addition, the acidity produced with a 5-mm gate width without cooling water was significant higher than that in the condition with cooling water. However, pH value and acidity were weakly correlated because of a complex effect among the various pyrolyzate components (Mun et al. 2007).

Under all conditions, the tar content of B was almost twice that of A. The least tar content was observed with a gate width of 7.5 mm. Japan Pyroligneous Liquor Association, an industrial body for pyroligneous liquor traders, established that the tar content of good-quality wood vinegar should not exceed 3% (Theapparat, Chandumpai, and Faroongsarng, 2018b). The tar content of B and obtained with a gate width of 5 mm without cooling did not meet this specification.

Suspended solids increased as the gate width became larger. For the 5-mm gate width with and without cooling water, the suspended solids increased 10 times in A but only twice in B. Compared with temperature record, the more obvious of burning condition, the higher suspended solid content would be.

3.5 Conclusion

This experiment revealed RHV properties obtained from CPSICF. The product condensed from lid and chimney was discussed respectively. The result shows that total weight of the RHV was 13.13–14.08 kg but only 7.23 kg without cooling water. The pH value, acidity, and tar content were 3.1–3.6, 5.9%–8.3 %, and 1.3 %–6.1 %, respectively. The suspended solid content was 2.1–8.5 mg/L but reached 20.6 mg/L in A without cooling water. In addition, the temperature change during pyrolysis affected the physical properties of RHV. According to the results, quality of RHV obtained from the CPSICF with gate width 5 and 7.5 mm did not have significant difference.

Gate width (mm)	Weight (kg)	рН (-)	Acidity (%)	Tar Content (%)	Suspended Solid (mg/L)
5	10.98	3.1	6.3	1.5	2.1
7.5	11.11	3.3	6	1.3	2.9
10	10.06	3.4	5.9	2.9	5.1
5 (no cooling water)	2.09	3.6	8.3	3.9	20.6

Table 3-3 RHV characteristic condensed from the lid (A)

Table 3-4 RHV characteristic condensed from the chimney (B)

Gate width	Weight	pН	Acidity	Tar Content	Suspended Solid
(mm)	(kg)	(-)	(%)	(%)	(mg/L)
5	2.94	3.2	6.3	3.3	4.7
7.5	2.97	3.2	6.5	3.1	7.4
10	3.07	3.4	6.2	3.7	8.5
5 (no cooling water)	5.14	3.6	6.9	6.1	8.4

CHAPTER 4

Pot Experiment of RHB and RHV

4.1 Experiment Background

In 2018, Taiwan harvested 2.93 million metric tons of vegetables from 152k hectare of land with a total value of NT\$ 73.3 billion. There are many kinds of various vegetables, especially short-term leafy vegetables, and they are one of the important sources of vitamins and fiber in Taiwan. Short-term leafy vegetables have an advantage of short growing period that has 8-12 production turns in a year. The main production areas in Taiwan are Yunlin County and Taoyuan City, which accounted for 56.2 % and 18.5 % of the total, respectively.

Like paddy cultivation, vegetable cultivation is also relied on the effectiveness of synthetic fertilizers and pesticides. Monocropping situations are also common. These problems not only cause waste of recourse but also degrade the soil health. In the previous chapters, researches revealed that the application of biochar in soil could improve soil health, soil nutrients and fertilizer utilization. The by-product of pyrolysis, PA, could increase plant growth and be used as a foliar fertilizer improving the yield and quality.

In order to confirm the effect of the product obtained in the previous experiment, RHB and RHV were used in agriculture. Since the growing period of vegetables are short and management during the planting period is also relatively simple comparing to paddy cultivation. Vegetables pot experiment were carried out to determine the efficiency of RHB and RHV

The experiment was conducted at MIN-YAN professional seedling factory located at Dapi Township, Yunlin County (23° 38' 6.7" N, 120° 26' 42.1" E). Fig. 4-1 and Fig. 4-2 showed the facilities of MIN-YAN seedling factory. In the greenhouse of seedling factory, the upper part was covered with plastic net and had an opened surrounding. There was movable seedbed for planting and each seedbed had a height of 70 cm, a length of 20 m, and a width of 1.8 m. The black net was at the top of greenhouse, which could cover the greenhouse at noon when the summer temperature was too high. The fans could help ventilate the interior of the greenhouse. The use of these facilities could prevent excessive high temperature affecting plant growth.

4.2 Purpose of Research

The yield of rice husk in Taiwan each year was nearly 300k metric tons and most of them cannot be reused in agricultural. Plenty of agricultural problems were caused by improper use of land. In Chapter 2 and 3, CPSICF revealed the potential of reusing rice husk and making it into valuable products. In Taiwan, although there were many researches on the application of BC and PA, there was still no detailed research including reuse of rice husk, TLUD equipment design, manufacturing method of BC and PA, also the appropriate using method of BC and PA.

In this chapter, vegetable pot experiments were carried to evaluate the effect of RHB and RHV. The efficiency was revealed through plant growth condition, plant nutrition and sensory test. Soil property was studied for RHB as soil amendment to determine the effect of soil health improvement. I hope this research plays the role of summary, someday in the future; farmers can use CPSICF to make a reuse of rice husk and use the products in agricultural cultivation. The goal of circular agriculture can be achieved.

4.3 Materials and Methods

4.3.1 Weather Station Records

The experiment was conducted from 2020/06/03 to 2020/07/07. About 3.2 km away from the experimental site, there was a Central Weather Bureau weather station (23° 38' 4.4" N, 120° 38' 38.4" E). According to weather station record in **Fig. 4-3a**, the mean temperature was 29.0 °C with a highest 30.8 °C on June 29 and the lowest 25.1 °C on June 8. Average temperature difference was 5.7 °C, with a maximum 7.3 °C and minimum 3.8 °C. **Fig. 4-3b** was the humidity record. The average relative humidity was 80.7 % with a maximum 94 % on June 8 and minimum 72 % on June 25. Total accumulated precipitation was 113.5 mm, with a maximum 54.0 mm on June 8. Others had rainfall on June 13, June 30 and July 2. The rainfall record was in **Fig. 4-3c**. Insolation record was in **Fig. 4-3d**, it showed the average amount of insolation was 21.0 MJ/m² with a highest 28 MJ/m² on June 18 and the lowest 1.1 MJ/m² on June 11. The average insolation duration during experiment was 10.3 hours.



Fig. 4-1 Movable seedbed in MIN-YAN seedling factory



Fig. 4-2 Black net and fans in greenhouse

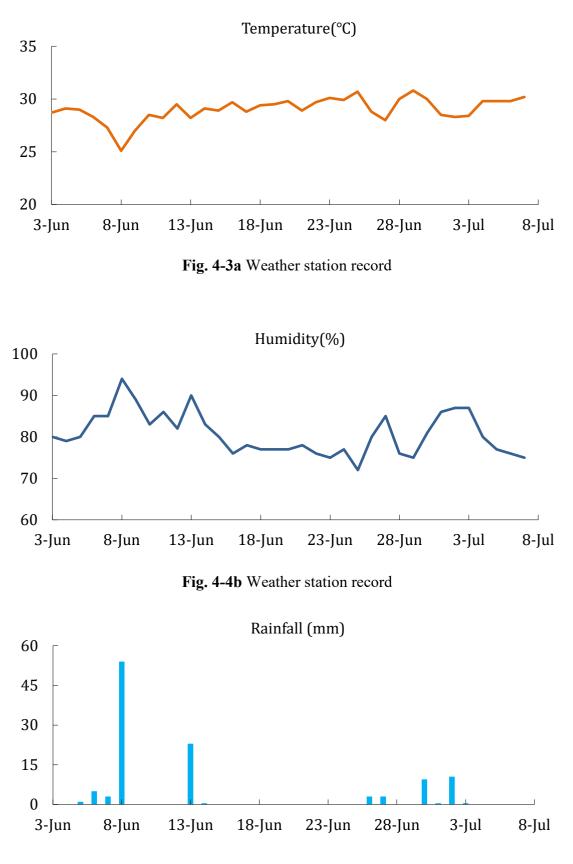


Fig. 4-5c Weather station record

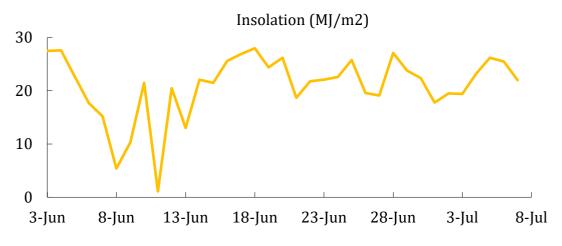


Fig. 4-6d Weather station record



Fig. 4-7 RHB used in pot experiment

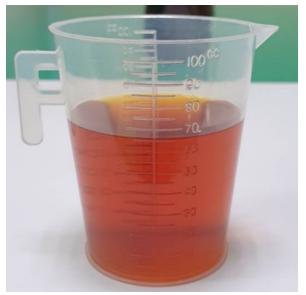


Fig. 4-8 RHV used in pot experiment

4.3.2 **RHB** Preparation

Fig. 4-4 was RHB used in pot experiment. It was obtained in the previous experiment at gate width 7.5 mm on 2019/5/29. RHB was stored appropriately to avoid deterioration.

Before pot experiment, RHB was sent to Taitung District Agricultural Research and Extension Station, which provided farmer with analysis of soil health, water quality, plant nutrition and substrate property. The pH value and EC was measured with a soil-water ratio of 1:10 by pH meter and conductivity meter, respectively. Total nitrogen content was used sulfuric acid (H₂SO₄), salicylic acid (C₆H₄(OH)(COOH)) and sodium thiosulfate (Na₂S₂O₃) under high temperature converted various types of nitrogen in the sample into ammonium nitrogen (NH₃-N) (Agriculture and Food Agency, COA, 2018). Other elements were treated with hydrochloric acid (HCl) and nitric acid (HNO₃) (V: V=3:1) and analyzed by inductively coupled plasma optical emission spectrometry (ICP-OES) (HORIBA Jobin Yvon, Germany; model Ultima 2) (Agriculture and Food Agency, 2018). **Table 4-1** was the physics and chemistry properties of RHB. The results showed that RHB contained a small amount of nitrogen, phosphorus and potassium that were the "Big 3" primary nutrients in commercial fertilizers. RHB was used as a soil amendment to improve soil properties in this experiment and the efficiency were evaluated through plant growth.

4.3.3 **RHV** Preparation

Fig 4-5 was RHV used in pot experiment. It was obtained in the previous experiment at gate width 7.5 mm with cooling water, collecting from the below PA collected port. The production date was the same with RHB on 2019/5/29.

The physical properties of RHV were analyzed by Department of Wood Science and Design, National Pingtung University of Science and Technology (NPUST), Taiwan. RHV had a pH 2.98 and specific gravity 1.018, measured with a pH meter and hydrometer at liquid temperature 20 °C. Acidity was 14.08 %, evaluated through titration. The increase in acidity of fresh and preserved sample from 6.1 % to 14.08 % was because of condensation reaction during preservation. Its color condition was light reddish brown measured by spectrophotometers (X-Rite, Incorporated, USA; model SP60), transparent and without suspended matter.

4.3.4 Soil Preparation

The soil type used in pot experiment was sand stone shale and slate alluvial soils (Department of Soil Sciences, 1975) collected from Huwei Township, Yunlin County, Taiwan (23° 43' 28.0" N, 120° 29' 43.4" E). Fig. 4-6 was the field for soil collection of which had a length of 115 m and a width of 25 m. This field was planted with Fushan lettuce (Lactuca sativa L.) before collecting soil. After harvest, the field was plowed by rotary tiller at depth of 20 cm. Before planting and fertilizing in the next period, the soil was collected under sunny day on 2020/4/9 2:00 PM. In order to avoid differences of soil fertility in the field, the field was divided into blocks with a size of width 9.2 m length 12.5 m total 18 blocks. In each block, a total of 40 kg soil was collected randomly with a shovel and putting it in a burlap bag. After the above steps, a total of about 720 kg of soil was collected from the field. After then, the soil was spread on carton like **Fig. 4-7**, placed under tin shelter and turned every day to mix the soil evenly for naturally air-dried. After 10 days the soil was sieved through #10 mesh sieve (particle size less than 2 mm) (Liao and Thomas, 2019) in Fig. 4-8. Soil sterilization was done by spread the sieved soil on plastic film and put under sun exposure for 8 hours. A total of 500 kg soil was collected for pot experiment. After all treatment, Fig. 4-9 was the soil for pot experiment.

The soil properties was analyzed by Tainan District Agricultural Research and Extension Station. pH was measured using pH meter with a soil-water ratio of 1:1 (Agricultural Research Institute, COA, 2013c). EC was measured by conductivity meter with a soil-water ratio of 1:5 filtered by Whatman No.5 filter papers (Agricultural Research Institute, COA, 2013b). Soil organic matter (SOM), C/N ratio and total nitrogen content were analyzed by total organic carbon (TOC) analyzers (Elementar Analysensysteme GmbH, Germany; model soli TOC® cube Analyzer). Other elements were extracted by Mehlich 3 method (Agricultural Research Institute, COA, 2013a) and analyzed by ICP-OES (Thermo Fisher Scientific Inc., USA; model iCAP 7000 Series ICP-OES). The analyzed result of material soil was in **Table 4-2**.

EC (dS/m)	2.66	Potassium (%)	1.16	Zinc (ppm)	47.10
pН	8.78	Calcium (%)	0.25	Copper (ppm)	7.09
Nitrogen (%)	0.81	Magnesium (%)	0.16	Iron (ppm)	167.83
Phosphorus (%)	0.66			Manganese (ppm)	389.09

Table 4-1 RHB properties

Table 4-2 Experimental soil properties

EC (dS/m)	0.67	Phosphorus (ppm)	336	SAR	1.16
	0.07	- molition (bbin)	200	2.110	
pН	6.07	Potassium (ppm)	242	Zinc (ppm)	7.22
SOM (%)	1.22	Calcium (ppm)	2054	Copper (ppm)	3.20
C/N ratio	6.62	Magnesium (ppm)	286	Iron (ppm)	686
Nitrogen (ppm)	1100	Sodium (ppm)	213	Manganese (ppm)	42



Fig. 4-9 The field for soil collection



Fig. 4-10 The soil in carton for air-dried



Fig. 4-11 #10 mesh sieve



Fig. 4-12 Pot experiment soil



Fig. 4-13 Five inch pot with gravel at the bottom



Fig. 4-14 Fushan lettuce plug seedling

4.3.5 Materials for Pot Experiment

The soil analysis result revealed that SOM was only 1.22 %, which was lower than 3 % that was suggested by Tainan District Agricultural Research and Extension Station. Savci (2012) revealed that chemical fertilizer might pollute water, soil and air due to unsuitable method. In addition, the usage of chemical fertilizer might compromises the term "sustainable" due to negative environmental consequences (Beeby et al. 2020). Therefore, organic fertilizer was used in this research. The organic fertilizer had an N- P_2O_5 -K₂O ratio of 5-2.5-2.5 and 81 % organic matter (Taiwan Fertilizer Company, Taiwan, #1 Biotec Organic Fertilizer). After soil preparation, the soil was placed in cardboard box and dig 11 ditches, each of which had an approximately depth and width of 15 and 20 cm, respectively. A total of 1.5 kg of organic fertilizer was evenly placed in each ditch, and mixed evenly with shovel.

The pot used in this experiment was 5-inch pot with a 14.7 cm outer diameter, 13.7 cm inner diameter, height 12.6 cm and a capacity of 1.2 L. In each pot, 350 g of gravel was placed at the bottom under the soil to avoid soil lose as **Fig. 4-10**. The gravel was purchased from a gravel factory, cleaned with water and dried by sun before using.

Fushan lettuce (*Lactuca sativa L.*), an annual species in the Asteraceae family, was used in this pot experiment. It was also the crop planted on the soil-collected field. According to Taipei Agricultural Products Marketing Corporation, Fushan lettuce was introduced by Taiwanese farmers from China in 1999. Since 2001, it had become a common vegetable in southern Taiwan and could be planted all year round. The main cultivation area was Yunlin County, the main producing area of Taiwan's leafy vegetables, and the daily market transaction volume was about 10 to 20 metric tons. Fushan lettuce plug seedling in **Fig. 4-11** was bought from Tung-Yi Sinon supply center (23° 30' 41.8" N, 120° 29' 20.0" E) and transplanted into pots.

Temperature and humidity (T&H) changes in the greenhouse were different from the outer environment. Therefore, in addition to the weather data from weather station, the T&H changes in greenhouse were recorded once an hour by using T&H data logger (JETEC ELECTRONICS CO., LTD, Taiwan; model SYS-TH-L).

In the greenhouse, an irrigation system could irrigate through showerhead by using tap water as it shown in **Fig. 4-12**. The crop was irrigated twice a day and the irrigation amount was adjusted according to plant growth and soil conditions. The daily irrigation amount was recorded by pots with plastic bag inside placed in order in each block. After

irrigation, the water in plastic bag was measured by measuring cup. The water volume of each pot was recorded in table and averaged the value as the irrigation amount of the day.

4.3.6 Experiment Design for RHB

RHB experiment was conducted 34 days from June 3 to July 6. Since RHB was used as a soil amendment, it was necessary to mix it with soil before putting in the pot. The application rate in soil (w/w) was divided into three levels and one control group: A: 0%, B: 2%, C: 4% and D: 6%. With a total of 1.2 kg mixed soil, the soil weight for A, B, C and D were 1200, 1176, 1152 and 1128 g, respectively. For each treatment, besides A, the soil was put in plastic bag to weigh the required weight then added RHB until the total weight reaching 1200 g. Then held the opening tightly with one hand and lifted up the bottom of the bag with the other hand to mix the soil evenly.

According to the application rate for each treatment, the soil was measured and put it into pot one by one to avoid differences in the application rate. Two days before planting, 200 g of water was irrigated in pot every day and loosen the soil made it suitable for planting crops.

In order to understand the effect of RHB as soil amendment, a randomized complete block design (RCBD), a standard design for agricultural experiments, was used for experiment design. The RHB experiment was designed in 4 treatments, 10 replications and 4 blocks with a total 160 pots. Each pot was planted one crop and identified by label. The placement on seedbed was shown in **Fig. 4-13** and **Fig. 4-14** was the photo of RHB experiment block 3. The planting seedbed was composed of black squares with a width 95 cm and length 180 cm. Each block of 40 pots was placed on one square. In each block, each treatment was placed in one column and 10 pots for each treatment.

4.3.7 Experiment Design for RHV

RHV was used as plant growth promoter and RCBD was also used for experiment design. RHV experiment was conducted 34 days, began one day later after RHB experiment, from June 4 to July 7.

RHV was used as a plant promoter; therefore, it was diluted 5×10^{-2} and using a sprayer spraying on leaves during planting period. The spraying amount of the sprayer was tested by spraying the sprayer into cylinder, spraying 20 times and recording the liquid volume in the cylinder. The spraying volume per shot was 0.75 ml.

The experiment was designed in different using frequency during planting period. The spraying amount for each time was depending on plant growing situation. The amount of each spray should be such that the leaves were covered by RHV and would not drip onto the soil, as it showed in **Fig 4-15**. There were three different using frequency and one control group: E: none, F: once, G: twice, H: three times.

The RHV experiment was designed in 4 treatments, 10 replications and 4 blocks with a total 160 pots. RHV experiment was in the middle of seedbed with one space block from RHB experiment as mentioned in **Fig. 4-13**. The soil for each pot was 1.2 kg and weighed one by one. Each pot was planted one crop identified by label.

4.3.8 Sampling Rule

The analysis methods to evaluate the effect of RHB and RHV on plant growth included four methods and statistical analyses. The methods were: 1. Plant biomass amount, 2. Plant nutrition, 3. Sensory test and 4.Soil property. RHB was used as soil amendment that may change the soil properties in many ways. However, the application of RHV was not sprayed directly on the soil. Therefore, the soil property was only studied in RHB experiment.

Since there were 10 pots for each treatment in each block, and four methods were analyzed, the sampling method should be established first. Before sampling, the 10 pots of each treatment in each block were arranged by size. This could avoid analysis errors caused by differences in crop growth. The sampling rules were in **Fig. 4-16** and as followed:

- 1. The largest and smallest crops were not sampled.
- 2. The 2nd was sampled for sensory test.
- 3. The 3rd to 7th were sampled for plant biomass amount.
- 4. The 8th and 9th were sampled for plant nutrition.
- 5. The soil in RHB experiment was collected for soil property analysis.



Fig. 4-15 Irrigation method during experiment



Fig. 4-16 Placement of RCBD for two experiments



Fig. 4-17 Placement of RCBD in RHB experiment block 3



Fig. 4-18 Spraying RHV with sprayer

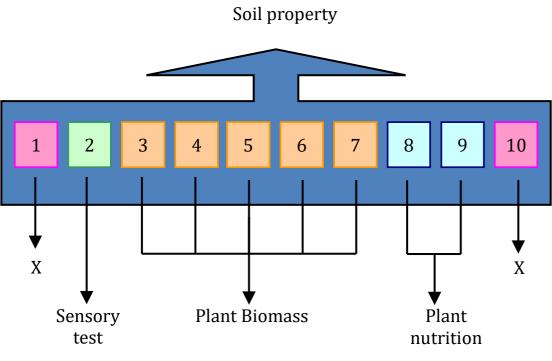


Fig. 4-19 Sampling rule



Fig. 4-20 Sample placed on paper towel for drying

4.3.9 Plant Biomass Amount

The measurement result of plant biomass amount can represent the growing condition of crop. This was carried out for both RHB and RHV experiment. After sampling, the crop was removed by tapping the bottom of the pot. The gravel at the bottom was removed by patting the soil block gently. The soil was gathered together according to each treatment. The roots and the remaining soil were slowly washed with water until the roots became clean. After cleaning, the crop was wiped with paper towel and put it in a cool place to dry for 10 minutes as **Fig. 4-17**, and then measured the plant biomass. After fresh plant biomass amount was measured, the crop was put in envelope and placed in oven (CHANNEL BUSINESS CO. LTD, Taiwan; model DV-1203L) for 72 hours to dry. The length was measured by digital caliper (Mitutoyo Corporation, Japan; model 500-173-30). The weight was measured by electronic balance scale (OHAUS Corporation, USA; model G4000D). The analysis items were as followed:

- 1. Root length (mm): From plant base to the longest root.
- 2. Plant height (mm): From plant base to the heights leaf.
- 3. Leaf number: Number of leaves on plant.
- 4. Wet root mass (g): The total weight of fresh root.
- 5. Wet above-ground biomass (g): The weight of fresh all above ground weight, including leaves and stem.
- 6. Dried root mass (g): The root weight after drying.
- 7. Dried above-ground biomass (g): The dried weight of all above ground part
- Moisture content: Both root and above-ground was evaluated by (Wet mass Dried mass)/Wet mass

4.3.10 Plant Nutrition

Nitrate and nitrite are natural components in plant. However nitrogen fertilization and light intensity had been identified as the major factors that influenced nitrate content in vegetables (Correia et al., 2010). Liu et al. (2014) reported that the application of organic fertilizers had lower nitrate content compared with conventional lettuce. In addition, the application of biochar could increase vitamin C and decrease nitrate in tomato fruits (Antonious, Turley, and Dawood 2019). Dietary fiber, which was known for its ability to prevent or relieve constipation, can be found in vegetables. Therefore,

the plant nutrition content studied in this research included the content of nitrate, nitrite, Vitamin C and dietary fiber.

The nitrate and nitrite content was analyzed by high performance ion chromatograph (HPIC) after extraction and purification (Food and Drug Administration, 2011). The analysis method was an expansion method based on HPIC method (Food and Drug Administration, 2016). Analysis method of dietary fiber was Total Dietary Fibers in Foods, Enzymatic-Gravimetric Method (AOAC 985.29) (Tsai, Lu, Yu, Lin, and Fu, 2007).

After sampling, the crop was packed in a plastic bag and labeled according to different treatment. The plastic bag was put in leisure cooler with ice at the bottom to keep sample fresh. The plant nutrition analysis was done for both RHB and RHV experiment. The results were carried out by SGS Taiwan Ltd.

4.3.11 Sensory Test

The sensory test was studied for both experiments and held on 2020/7/7 (THU). The area was as **Fig. 4-18a**. There were ten people attended sensory test, comparing the color, flavor and texture of fresh and cooked samples. The participants did not have the habit of smoking which might affect the sense of taste. Prior to the sensory test, the following steps were done.

- 1. Vegetable preparation
 - (1) Take out the sample for sensory test and cut off the above ground part.
 - (2) Cut off the area close to the root and separate the leaves.
 - (3) Wash the leaves one by one with clean water then soak in clean water for 5 minutes as Fig. 4-18b.
 - (4) Then take it out of the water and wash it again, and put it on the strainer to drain the water.
 - (5) Cut the vegetables into 4-5 cm size and wait to be cook.
- 2. Materials
 - (1) For cooking: induction stove, stockpot, strainer, teaspoon, tongs, salt and soybean oil.
 - (2) For participants: toothpaste, chopsticks, plate and toothbrush.
- 3. Cooking recipe

The cooking method was blanching like Fig. 4-18c and the procedure was as followed:

- (1) Put 1.5 L of water in the stockpot and heat with induction stove until the water boils.
- (2) Add a teaspoon of salt and soybean oil (about 1.25 ml) in boiling water.
- (3) Then put the sample vegetable in stockpot and boil for 30 seconds. During cooking, use tongs to stir the vegetables to avoid uneven heating of the vegetables.
- (4) Use the strainer to pick it up and set aside to dry for 1 minute.
- 4. Procedure for participants

In order to avoid the error of the test results, the participants was asked to brush their teeth with the same brand of toothpaste and the same brand of toothbrush before each sensory test trial, then followed the steps below:

- Participants were asked to score the color of fresh sample after above ground part was cut off and before further steps of vegetable preparation.
- (2) After vegetable preparation, participants tasted the fresh vegetable, filled out the texture, and flavor score of fresh vegetable on scorecard.
- (3) Gargle with warm water (30 $^{\circ}$ C) to clean up the mouth.
- (4) The chef cooked the sample by recipe above.
- (5) After cooking, the participants taste and score cooked sample.
- (6) Complete the above steps, participants brushed teeth and proceeded to the next round of testing.
- 5. Standard of scoring

In sensory test, the fresh and cooked sample was score individually. The score item included color, texture and flavor. Participants scored 1 to 5 points according to their experience. There were some scoring standard below which was provided to participants before sensory test (Barrett, Beaulieu, and Shewfelt, 2010).

- (1) Color: Color remained the appearance of vegetables including the size, the shape, the wholeness, the presence of defects (blemishes, bruises, spots, etc.), finish or gloss, and consistency. After cooking, whether the color of the sample still remained fresh and light green.
- (2) Texture: Texture could be defined the sense of touch including juiciness, crispness, toughness, hardness, fibrousness, and mealiness.
- (3) Flavor: The flavor of vegetables could divided into five primary tastes—sweet, sour, salty, bitter, and umami. The bitterness came from the organic alkali that

might be caused by irrigation, fertilization, temperature, timing of harvesting and storage.

4.3.12 Soil Property for RHB Experiment

Biochar as soil amendment might change the soil properties in many ways. Therefore, it was necessary to study soil properties. The analysis items were the same with **Table 4-2**. By comparing the difference of before and after experiment and the difference between different treatments, the effect of biochar could be studied.

After removed the crop of each pot, the soil was gathered together according to different treatment. After the soil was collected, it was placed in a cool place air-dry for 2 weeks. The soil was grinded by wooden pole and sieved through #10 mesh stainless steel standard sieve (particle size less than 2 mm) then using quartering method (Campos-M and Campos-C, 2017) to sample 1 kg soil. The sampled soil was sent to Tainan District Agricultural Research and Extension Station for soil properties analysis.

1. pH value (Agricultural Research Institute, COA, 2013c)

Took out 20 g of soil sample and mixed it with deionized water at a soil-water ratio of 1:1 (w: w) in a 50 ml beaker. Then stirred the mixed liquid with a glass rod every 10 minutes. The pH value was measured after one hour standing with a pH meter (DKK-TOA Corporation, Japan; model HM-25R)

2. EC (Agricultural Research Institute, COA, 2013b)

Took out 20 g of soil sample and mixed it with deionized water at a soil-water ratio of 1:5 (w: w) in 250 ml Erlenmeyer flask. Put Erlenmeyer flask on reciprocal shaker and shook at 140 rpm for one hour. After shaking, the soil solution was sieved through Whatman No.5 filter paper and used a conductivity meter (WTW, German; model inoLab Cond730) to measure liquid EC.

3. SOM and nitrogen content (Agricultural Research Institute, COA, 2013d)

SOM and nitrogen content was analyzed by TOC analyzers (Elementar Analysensysteme GmbH, Germany, soli TOC® cube Analyzer) with 5 g of sample soil (Pan, Wang, and Jiang 2018).

4. Macro element and minor element (Agricultural Research Institute, COA, 2013a)

5 g of sample soil was mixed with Mehlich 3 extract at a soil- extract ratio of 1:10 (w: w) and shook at 140 rpm by reciprocal shaker. After shaking, the soil solution was sieved through Whatman No.5 filter paper. The element content of the liquid was

analyzed by ICP-OES (Thermo Fisher Scientific Inc., USA; model iCAP 7000 Series ICP-OES).

4.3.13 Statistical Analysis

The results of plant biomass amount were analyzed by using one-way analysis of variance (ANOVA) at a significance of P = 0.05. The differences between mean values were identified using the Fisher's protected least significant difference test (LSD test) at a significance of P = 0.05. The results of sensory test were analyzed by using one-way ANOVA at a significance of P = 0.05. The differences between mean values were identified using the Student's t-test at a significance of P = 0.05. The data of plant biomass amount and sensory test were carried out by SPSS version 22 (IBM Corporation, USA).

4.4 Result and Discussion

4.4.1 T&H Record and Irrigation Amount

Fig. 4-19 revealed that the average temperature was 31.8 °C during June 3 to July 7, with a highest 53.6 °C on June 26 14:30 and a lowest 23.1 °C on June 18 04:30. In **Fig. 4-20**, the average humidity was 69 %, with a highest 94.4 % on July 3 06:30 and a lowest 22.6 % on June 26 14:30. Comparing with weather station record, which had a highest temperature 30.8 °C on June 29 and the lowest temperature 25.1 °C on June 8, although external factors might affect the environment in the greenhouse, there didn't have significant relationship.

The irrigation amount of RHB and RHV experiment was recorded separately. It was found in **Fig. 4-21** that irrigation amount increased with plant growth from 50 ml/day to 150 ml/day. It was known that irrigation water supply is a key to horticultural productivity. Weather and plant growth might affect irrigation amount. According to weather station record, it rained on June 8, June 13, June 30 and July 2 resulted in lower irrigation amount on these days.



Fig. 4-21a Preparation of sensory test



Fig. 4-22b Preparation of sensory test



Fig. 4-23c Preparation of sensory test

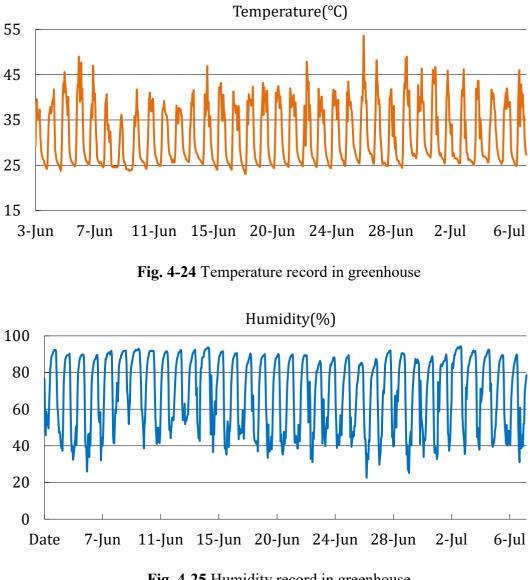


Fig. 4-25 Humidity record in greenhouse

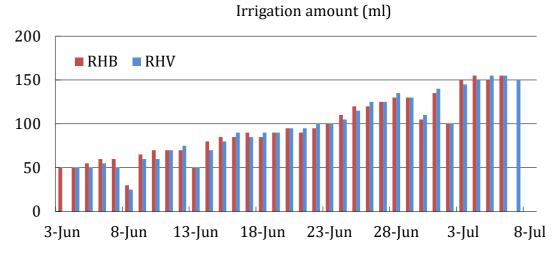


Fig. 4-26 Irrigation amount record of RHB and RHV experiments

4.4.2 RHB Plant Biomass Amount Result

Table 4-3 was the plant biomass amount of RHB experiment. It showed that the use of RHB had a significant effect on the plant biomass. The root length increased by 29.1 % at B4 and 61.2 % at B6, comparing with Control. However, Control and B2 did not have significant difference. Plant height did not have significant difference at Control and B2. It increased 24.0 % from Control to B6. Leaf number only showed significant difference at B4 and B6, increased by 21.5 % from Control to B6. The wet weight of root and aboveground did not have significant difference at Control and B2. Comparing Control and B6, it revealed a significant increased at wet root mass by 175.7 % from control to B6. The wet aboveground biomass also increased by 61.4 % from control to B6. At dried weight, the application of RHB had significant difference comparing with Control. The increasing rate of dried root mass weight at B2, B4 and B6 were 33.7 %, 81.4 % and 112.8 %, respectively. The increasing rate of dried aboveground weight at B2, B4 and B6 were 9.5 %, 12.3 % and 40.1%, respectively. The moisture content of root and aboveground showed a decrease at B2 and B4 comparing to Control, but increase at B6.

Fig. 4-22 and Fig. 4-23 was the growth condition and measurement result of RHB experiment started on 2020/6/3. In Fig. 4-22a, six days after transplanted on June 8, the effect of RHB was not significantly. However, after 20 days in Fig. 4-22b on June 22, a significant difference in appearance of A (Control) and D (B6) could be found. Fig. 4-23 showed that the roots and crops had significant differences in weight, length and density. Therefore, the effect of RHB on plant growth could be proven.

Treatments	Root Length	Plant height	Wet weight (g		eight (g)	Dried w	veight (g)	Moisture content (%)		
	(mm)	(mm)	Leaf Number	Root mass	Aboveground biomass	Root mass	Aboveground biomass	Root mass	Aboveground biomass	
Control	128.11±32.2 d	152.66±18.1 c	20.95±2.3 c	6.34±2.7 c	48.55±14.7 c	0.86±0.3 d	3.89±0.9 d	86.4	92.0	
B2	137.00±34.9 cd	164.82±19.1 c	21.25±2.2 c	7.57±2.1 c	50.93±19.3 c	1.15±0.4 c	4.26±0.9 c	84.8	91.6	
B4	165.45±38.0 bc	167.47±18.07 b	23.10±2.79 b	10.10±3.06 b	50.94±12.78 b	1.56±0.58 ab	4.37±1.09 ab	84.6	91.4	
B6	206.51±32.41 a	189.31±21.16 a	25.46±3.04 a	17.48±5.60 a	78.35±25.36 a	1.83±0.57 a	5.45±1.66 a	89.5	93.0	

 Table 4-3 Plant biomass of RHB experiment

B2, B4 and B6 = RHB application rate (w/w) 2,4 and 6%

a, b. Means with the same letter are not significantly different at 5% level, according to Fisher LSD test and \pm value indicate mean standard deviation



Fig. 4-27a Growing condition of RHB experiment



Fig. 4-28b Growing condition of RHB experiment

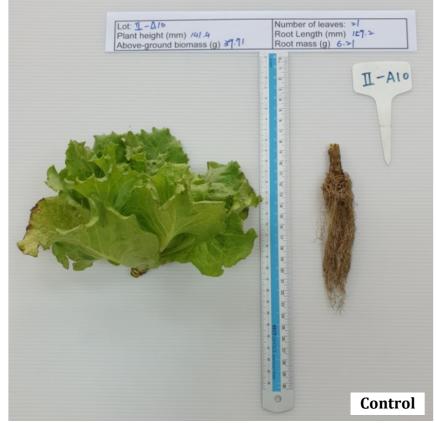


Fig. 4-29a Measurement result of RHB experiment

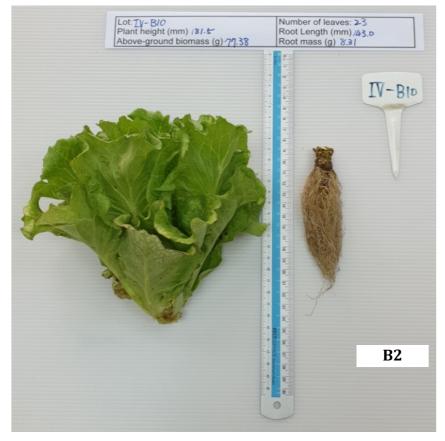


Fig. 4-30b Measurement result of RHB experiment



Fig. 4-31c Measurement result of RHB experiment



Fig. 4-32d Measurement result of RHB experiment

4.4.3 RHV Plant Biomass Amount Result

Table 4-4 is the plant biomass amount of RHV experiment. It showed that the application of RHV did not have any significant effect on leaf number, wet and dried weight of root mass. Comparing to Control, root length increased by 7.2 % and 9.6 % at V2 and V3, respectively, and had no significant difference at V1. The same as plant height did not have significant difference at V1 but increased by 12.8 % at V2 and 13.5 % at V3. The wet weight of aboveground only had significant difference at V3, increased by 16.9 %. The dried weight of aboveground increased by 31.5 % at V2, 34.8 % at V3 and didn't have significant difference at V1.

Fig. 4-24 and **Fig 4-25** was the growth condition and measurement result of RHV experiment started on 2020/6/4. In **Fig. 4-24**, neither on June 8 nor June 22, the effect of RHV could not be discovered significantly. After harvesting in **Fig 4-25**, the difference could be found at root density, but there was no correlation between density and RHV using frequency.

Treatments	Root Length	Plant height		Wet w	veight (g)	Dried v	weight (g)	Moisture content (%)	
	(mm)	(mm)	Leaf Number	Root mass	Aboveground biomass	Root mass	Aboveground biomass	Root mass	Aboveground biomass
Control	159.08±32.59 c	160.85±21.50 c	20.55±2.89 a	7.19±3.52 a	50.95±18.92 b	0.73±0.32 a	3.65±1.13 c	89.8	92.8
V1	142.51±28.71 c	172.47±18.02 c	21.35±2.97 a	7.61±3.98 a	54.83±17.92 b	0.87±0.35 a	4.06±1.25 bc	88.6	92.6
V2	170.51±28.38 b	181.41±19.26 ab	20.51±2.58 a	8.15±3.73 a	57.18±14.30 ab	0.83±0.33 a	4.80±1.05 ab	89.8	91.6
V3	174.43±25.49 a	182.63±24.01 a	21.75±3.28 a	9.72±4.12 a	59.58±21.91 a	0.89±0.33 a	4.92±1.57 a	90.8	91.7

 Table 4-4 Plant biomass of RHV experiment

V1, V2 and V3 = RHV using frequency once, twice and three times

a, b. Means with the same letter are not significantly different at 5% level, according to Fisher LSD test and \pm value indicate mean standard deviation



Fig. 4-33a Growing condition of RHV experiment



Fig. 4-34b Growing condition of RHV experiment



Fig. 4-35a Measurement result of RHV experiment

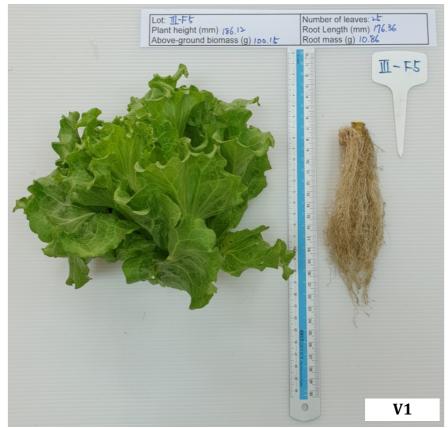


Fig. 4-36b Measurement result of RHV experiment

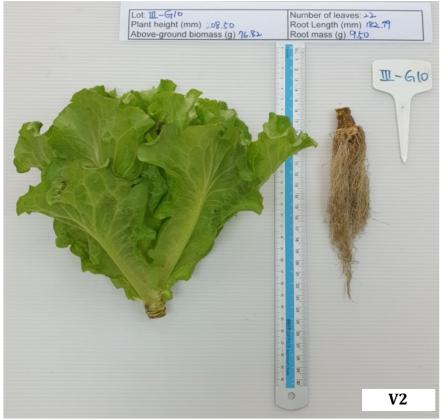


Fig. 4-37c Measurement result of RHV experiment



Fig. 4-38d Measurement result of RHV experiment

4.4.4 RHB Plant Nutrition Result

Table 4-5 was the result of RHB plant nutrition analyzed by SGS. Nitrite and vitamin C were not detected. According to Food Database (Food and Drug Administration n.d.), the vitamin C content of Fushan lettuce was 3.8 mg/100g. However, the detection limit was 5 mg/100g. Therefore, the effects of RHB on the vitamin C content of Fushan lettuce could not be evaluated.

Generally, fresh, undamaged and well-preserved vegetables had extremely low nitrite content and much lower than nitrate. It was because the activity rate of nitrite reductase was in equilibrium with nitrate reductase. Bacteria also affected nitrite content of fresh vegetable (Chung, Chou, and Hwang, 2004; ND, E, and S, 2001). If the activity of nitrite reductase could not match with nitrate reductase, the nitrite levels might also increase (Walker, 1975). According to the result and above researches, the sample vegetable was well preserved and fresh, resulted in no nitrite content.

Dietary fiber content of RHB experiment had a highest at Control and lowest at B2. The application of RHB in soil did not have significant effect on plant dietary fiber content.

The change of nitrate content was significantly, with a highest at B2 and lowest at B6. **Table 4-5** showed that the application of RHB in soil could reduce the nitrate content in fresh vegetable by 39.4 % comparing with B6 and Control. The application of RHB had a negative effect on nitrate content of crop.

4.4.5 RHV Plant Nutrition Result

Table 4-6 was the result of RHV plant nutrition analyzed by SGS. The same as RHB experiment, nitrite and vitamin C were not detected. It showed that the application of RHV revealed a 58.5 % decrease in nitrate content comparing with V3 and Control. The nitrate content increased from V2 to V3,however RHV still had a negative effect on nitrate content of crop.

Nitrate was known as a major form that plants absorb nitrogen. Chung et al. (2003) reported that nitrate content of plants products depended on time of harvesting, nitrogenous fertilizers, irradiation, temperature, soil and light. Nitrate was relatively low in toxicity, but was is degraded into nitrite, which was more toxic and known carcinogenic to humans (Gangolli et al., 1994). Concentrations of nitrates and nitrites content in vegetables had become an important quality indicator (Susin, Kmecl, and

Gregorcic, 2006). Growing concern over nitrate toxicity had produced a number of research on nitrate and nitrite contents of fresh vegetable. Even though nitrate and nitrite maight be carcinogenic to human, antioxidant compounds, such as ascorbate, tocopherol, b-carotene, phenol compounds and indol, could suppress the formation of carcinogenic agents. Consumption of vegetables still could decrease in incidences of cancer (Chung et al., 2003; Correia et al., 2010). However, there was no effective database for nitrate and nitrite in vegetables. According to researches, nitrate content of vegetables had a wide variation, ranging from 10 ppm to 10000 ppm (Hord, Tang, and Bryan, 2009; Reinik, Tamme, and Roasto, 2008). The European Union also established the limit of nitrate concentration in lettuce (*Lactuca sativa L.*) depending on the season of cultivation. During summer (harvested 1 April to 30 September), the maximum levels of lettuce nitrate content was 4000 mg NO₃/kg grown under cover and 3000 mg NO₃/kg grown in open air (Commission Regulation (EU) No 1258/2011. 2011). The nitrate content of Fushan lettuce in this experiment was within this range.

The application of RHB and RHV revealed a potential in reducing nitrate content in vegetables. The decrease of nitrate content in vegetable to human helath could be further study.

4.4.6 RHB Sensory Test Result

Table 4-7 was the sensory test result of RHB experiment. The result of fresh sample in **Table 4-7** and **Fig. 4-26a** revealed that B4 had the lowest score in color, texture and flavor. It resulted in the lowest average score with a significant difference. The highest average score was at B6 that was also the first in every item. However, the result did not reveal a significant different according statistical analysis.

The cooked scores in **Table 4-7** and **Fig. 4-26b** revealed a decrease comparing to fresh sample. In color, B4 revealed a maximum decrease 1.1 while B6 only decreased by 0.1. The decrease in texture was ranged from 0.2 to 0.5 with a highest 0.5 at B2, B4 and a lowest 0.2 at Control. There was a big difference in flavor score reduction with a highest 0.7 at B4 and only 0.1 at B6. The average result of cooked sample showed a highest at B6 and a lowest at B4. The ranking of fresh and cooked sample did not change.

The sensory test result of RHB experiment, neither in fresh nor in cooked, the relationship between control and the application of RHB could not be found. According to the above results, there was no significant relationship between Control and the

application of RHB in sensory test either fresh or cooked. Therefore, it was impossible to determine whether the use of RHB would affect the taste of vegetables.

4.4.7 RHV Sensory Test Result

Table 4-7 was sensory test result of RHV experiment. The fresh sample of RHV experiment in **Table 4-8** and **Fig. 4-27a** showed that V3 had the lowest score in color, texture and flavor. It resulted in the lowest average score with a significant difference. The highest average score was at V1 that was also the first in every item. However, the result did not reveal a significant different according statistical analysis.

The cooked scores in **Table 4-8** and **Fig. 4-27b** revealed that most of the scores decreased. The score of texture and flavor of V2 remained the same. The score of texture and flavor of Control increased 0.1. The most reduction was found in V1 that the difference between fresh and cooked in color, texture and flavor was 1.9, 1.5, and 1.7, respectively. The reduction in average of each treatment was 0, 1.7, 0.17 and 0.7, respectively. The average score ranking of fresh sample was the lowest in V3, then Control and V2, with a highest in V1. But the average score ranking of cooked sample changed, of which was the lowest in V1, then V3 and V2, with a highest in Control.

According to the sensory test result of RHV experiment, the relationship between different treatments could not be found. In the comparison between RHV fresh and cooked sample, even if the score was higher when fresh, it may change after being cooked. The above results showed the effect of RHV on the taste of vegetables could not be evaluated.

	Nitrate	Nitrite	Vitamin C	Dietary fiber
	(ppm)	(ppm)	(mg/100g)	(g/100g)
Control	2360	-	-	3.1
B2	2680	-	-	2.1
B4	2120	-	-	2.8
B6	1430	-	-	2.8

 Table 4-5 RHB plant nutrition result

 Table 4-6 RHV plant nutrition result

	Nitrate (ppm)	Nitrite (ppm)	Vitamin C (mg/100g)	Dietary fiber (g/100g)
Control	2360	-	-	3.1
V1	2340	-	-	3.1
V2	765	-	-	2.8
V3	980	-	-	2.7

 Table 4-7 Sensory test result of RHB experiment

Treatment		F	resh		Cooked					
meannent				Average				-		
Control	3.7	3.4	3.3	3.47	2.8	3.2	3.1	3.03		
B2	4	3.5	3.5	3.67*	3.3	3.0	2.9	3.07**		
B4	3.6	2.8	2.7	3.03*	2.5	2.3	2	2.27**		
B6	4.1	3.7	3.4	3.47 3.67* 3.03* 3.73	4.0	3.4	3.3	3.57*		

Significance: * p<0.05; ** p< 0.01; *** p<0.001, according to Student's t-test

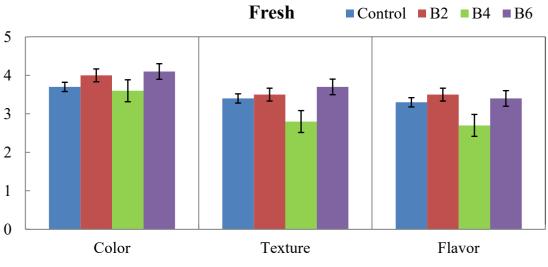


Fig. 4-39a Sensory test result of RHB experiment

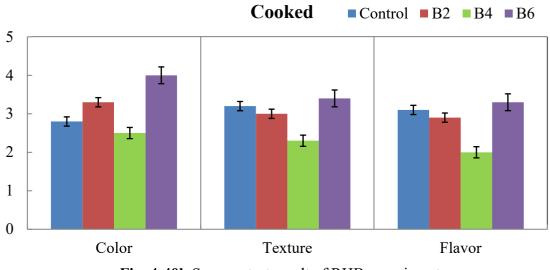
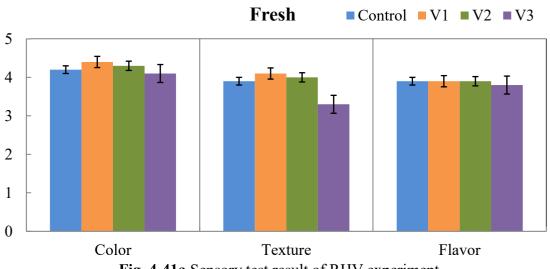


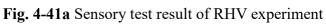
Fig. 4-40b Sensory test result of RHB experiment

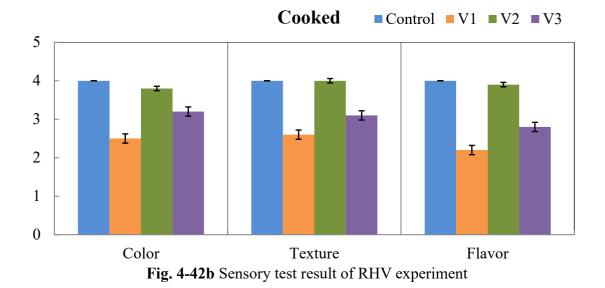
Table 4-8 Sensory test result of RHV experiment

Treatment		Fı	resh		Cooked				
meatiment	Color	Texture	Flavor	Average	Color	Texture	Flavor	Average	
Control	4.2	3.9	3.9	4.00	4.0	4.0	4.0	4.00	
V1	4.4	4.1	3.9	4.13	2.5	2.6	2.2	2.43***	
V2	4.3	4.0	3.9	4.07	3.8	4.0	3.9	3.90	
V3	4.1	3.3	3.8	3.73*	3.2	3.1	2.8	3.03**	

Significance: * p<0.05; ** p< 0.01; *** p<0.001, according to Student's t-test







4.4.8 Soil Property Result for RHB Experiment

Table 4-9 represented the soil properties by different RHB application rates after harvest and before planting. Comparing Control and Material, there was a decrease by 0.06 at EC, 0.68 at pH, 1.16 at C/N ratio and most of the element content, especially in phosphorus, potassium, calcium. The decrease might be due to absorption of crop and irrigation. However, there was an increase at SOM and nitrogen content. The increase could be considered as the effect of organic fertilizer that had a benefit of increasing SOM and can release nutrient long lasting.

In the results of soil analysis after harvest, it demonstrated that variations after the application of RHB. EC had a slightly change from 0.61 dS/m at Control and B4, increased by 0.22 at B6. There was a significant increase at pH value at all levels of RHB application rate, it increased by 0.62 from Control to B6. In addition, SOM showed a significant difference, it increased with the increase of RHB application rate. Comparing each treatment with control, it increased by 2.61 % at B2, 3.87 % at B4 and 5.45 % at B6. The result of RHB increased soil EC, pH and SOM was agreed with other researches (Ghorbani and Amirahmadi, 2018; Sang et al., 2018). With the increase of SOM, the C/N ratio also revealed an increase, form 5.46 at Control to 12.60 at B4, increasing by 7.14. However, it decreased 1.09 from B4 to B6. The application of RHB in soil had a positive effect on C/N ratio.

In **Table 4-9**, it showed the result of element content in soil. The nitrogen content for B2, B4, and B6 was 9.0 %, -8.0 %, and 40.5% comparing with Control, respectively. The phosphorus and potassium content increased with the addition of RHB. The phosphorus content of B2, B4 and B6 was 56.0 %, 26.4 %, and 40.8% higher than Control, respectively. The increase in potassium content was 83.6 %, 33.6 % and 289.7%. Although most studies have revealed that the content of N, P, and K should increase with the use of biochar (Huang et al., 2019; Oladele, Adeyemo, and Awodun, 2019; Singh et al., 2018), the increase of N, P and K did not relate to the application of RHB in this research.

According to the soil properties recommended by Tainan District Agricultural Research and Extension Station, EC should less than 0.6 dS/m, pH ranged from 5.5 to 7.5, SOM should higher than 3% and C/N ratio should less than 33. The application of RHB revealed a potential to change soil properties in EC, pH, SOM and C/N ratio. However, with the risen of EC, pH, SOM and C/N ratio, it was recommended to analyze the soil properties first before using RHB. The RHB usage in field should base on the soil analysis report of each field. In addition, the effect of RHB on soil fertility needs to be further studied.

4.5 Conclusion

RHB and RHV were used as soil amendment and plant growth promoter, respectively. Through pot experiment, the effect was revealed by studying plant biomass amount, plant nutrition, sensory test and soil property.

The result of plant biomass showed that the application of RHB had significant effect on plant growth; RHV only had slightly effect on root length, plant height and above ground weight. In plant nutrition, both RHB and RHV had an effect on nitrate content that was believed to be harmful to human health. The nitrate content of B6 and V3 was 39.4 % and 58.8 % lower than Control, respectively. However, the effect of RHB and RHV on reducing crop nitrate content should apply further study. The sensory test result revealed there was no relationship between each treatment, neither in fresh nor after cooked. Even the score was higher in fresh; it might change after cooking. The soil property result for RHB experiment showed a positive effect on EC, pH SOM and C/N ratio. The results were agreed with other researches. However, the element content in this research could not be correlated with each treatment. The effect of RHB on soil the element content can be further studied.

	EC (dS/m)	рН	SOM (%)	C/N Ratio	SAR	Ν	Р	K	Ca	Mg	Na	Zn	Cu	Fe	Mn
Material ¹	0.67	6.07	1.22	6.62	1.16	1100	336	242	2054	286	213	7.22	3.20	686	42
Control	0.61	5.39	1.76	5.46	1.01	2000	125	116	1260	188	145	4.25	2.45	467	23
B2	0.75	5.48	2.61	7.25	1.08	2180	181	213	1427	254	169	4.85	3.00	693	29
B4	0.61	5.69	3.87	12.60	1.11	1840	158	155	1156	190	155	4.19	2.46	545	27
B6	0.83	6.01	5.45	11.51	0.92	2810	176	452	1155	210	130	4.30	2.27	568	31

 Table 4-9 Soil properties of RHB experiment

Note: 1. Material is the analysis result of material soil listed in Table 4-2.

2. Unit of element content: ppm

CHAPTER 5

Conclusions and Future Prospective

In this research, rice husk was used as raw material to produce rice husk biochar (RHB) and rice husk vinegar (RHV), simultaneously, by the process of CPSICF. The efficiency of CPSICF was studied by conducting the airflow gate at different width and physical properties of RHB and RHV. It has been announced in previous research that a gate width of 5.0-7.5 mm provides a high-quality RHB and RHV.

High-quality RHB and RHV were used as soil amendment and plant growth promoter, respectively. Through pot experiment, the effect was revealed by studying plant biomass amount, plant nutrition, sensory test and soil property. The result of plant biomass showed that the application of RHB had significant effect on plant growth; RHV only had slightly effect on root length, plant height and above ground weight. In plant nutrition, both RHB and RHV had an effect on nitrate content that was believed to be harmful to human health. The nitrate content of B6 and V3 was 39.4 % and 58.8 % lower than Control, respectively. However, the effect of RHB and RHV on reducing crop nitrate content should be further study. The sensory test result revealed there was no relationship between each treatment, neither in fresh nor after cooked. Even the score was higher in fresh; it might change after cooking. The soil property result for RHB experiment showed a positive effect on EC, pH SOM and C/N ratio. The results were agreed with other researches. However, the element content in this research could not be correlated with each treatment. The effect of RHB on soil the element content should be further study.

In conclusion, CPSICF demonstrated a potential of turning rice husk into valuable products. RHB had an ability to increase crop growth, change soil property and act as a long-term soil carbon sequestration. The by-product RHV also had a potential in agriculture. Both products showed that the reuse of rice husk by CPSICF process could recycle back on agriculture use. We hope that through the results of this research, CPSICF, RHB and RHV could be extended to farmers for use. Also through cooperation with the government, we can further build an industrial structure for rice husk recycling and making use of wasted rice husk by using economically viable processes and procedures to increase its value.

However, the use of RHB and RHV on field cropping should be adjusted based on the soil conditions of the field and the crops being planted. It is recommended that before and after using RHB for planting, the soil can be sent to Agricultural Research and Extension Station in Taiwan for soil analysis to ensure the soil health. Both the use of RHB and RHV has a positive effect on crops growth, but the use of both together can be further studied.

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Appendix

Publication included in dissertation:

- Wei-Puo Kuo, Yutaka Kitamura, Yoshiyuki Hara, Ching-Chen Hsieh, Yi-Hong Lin and Chen-Pin Chen (2019): Pyroligneous Acid Produced by Rice Husk Using the Charcoal Processing System with Internal Combustion Furnace. *Agricultural Research & Technology: Open Access Journal*, 23(4), 344–350. https://doi.org/10.19080/ARTOAJ.2019.23.556238
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