PHYSICOCHEMICAL CHARACTERIZATION AND EFFECTS OF FLY ASH GENERATED FROM BIOCHAR BRIQUETTES ON THE GROWTH OF CHINESE CELERY CABBAGE (*BRASSICA RAPA* SUBSP. *PEKINENSIS*)

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Abstract. Agricultural waste biomass is considered a valuable source of eco-friendly renewable energy. Generally, biomass is processed through pyrolysis and is compressed to form biochar briquettes. After combustion, the remaining ash is the incombustible material that can be used in agriculture. The aim of this research is to investigate the physicochemical properties of fly ash derived from biochar briquettes and to evaluate its effect on the growth of Chinese celery cabbage (*Brassica rapa* subsp. *pekinensis*). Fly ash was analyzed using a scanning electron microscopy (SEM) equipped with an energy dispersive X-ray spectroscopy (EDS) and X-ray diffractometer (XRD). The results exhibited the major elements were carbon (C, 59.31%), oxygen (O, 24.48%) with additional elements including potassium (K), silicon (Si), aluminum (Al), and calcium (Ca). The XRD pattern confirmed the presence of silica (SiO_2), calcite ($CaCO_3$) and hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$). Seedlings of the tested plants were grown in pots containing varying concentrations of local soil and biochar briquette ash (soil:ash, v/v), with treatments including 100% soil (control, T1), 80:20 (T2), 60:40 (T3), and 40:60 (T4). Growth parameters improved significantly with 40% ash (T3), while 60% ash (T4) had adverse effects. These results indicated that biochar briquette ash can be effectively used as an ash-based fertilizer.

Keywords: *oil palm wastes, biomass, ash-based fertilizer, growth enhancement, leafy green vegetables*

Introduction

Thailand is a tropical country with the main economy based on agriculture. Each year, Thailand produces large amounts of agricultural wastes from harvesting and the agro-industrial processing. The biomass wastes have varied essential elements that can be applied in many beneficial materials. Most agricultural wastes consist of cellulosic compounds and are considered important renewable energy sources to replace fossil fuels (Khawkomol et al., 2021; Saberi-Riseh et al., 2024). Converting agricultural biomass into biochar briquettes is an effective waste management method, with biochar recognized as a renewable energy source. The combustion of biochar briquettes generates less pollution, resulting in lower CO, NO_x, and SO_x emissions compared to fossil fuels (Wang et al., 2017; Khawkomol et al., 2021). After the combustion of biochar briquettes, the remaining

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ash represents the incombustible component remaining after the biomaterial has been incinerated (Sarkar, 2015). Moreover, ash is generally considered to be plant nutrients source. It is typically enriched in macro and micronutrients such as calcium (Ca), potassium (K), magnesium (Mg), iron (Fe) and manganese (Mn), which are necessary for plant physiological processes (Buneviciene et al., 2021). Although biomass fly ash is rich in plant beneficial nutrients, the elemental composition varies depending on the type of agricultural wastes (Khan et al., 2021). Thus, the elemental characterization is essential before supplementing the ash as a soil supplement or in other applications.

Southern Thailand is experiencing frequent flooding and coastal erosion. The biological and physical properties of the soil is disturbed, and the beneficial nutrients is diminished by floods affecting soil fertility (Langkulsen et al., 2022; Haseena et al., 2023). Vegetable cultivation in low fertility soil, cultivators usually apply inorganic fertilizers to promote crop productivity (Mohawesh et al., 2018). Goswami et al. (2024) reported the use of coal ash to enhance growth and productivity of cabbage (*Brassica oleracea* var. *capitata*), and they found that the application of vermicomposted coal ash doubled cabbage yield. Napa cabbage, or Chinese celery cabbage (*Brassica rapa* subsp. *pekinensis*), is a type of vegetable that grows a tightly packed head of light green, curly leaves. The height of the cylindrical heads is roughly 20–30 cm. It is a popular crop in southern Thailand, exhibiting various morphotypes that differ in shape and color (Rubab et al., 2018; Punsawad et al., 2019). Not only Thailand but many countries around the world, especially in eastern Asia usually consume both fresh and pickled cabbage. The various traditional fermented cabbage products such as kimchi (Korea), pao cai (China), sunki (Japan), phak-gard-dong (Thailand) and sauerkraut (Europe). Thus, Chinese celery cabbage is an important economic vegetable with a high consumption rate (Özer and Yildirim, 2019; Yongsawas et al., 2022; Chae et al., 2024).

Agricultural soil amendment with biochar ash in appropriate concentration is useful for promote crops growth and for sustainable agriculture. However, the use of ash in agriculture depends on many factors such as types of agricultural waste, soil properties, ash concentration and types of crops in order to prevent negative effects on crops and agricultural environment in long period (Rusanescu et al., 2023). Therefore, the ultimate goals of this research were to characterize fly ash derived from oil palm frond biochar briquettes combustion and investigate its effects on the growth of Chinese celery cabbage.

Material and methods

Preparation and characterization of oil palm frond briquettes ash

Biomass briquettes were fabricated by oil palm frond combined with red clay and tapioca starch. The ratios of bio-briquettes (biochar: red clay: tapioca starch, w/w) were 60:35:5. The pyrolysis process was conducted as described by Mahmoud et al. (2024), and the briquetting was carried out using a screw briquetting machine at a pressure of 0.5 MPa (Saputro et al., 2021). The obtained briquettes did not contain any chemical additives, making their ash did not harmful to the tested plants. After combustion, the collected ash was kept at room temperature (25 °C) and dry condition for characterization. The morphological and elemental characterization were carried out by scanning electron microscope (SEM) coupled with energy dispersive X-ray spectroscopy (EDS) (JEOL JSM-IT300, Oxford X-Max 20). The gold coated samples were observed under vacuum at 10 kV. Moreover, it is also attainable to investigate the elemental distribution on the ash sample (Ngo et al., 2024). The chemical composition was examined by X-ray diffractometer (XRD, Rigaku, SmartLab), determining under conditions of 40 kV and 50 mA, with 2θ range 5–90°. The pH values and electrical conductivity (EC) of fly ash were measured by pH meter (Mettler Toledo, SevenCompact) and portable EC meter (EXTECH, EC 100) (Taupedi and Ultra Jr., 2022).

Plant materials and trial implementation

This experiment was performed in Nakhon Si Thammarat Province, southern Thailand, from August 17 to October 15, 2023. Seeds of Chinese celery cabbage (East-West Seed Ltd, Thailand) were soaked in the water for 2 h and then transferred to the nursery tray for preparation of plant seedlings. Three fifteen-day-old seedlings were planted in pots with dimensions of 19.05 × 50.8 × 16.51 cm, with three replications. The experimental pots contained different concentrations of local soil and bio-briquette ash (soil:ash, v/v), specifically soil 100% (control, T1), 80:20 (T2), 60:40 (T3), and 40:60 (T4) (Chalatchaliao et al., 2022). Soil organic carbon and organic matter were determined using a modified Walkley-Black procedure (Walkley and Black, 1934). The pH and electrical conductivity (EC) values of soil samples were measured using a pH meter (Mettler Toledo, SevenCompact) and a portable EC meter (EXTECH, EC 100), respectively (Taupedi and Ultra Jr., 2022). All experimental pots received equal irrigation of 0.75 L of water, applied twice daily. The conditions during the cultivation period were 70-85% of ambient humidity, ambient temperature at 26-30°C, and growing under natural light.

Evaluation of plants growth

After 45 days of cultivation, the tested plants were harvested from each pot trial and washed with distilled water to remove soil and other particles. The growth of the tested plants was then analyzed. Three samples were used to measure growth parameters, including plant height, root length, leaf width, and fresh weight, as described by Mitchell and Frisbie (2017) and Schrader et al. (2021).

Statistical analysis

The plant growth evaluation experiment was repeated three times. The data obtained were analyzed using one-way analysis of variance (ANOVA) with GraphPad Prism software version 10.0 (GraphPad Software, San Diego, CA, USA). Significant differences between treatment means were determined using Tukey's test at a significance level of $P \leq 0.01$.

Results

Morphological structure of biochar briquettes fly ash was investigated by SEM, and the SEM image at a magnification of 5,000 revealed an irregular and complex structure (*Fig. 1*). The ash sample combined both amorphous and crystalline structure. EDS analysis exhibited the elemental composition of biochar briquettes ash as illustrated in *Figure 2*. The EDS spectrum demonstrated the major elements consist of carbon (C, 59.31%), oxygen (O, 24.48%), and several important elements such as potassium (K, 3.35%), silicon (Si, 2.20%), aluminum (Al, 1.66%) and calcium (Ca, 1.07%) together with other minor elements. SEM-EDS examination was also used to study the distribution of elements on the ash sample and the result was shown in *Figure 3*. The result only shows spectral images for some important and the most abundant elements detected. It could be demonstrated that C (green), K (red), Al (violet), Si (orange) and Ca (purple). For chemical composition analysis, the XRD pattern confirmed the exact inorganic compounds composition of the obtained biochar briquettes ashes and the XRD diffractogram represented in *Figure 4*. The XRD diffractogram showed the presence of calcite ($CaCO_3$), silica or quartz (SiO_2) and hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$).

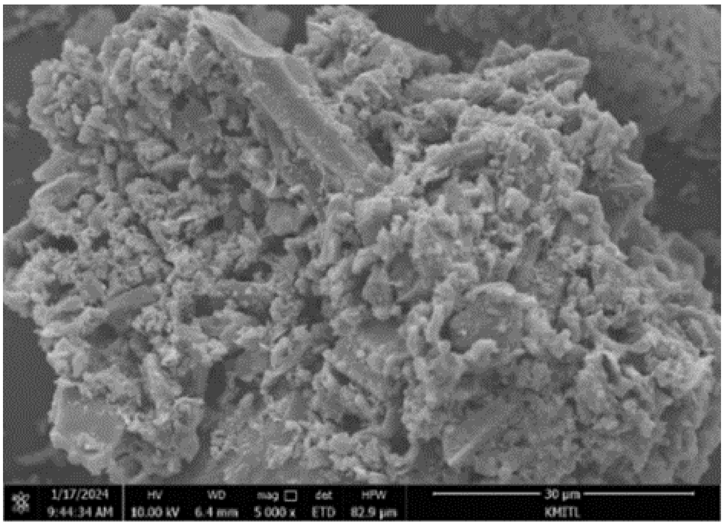
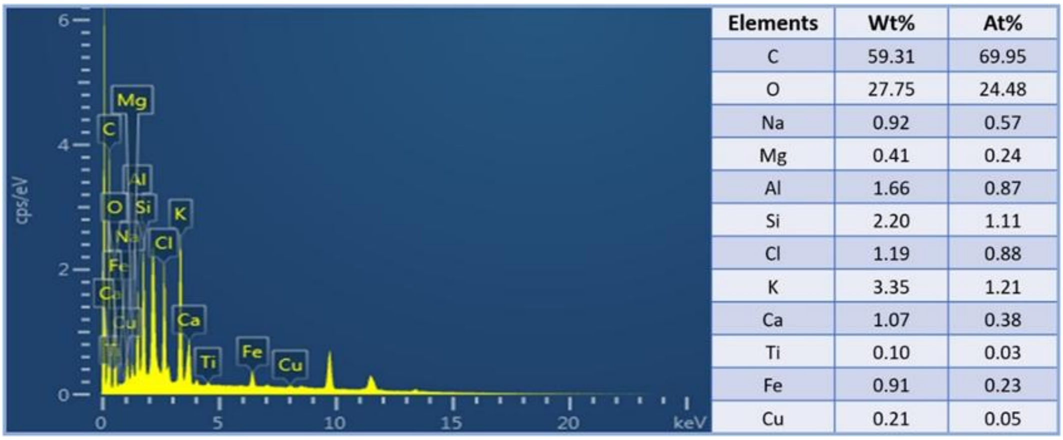


Figure 1. *Scanning electron micrograph of fly ash generated from oil palm frond biochar briquettes combustion with ×5,000 magnification (Sale bar = 30 μm)*



Elements	Wt%	At%
C	59.31	69.95
O	27.75	24.48
Na	0.92	0.57
Mg	0.41	0.24
Al	1.66	0.87
Si	2.20	1.11
Cl	1.19	0.88
K	3.35	1.21
Ca	1.07	0.38
Ti	0.10	0.03
Fe	0.91	0.23
Cu	0.21	0.05

Figure 2. *Energy dispersive spectrum and elemental profile of fly ash from oil palm frond biochar briquett*

Soil organic carbon and organic matter content of the planting soil were 0.89% and 1.53%, respectively. Additionally, pH and electrical conductivity (EC) values of the planting soil for each treatment were measured, with the average values presented in *Table 1*. As the percentage of biochar briquette ash increased, both the pH and EC values exhibited a corresponding increase.

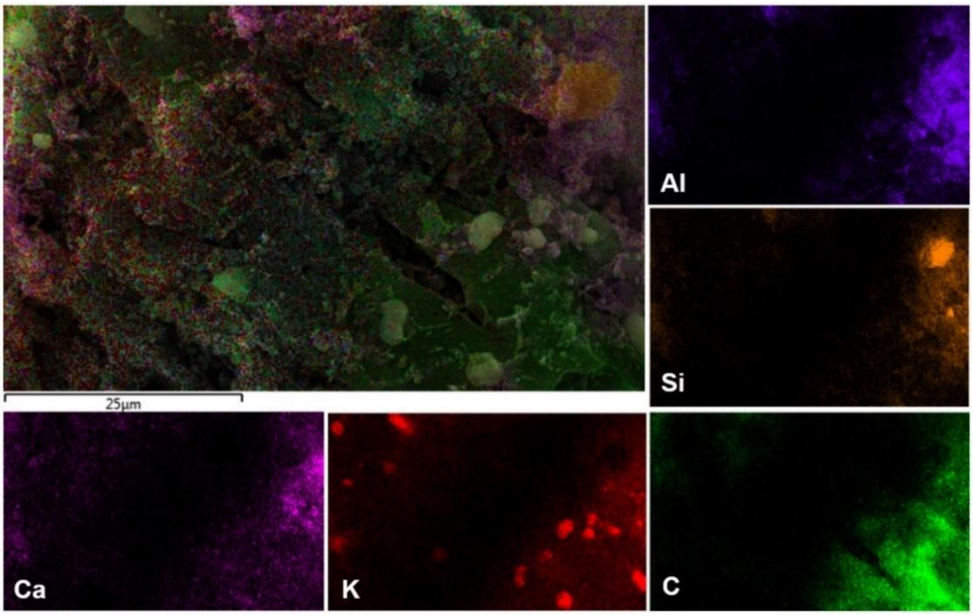


Figure 3. *Elemental mapping (Ca, K, C, Si and Al) of fly ash obtained from biochar briquettes (Scale bar = 25 µm)*

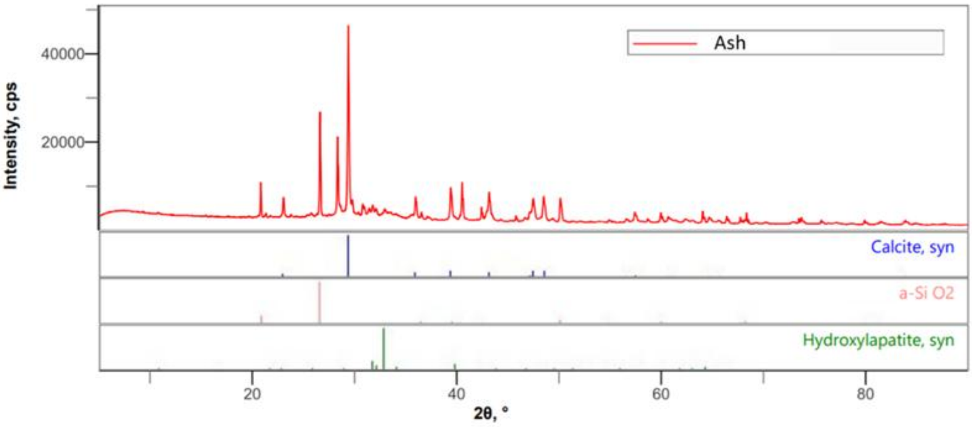


Figure 4. *X-Ray diffractogram of oil palm frond ash from biochar briquette combustion*

Table 1. *The pH and EC value of planting soil for each treatment*

Treatments	pH	EC (ds/m)
T1: soil 100% (control)	6.71	0.29
T2: soil 80%:ash 20%	7.20	1.23
T3: soil 60%:ash 40%	7.72	2.26
T4: soil 40%:ash 60%	8.97	2.83

Evaluation of Chinese celery cabbage growth, the application of biochar briquettes ash had a significant effect on growth parameters of tested plant such as plant height, root length, leaf width and fresh weight. These growth parameters were evaluated after 45 days of cultivation. *Figure 5* shows the growth effects of tested plant in various concentration of fly ash. The results exhibit that the widest leaf diameter at 8.33 cm were found at the concentration of 40% ash (T3), and the leaf width of Chinese celery cabbage grown in different concentration of biochar briquette ash were illustrated in *Figure 6(a)*. The highest value of plant height and root length was also observed at an ash concentration of 40% with value of 25.66 cm and 7.66 cm, respectively. Moreover, fresh weight is considered to the productivity of leafy vegetables. The maximum fresh weight was 51.66 g at the same ash concentration. The differences in growth characteristics of Chinese celery cabbage grown in different concentration of biochar briquette fly ash were shown in *Figure 6(b)*.

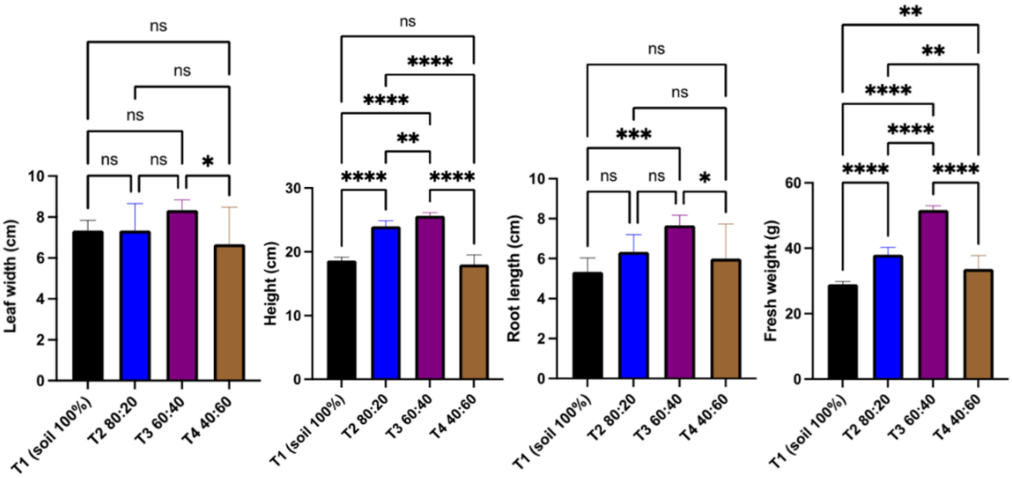


Figure 5. *The growth response of Chinese celery cabbage in various concentration of ash. (T1: soil 100% (control); T2: soil 80: ash 20; T3: soil 60: ash 40 and T4: soil 40:ash 60)*

Figure 6. *The leaf width of Chinese celery cabbage grown in different concentration of biochar briquette fly ash (a) and the growth characteristic of Chinese celery cabbage grown in different concentration of biochar briquette fly ash (b). (T1: soil 100% (control); T2: soil 80:ash 20; T3: soil 60:ash 40 and T4 : soil 40:ash 60)*

Discussion

Morphological investigation revealed the scanning electron micrograph of fly ash showed many irregular shapes, possibly due to the low combustion degree of the biochar briquette; however, some crystalline structures were found. The strong peaks of Si, Ca, Mg and O confirms the metal oxide compounds in the ash sample (Chen et al., 2022). Murthy et al. (2014) reported that the morphological structure of ash is depending on the combustion temperature, while the SEM-EDX analysis indicated that the biochar briquettes ashes contain a broad range of essential inorganic elements for plant physiological processes. The spectrum exhibits that C and O are the major elements in the ash, whereas Ca, K, Na, Si and Al were also detected (*Fig. 2* and *Fig. 3*). Some of trace elements represented amounts of alkali metals such as Al, Ca, K, Mg and Na. These metals could increase the alkalinity of the planting soil (Mohammadi et al., 2022). XRD diffractogram of biochar briquettes ash exhibited the identical peaks of calcite, silica and hydroxyapatite. The results agree with the report of Du et al. (2024), they reported that silica and calcite are the major components in most of wood ash. The obtained inorganic compounds can be used in agricultural application (Grafmüller et al., 2022; Damayanti et al., 2023).

The addition of 40% ash in the planting soil resulted in an 78.14% increase in the average fresh weight of the tested plants compared to the control. This result is connected to pH and EC value of the soil. The biochar briquette ash enhanced the pH of the planting soil by a few units, while the increased EC value indicated a rise in soil nutrients (Chima-Ezika et al., 2024; Kim and Park, 2024). Low EC in the soil can lead to nutrient deficiencies in plants (Zhang and Li, 2015). The 40% ash concentration showed the highest yield, likely due to the readily bioavailable elemental nutrients from the ash. However, a high value of EC can adversely affect to plant. Extremely high pH and EC due to ash amendments was probably related to poor bioavailability of some inorganic nutrient by increasing the osmotic pressure (Ding et al., 2018). This finding is consistent with the result in pumpkin (*Cucurbita moschata* Duch. Ex Poiret L.) where ash concentrations above 40% had negative effects, reducing growth and the content of photosynthetic pigments (chlorophyll, carotenoids) (Ahmad et al., 2021).

In addition, inorganic minerals found in the ash play an important role in plant physiological process and have a direct effect on growth and yield of chinses celery cabbage. Hydroxyapatite is a major source of macro-element, which was a vital nutrient required for leafy vegetable growth. Cruz-Hernández et al. (2023) studied the effect of hydroxyapatite on lettuce plant growth, and they noted that hydroxyapatite promoted a 275% to 350% increase in lettuce height at 30 days compared to control. Hydroxyapatite is also reported to be the Ca source. This trace element is involved in promoting the photosynthesis process of plants by regulation of proteins in the photosynthesis reaction (Chong et al., 2023). In this research, the tested plants obtained silicon (Si) from silica. Silica improves plant stem erection and increases resistance to physical stress such as nutrient imbalances and heavy metal-induced stress (Sadaf et al., 2024). Previous research reported that Si can increased contents of carotenoids and chlorophyll (a and b) and decreased oxidative damage by increasing activity of antioxidation enzymes in plants (Neto et al., 2020). Additionally, acidic soil is a major problem in the southern region of Thailand. To solve this problem, biochar briquette ash is amened to increase the alkalinity of the planting soil. Calcite is another main component was detected in the biochar briquettes fly ash (*Fig. 4*). It occurs naturally in plant tissue and is also found in plant biomass ash (Neina et al., 2020; Smołka-Danielowska and Jabłońska, 2022). Calcite was reported to be an excellent source of importance nutrients. Kim et al. (2022) stated that the biomass ash application at an appropriate dose can be used for amelioration of acidic soil. Microorganism proliferation in the rhizosphere is positively impacted by raising the pH of the soil. Next-generation sequencing technology was used by Błonska et al. (2023) to investigate the impact of wood ash amendment on the microbial population. They discovered that the short-term use of wood ash enhanced the microbial population. However, the application of high concentrations of wood ash (6 t/ha) had a huge impact on mycorrhizal fungi, which are soil fungi that play an important role in plant nutrients solubilization. This study aligns with the findings of Szostek et al. (2023), who reported that the short-term use of fly ash has positive effects on agriculture, whereas long-term use should account for potential risks. The application of high concentration of ash may result in excessive alkalinity and salinity of soil environment. Therefore, the use of ash-based fertilizer should take into account its physicochemical properties, concentration and appropriate duration to avoid soil pollution.

Conclusion

In conclusion, we have demonstrated that the ash from biochar briquettes combustion is an excellent source of several inorganic minerals such as silica, calcite and hydroxyapatite that Chinese celery cabbage need to grow and thrive. The use of ash based-fertilizer has shown many potentials for leaf green vegetable cultivation include provides essential nutrients, increases pH and EC of planting soil. The optimum concentration for Chinese cabbage was 40%, which was the concentration level that shows the highest average fresh weight. Increasing the concentration more than this might reduce the yield. However, the long-term use of ash-based fertilizers might lead to soil pollution and soil fertility. Thus, further studies on the use of biochar briquettes ash as fertilizer are necessary because the suitable concentrations and types of elements may differ depending on economic vegetables.

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APPENDIX

Table A1. *Analysis of variance (ANOVA) for leaf width*

Tukey's multiple comparisons test	Mean Diff.	99.00% CI of diff.	Below threshold?	Summary	Adjusted P Value
T1 (soil 100%) vs. T2 80:20	0.000	-1.866 to 1.866	No	ns	>0.9999
T1 (soil 100%) vs. T3 60:40	-1.000	-2.866 to 0.8656	No	ns	0.2878
T1 (soil 100%) vs. T4 40:60	0.6667	-1.199 to 2.532	No	ns	0.6276
T2 80:20 vs. T3 60:40	-1.000	-2.866 to 0.8656	No	ns	0.2878
T2 80:20 vs. T4 40:60	0.6667	-1.199 to 2.532	No	ns	0.6276
T3 60:40 vs. T4 40:60	1.667	-0.1989 to 3.532	No	*	0.0245

Table A2. *Analysis of variance (ANOVA) for plant hight*

Tukey's multiple comparisons test	Mean Diff.	99.00% CI of diff.	Below threshold?	Summary	Adjusted P Value
T1 (soil 100%) vs. T2 80:20	-5.333	-6.822 to -3.845	Yes	****	<0.0001
T1 (soil 100%) vs. T3 60:40	-7.000	-8.488 to -5.512	Yes	****	<0.0001
T1 (soil 100%) vs. T4 40:60	0.6667	-0.8215 to 2.155	No	ns	0.4425
T2 80:20 vs. T3 60:40	-1.667	-3.155 to -0.1785	Yes	**	0.0034
T2 80:20 vs. T4 40:60	6.000	4.512 to 7.488	Yes	****	<0.0001
T3 60:40 vs. T4 40:60	7.667	6.178 to 9.155	Yes	****	<0.0001

Table A3. *Analysis of variance (ANOVA) for root length*

Tukey's multiple comparisons test	Mean Diff.	99.00% CI of diff.	Below threshold?	Summary	Adjusted P Value
T1 (soil 100%) vs. T2 80:20	-1.000	-2.687 to 0.6875	No	ns	0.2092
T1 (soil 100%) vs. T3 60:40	-2.333	-4.021 to -0.6459	Yes	***	0.0003
T1 (soil 100%) vs. T4 40:60	-0.6667	-2.354 to 1.021	No	ns	0.5491
T2 80:20 vs. T3 60:40	-1.333	-3.021 to 0.3541	No	ns	0.0550
T2 80:20 vs. T4 40:60	0.3333	-1.354 to 2.021	No	ns	0.9088
T3 60:40 vs. T4 40:60	1.667	-0.02080 to 3.354	No	*	0.0111

Table A4. *Analysis of variance (ANOVA) for fresh weight*

Tukey's multiple comparisons test	Mean Diff.	99.00% CI of diff.	Below threshold?	Summary	Adjusted P Value
T1 (soil 100%) vs. T2 80:20	-9.000	-12.94 to -5.063	Yes	****	<0.0001
T1 (soil 100%) vs. T3 60:40	-22.67	-26.60 to -18.73	Yes	****	<0.0001
T1 (soil 100%) vs. T4 40:60	-4.667	-8.604 to -0.7292	Yes	**	0.0019
T2 80:20 vs. T3 60:40	-13.67	-17.60 to -9.729	Yes	****	<0.0001
T2 80:20 vs. T4 40:60	4.333	0.3959 to 8.271	Yes	**	0.0041
T3 60:40 vs. T4 40:60	18.00	14.06 to 21.94	Yes	****	<0.0001