Inertization of metals and hydrogen production as a byproduct from water hyacinth and water lettuce via plasma pyrolysis

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Water hyacinth and water lettuce have been extensively used for phytoremediation of metals and metalloids. However, the reasonable disposal of phytoremediation plants is a difficult problem. This study aims to reduce metals and metalloids from water hyacinth and water lettuce, and produce hydrogen (H₂) and methane (CH₄) via an atmospheric-pressure microwave plasma reactor based on the circular economy concept. Inertization of metals and metalloids can be obtained by more than 60% for both water hyacinth and water lettuce. H₂ and CH₄ production of water hyacinth and water lettuce were 56.28%/57.30% and 3.75%/2% of volume fractions, respectively. Furthermore, total VOCs concentrations from the effluent gas were detected only at the values of 0.511% (water hyacinth) and 0.08% (water lettuce) of volume fractions, respectively. Overall, water hyacinth and water lettuce treated by atmospheric-pressure microwave plasma showed the potential of H₂ and CH₄ production as a by-product for alternative energy and inertization of metals/metalloids for the phytoremediation plants.
for obtaining gases with fast heating performances, robust systems, and less time-consuming operations that are suitable for supplying the demand of industries should be recommended.

Plasma technology has been used in the treatment of hazardous elements from solid waste and biomass conversion to obtain gases (Lin et al., 2014b; Cubas et al., 2015). Pyrolysis of plasma jets is a promising thermochemical conversion technology for recovering energy from biomass because of the biomass transformation to gases (H2, CH4, and syngas) (Chen et al., 2008; Lei et al., 2009; Bu et al., 2012). Lin et al. (2014b) studied that pyrolysis of dry banyan in an atmospheric-pressure microwave plasma reactor to generate the H2, CO2, and CO fractions with 90.57%, 91.16% and 91.25% at 800, 900, and 1000 W, respectively. Lei et al. (2009) stated that 42% of biogas yield can be achieved at 650 °C in 8 min of pyrolysis. Thus, plasma gasification technology is the most effective and environmentally friendly procedure for generating gases at the high temperatures. In the treatment of toxic metals from hazardous waste, Cubas et al. (2015) reported that Mn and Zn can be eliminated with a removal efficiency of more than 96% via plasma technology in a moist paste battery. Likewise, Cubas et al. (2014) stated that Cr, Fe, and Zn can be obtained with values of 100%, 100%, and 99%, respectively, in the treatment of galvanic sludge in plasma technology. Currently, there is no report of the use of plasma technology focusing on the treatment of water hyacinth and water lettuce for generating H2 and degrading elements, where the previous study focused on pyrolysis (Li et al., 2021), solar furnaces (Huang et al., 2018), tube furnaces (Liu et al., 2017a), and incinerators (Zhu et al., 2019). Thereby, plasma technology can be considered as a proper technology to deal with hazardous elements and biomass gasification.

Despite plasma technology shows a promising results on degrading metals, metalloids, and H2 as the byproduct from biomass, the interactions between pyrolysis on water hyacinth and water lettuce via an atmospheric-pressure microwave plasma reactor are not fully understood. Thus, a study focused on the treatment of water hyacinth and water lettuce via plasma technology was performed.

This study was based on the circular economy concepts. Inertization of metals and metalloids, and potential production of H2 from the treatment of water hyacinth and water lettuce as the byproduct via an atmospheric-pressure microwave plasma reactor were investigated. Furthermore, the production of gases, such as H2 from the treatment of water hyacinth and water lettuce in an atmospheric-pressure microwave plasma reactor as an alternative fuel, is still unknown. Plants from the post-phytoremediation are also used in co-composting (Song and Park, 2017), compaction (Mohanty, 2016), and incineration (Liu and Tran, 2021). Moreover, biomass from water hyacinth and water lettuce can be considered for replacing the fossil fuels from the post-phytoremediation. Also, bio-chars can be generated from the pyrolysis process (Figure 1).

Therefore, using water hyacinth and water lettuce is an alternative procedure to reuse the biomass and produce gases, while at the same time, inertization of metals and metalloids can be performed via plasma technology. Lastly, volatile organic compounds (VOCs) from pyrolysis treatment are also being assessed in this study to provide important information about biomass pollutants from the plasma pyrolysis process.

2. Materials and methods

2.1. Collection and preparation of samples

Approximately 1 kg of water hyacinth and water lettuce was collected freshly from the river near Taoyuan City Park, Taoyuan, on January 15th, 2020. The specific parks of Taoyuan city park are Qingbugong Park and Chintan Park. The latitude and length of Qingbugong park are 25.01° and 121.21°, respectively. The latitude and length of Chintan Park are 25° and 121.20°. Latitude and length show the geocoordinate locations. The Universal Transverse Mercator (UTM) of Qingbugong park and Chintan park is described as follow: 319 km east and 2767.73 km north for Qingbugong park and 318,941.83 km east and 2,766,901.56 km north for Chintan Park. The google earth software was used to obtain the specific geocoordinates and locations (Figure S1). Water hyacinth and water lettuce were transported in plastic bags. After that, they were immediately washed to clean sediments from roots and leaves, which can cause mechanical problems. In the preparation of feed stocks of water hyacinth and water lettuce, petioles, roots and leaves are mixed. Leaves and petioles of water hyacinth were also prepared separately as the samples.

There are two types of samples, namely dry and wet samples. In preparation for the samples, pretreatments were performed for obtaining fresh slurry and dry powder of water hyacinth and water lettuce. For slurry, the rhizome and leaves are roughly chopped by knife, and crushed with kitchen blenders. All parts of water hyacinth and water lettuce were dried in an oven for 24 h at a temperature of 100 °C for 2 days to obtain the dry powder, then crushed with a pulverizing machine (Rong Tsong 0-2B 133 Taiwan) to obtain a smaller size, generally <100 mm. Furthermore, 200 mesh powder was obtained by sieving. All types of samples were labeled in 50 mL containers. The dry samples and wet samples were saved at room temperature (25 °C) and refrigerator (4 °C), respectively, until they were required in experiments. Information about sample collection is shown in Figure 2.

2.2. Analysis of gas product and volatile organic compounds

For analyzing the hydrogen (H2), methane (CH4), carbon monoxide (CO), and carbon dioxide (CO2), respectively, the gas chromatography (GC-2030-Nexis Shimadzu) with the column barrier discharged ionization detector (BID) and micropacked ST were used. The length, inner diameter and film thickness for both dummy and real columns were 250 m, 0.5 mm, 10 μm, and 2 m, 1 mm, 10 μm, respectively. Helium (He) was purged into the system as a carrier gas. The temperature program was maintained at 35 °C–280 °C. The rate was controlled at 14–60 °C/min. The total time of analysis was 15 min. Furthermore, the injector temperature and BID temperature were controlled at 150 °C and 280 °C, respectively. The injection mode was controlled by the split mode. The pressure, column flow, linear velocity and purge flow were controlled at 303.1 kPa, 11 mL/min, 36.7 cm/s, and 3 mL/min, respectively. The discharge flow and BID temperature were maintained at 80 mL/min and 280 °C, respectively. A 0.99 R2 value was obtained from the analysis, confirming the fit model of gas test standards. The percentage compositions of each gas in L gas bags were calculated as follows:

\[
\text{% gas} = \frac{\text{Concentration of gas}}{\text{Total concentration of gas}} \times 100\%
\]  

(1)

To investigate VOCs from the treatment of water hyacinth and water lettuce from pyrolysis, emissions were captured using a 1 L gas bag collector. A gas chromatography (GC; Agilent 6890 N-USA) was used to determine the VOCs composition in the treatment. The GC oven temperature was set up at 32 °C and increased to 200 °C after 3 min after being turned on. The auto-sampler was an Entech 7032AB and the GC column size was 60 mm × 0.25 mm x 1.00 μm R2 > 0.99 showed good linearity to gas standards.

2.3. Elements in water hyacinth and water lettuce

Investigations of the initial concentration and final concentration of water hyacinth and water lettuce were performed based on Taiwan Standard NIEA S321.63B (TEPA, 2019; Sanito et al., 2020). First, 3 g of the sample was placed in a 500 mL flask. Then after, 21 mL of HCl 37% and 7 mL of HNO3 70% (3:1 v/v) were added into the 500 mL flask in a batch for 16 h for the chemical reaction to occur. A strong oxidation process from HCl and HNO3 indicates a good performance for digestion of elements.

A soxlet extractor was used to heat the samples for 2 h to purify elements (metals and metalloids). In addition, aqueous solutions were
transferred into a 50 mL reaction tube and placed in a centrifuge machine (Kubota 2000, Japan). Aqueous solutions from previous processes were centrifuged at 2500 rpm for 10 min to separate and remove particles. The purpose of this process is to prevent mechanical problem in element analysis. The supernatant in the aqueous solution was filtered through a 0.45 μm Polyvinylidene difluoride (PVDF) hydrophilic syringe filter (Thermofisher, USA) with a 25 mm diameter to separate the particles using a 25 mL syringe (Terumo, Taiwan). Then afterward, precipitates were discarded from it.

To determine the concentration of hazardous elements in the water hyacinth and water lettuce, an inductively coupled plasma atomic emission spectroscopy (ICP AES; Shimadzu ICPE 9820, Japan) was used, considering dilution factor. Argon gas, auxiliary gas, and carrier gas were controlled at 10 L/min, 0.60 L/min, and 0.31 L/min, respectively. ICP AES power and

![Figure 1. The illustration of circular economy concept of water hyacinth and water lettuce for the environmental application.](image)
exposure time were controlled at 1.20 kW and 30 s, respectively. The multi-elements standard solution (MERCK, Germany) was used for ICP AES, containing 5, 2.5, 1.25, 0.625, and 0.3125 mg/L (Sanito et al., 2020). The multi-element standard solutions were serially diluted. All the elements in the sample were moved from a solid phase to a liquid phase during the ICP-AES test, which shows that they are all in the samples.

2.4. Characterization analysis

The surface morphology and elemental composition of samples were analyzed using scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX; Hitachi S-4800, Japan). The purpose is to provide the information of elemental composition. In this analysis, only the best results of samples were analyzed. The elemental analyzer was used for analyzing the carbon, nitrogen, and hydrogen composition of the water hyacinth and water lettuce.

To analyze the functional groups available in the sample before and after treatment in an atmospheric-pressure microwave plasma reactor, fourier-transform infrared spectroscopy (FTIR: FT/IR-6600 JASCO, USA) was used in the wavenumber range of 400–4000 cm\(^{-1}\). Samples were pelleted with KBr. Then, it was pressed using a manual hand press until a 1 mm thickness of samples was obtained. A scanning of samples was performed in the FTIR instrument for 5–8 min.

Characterization of crystalline structures from water hyacinth and water lettuce was performed by X-ray diffraction (XRD; Bruker D8 Advance Eco, Germany). The X-rays scanned the samples for 5 min with \(2\theta\) scanning from 10° to 80°. The X-ray source was from asymmetric diffraction from the modular components. It was controlled at value of 40 kV, and an electric current was set up at a value of 25 mA. Then, the power was set up at 1000 W. The \(2\theta\) peaks represent the crystalline structures of the final residue from the material. All the characterization was performed only for the best samples.
2.5. Pyrolysis of water hyacinth and water lettuce samples

The pyrolysis of water hyacinth and water lettuce was performed in a crucible with a volume of 20 cm³. The height of the crucible is set at 4 cm and diameter of the crucible is 2.5 cm. Size of quartz tube is 0.1 cm thickness, 3 cm in diameter, and 32 cm in the length. The jet discharge was generated from the magnetron where the quartz tube was positioned. In this experiment, each sample of water hyacinth and water lettuce was used with a weight of 2 g, respectively. The information of samples in this experiment is shown in Table S1.

Figure 3 illustrates a schematic of the atmospheric-pressure microwave plasma reactor system in this experiment. H₂ can be obtained from the plasma treatment.

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3. Results and discussion

3.1. Functional groups of water hyacinth and water lettuce before treatment

Figure 4 illustrates the information of functional groups of water lettuce and water hyacinth before treatment via an atmospheric pressure microwave plasma reactor system. H₂ can be obtained from the plasma treatment. 

The thermochemical conversion of biomass via pyrolysis for generating hydrogen is explained as follow (Demirbas and Arin, 2004; Tran et al., 2021):

\[
\text{Pyrolysis of biomass} \rightarrow \text{H}_2 + \text{CO}_2 + \text{CO} + \text{CH}_4 + \text{Char} \quad (R1)
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1458.88 cm\(^{-1}\) confirm the presence of lignin. C–H bonds are contributed by wavenumbers in the 1300–1500 cm\(^{-1}\) ranges, respectively. C–H functional groups from all parts of plants might promote the H\(_2\) gases in plasma pyrolysis. Furthermore, wavenumbers from 782.95 cm\(^{-1}\) to 909.27 cm\(^{-1}\) represent the SiO\(_2\) (Ibrahim et al., 2015). Preethi et al., (2015) stated that functional groups, such as –OH, C=O, and C–H, can be found in water hyacinth. Wavenumber bands from 1059.69 to 1286.28 cm\(^{-1}\) were C–O groups, confirming cellulose from water hyacinth and water lettuce (Yang et al., 2007; Wu et al., 2013). C–C functional groups were shown at 1020 cm\(^{-1}\)–1250 cm\(^{-1}\), confirming the CO production (Huang et al., 2016). A study by Tran et al. (2017) stated that –OH hydroxyl groups can be found widely in the golden shower fruits, ranging from 3409 to 3417 cm\(^{-1}\). The existence of –OH groups ranging from 3300 to 3500 cm\(^{-1}\) (Sukhawipat et al., 2021). Tran et al. (2021) also stated that the –OH hydroxyl groups from water hyacinth can be detected at 3450 cm\(^{-1}\). In this study, –OH hydroxyl groups can be detected in the bio-chars from the plasma post–treatment of water hyacinth and water lettuce (Figures S2 and S3). Thereby, the wavenumber detections of C, H, and O represent the structure formation of lignocellulose.

Other striking findings of leaves and petioles of water hyacinth indicated the different transformation chemical structures (Figures S4 and S5). Higher set up flow rate of gas (12 SLM) and microwave power (1200 W) showed the high transformation of –OH hydroxyl group of water hyacinth (petioles and leaves). Furthermore, the NH chemical structure was detected at 1550–1650 cm\(^{-1}\) from leaves. It confirms that the leaves of water hyacinth absorb nitrogen from the water bodies. Fox et al. (2008) found that 60–85% of nitrogen can be absorbed from the water bodies by water hyacinth. Furthermore, water hyacinth is also recognized for its crucial role to remove ammonia from the water bodies (Ting et al., 2018). Other functional groups that can be found in the leaves and petioles of water hyacinth, such as C–O–C and C–O sym were detected between 1090–1020 cm\(^{-1}\) and 1624.7 cm\(^{-1}\), respectively. It confirms the composition of carbon in the materials that exists in water hyacinth.

Cellulose is a long polymer of glucose without branches where the structures are well-organized and extremely strong. Furthermore, cellulose has a high thermal stability. On the contrary, hemicellulose consists of different types of saccharides, such as xylose, mannose, glucose, and galactose. Moreover, it contains of many branches and amorphous structures. Thus, hemicellulose is easier to be degraded as the CO, CO\(_2\) and other hydrocarbons. Due to the abundance of aromatic rings with diverse branches in lignin, the activity of the chemical bonds was quite broad for the lignin degradation over a wide temperature range (100–900 °C) (Yang et al., 2007).

According to Leins et al. (2015) lower gas flow rates and higher microwave powers setup confirmed the transformation of plasma ignition, creating the particle collision of the atom from plasma ignition to pollutants. As a result, a transformation of chemical structure occurs (Mattox, 2010; Sanito et al., 2020). Therefore, the plasma technology converts complex structures into simpler compounds significantly, comparing to the setup of flow rate and microwave power at 6 SLM/800 W, 6 SLM/1000 W, 6 SLM/1200 W, 9 SLM/800 W and 9 SLM/1200 W, respectively.
3.2. Crystalline structure of water hyacinth and water lettuce

The characterizations of the raw material of crystalline structures from water hyacinth and water lettuce before the treatment are shown in Figure 5(a–d). From the analysis, some peaks can be found from the water hyacinth, such as petioles, leaves, and water lettuce. The presence of a peak confirmed the appearance of carbon structure from samples. Das et al. (2014) confirmed that the main composition of carbon (C), with a mass percentage of 52.88%–62.43%, contains the lignocelluloses, namely, cellulose, hemicellulose, and lignin. The crystalline structures of lignin and hemicellulose are amorphous. For this reason, hemicellulose and lignin are not detected in the XRD. In contrast, the structure of cellulose can be detected because of its non-amorphous structure. Thereby, the peaks detected from the XRD represent the cellulose (Maulina et al., 2019). The presence of carbon is also explained by the results of elemental analyzer, where percentages of C in water hyacinth and water lettuce were 36.20 ± 0.014 wt % and 33.60 ± 0.127 wt %, respectively (Table 1).

Figures 6 and 7 show the information of crystalline structures from water hyacinth and water lettuce (before and after treatment), respectively. It can be seen that both materials from this species show identical morphological structures. The structures of material were irregular and thicker. Furthermore, the structure is hollow (Zhang et al., 2015). Pratama et al. (2020) confirmed that the thicker structure of water hyacinth occurs because of its hemicellulose and cellulose from ash structures. This analysis confirms that the water hyacinth and water lettuce structures contain lignocellulose. The main compositions of carbon after treatment in plasma via different types of carrier gases, especially, in water hyacinth and water lettuce, were 40.1% (nitrogen) and 34% (argon), and 34% (nitrogen) and 37.8% (argon), respectively. The high composition of C is probably because of the transformation of material to become ash. Thereby, the percentage of carbon can be detected from a sample larger than the main materials.

3.3. Inertization of harmful elements in water hyacinth and water lettuce

Figure S6 shows the initial concentration of water hyacinth and water lettuce, respectively, before and after treatment via an atmospheric-pressure microwave plasma reactor. Figure S6a, S6b, S6c, and S6d give the information of mixed dry water lettuce powder, mixed dry water hyacinth powder, mixed fresh water lettuce, and mixed fresh water hyacinth.
water hyacinth, respectively. 63.15 ± 3.15 ppm of As can be obtained from water hyacinth with the highest concentration. Other elements, such as Cd, Cr, Cu, Pb, Ni and Zn were 20.8 ± 1.57, 16.25 ± 0.81 ppm, 12.13 ± 0.66 ppm, 23.3 ± 1.12 ppm, 24.3 ± 1.20 ppm and 31.4 ± 1.56 ppm, respectively. Moreover, the highest concentration of As in water lettuce was achieved at a value of 63.6 ± 3.85 ppm. Cd, Cr, Cu, Pb, Ni, and Zn can be obtained with values of 20.35 ± 1.01 ppm, 15 ± 0.75 ppm, 13.65 ± 0.65 ppm, 28.75 ± 1.43 ppm, 26.8 ± 1.34 ppm, and 49.05 ± 2.85 ppm, respectively. Interestingly, initial concentrations of all elements from fresh water hyacinth and fresh water lettuce were less than 30 ppm (Figure S6c and S6d). Figure S6e and S6g also indicates that dry leaves and petioles of water hyacinth element concentrations were higher than fresh water hyacinth leaves and fresh water hyacinth petioles (Figure S6f and S6h). The higher concentrations of elements in dry water hyacinth probably due to the efficient extraction from the smaller surface area of the material compared to the wet samples.

The concentrations of elements from water hyacinth leaves, such as As, Cd, Cr, Cu, Pb, Ni, and Zn were 54.5 ± 2.72 ppm, 19.67 ± 0.98 ppm, 13.37 ± 0.65 ppm, 16.7 ± 0.88 ppm, 29.67 ± 1.45 ppm, 21.47 ± 1.07 ppm and 33 ± 1.65 ppm, respectively. Water hyacinth petioles consist of elements, namely As, Cd, Cr, Cu, Pb, Ni, and Zn were 48.77 ± 2.43 ppm, 9.67 ± 0.48 ppm, 13.23 ± 0.65 ppm, 7.51 ± 0.35 ppm, 17.53 ± 0.85 ppm, 21.2 ± 1.01 ppm, and 35.10 ± 1.75 ppm, respectively. Adeyami and Osibor (2016) reported elements, namely Cd, Cr, Cu, Co, and Zn ranging 0.001–0.13 mg/kg. Lu et al. (2011) and Rezania et al. (2015a, b) confirmed that water hyacinth absorbed high concentrations of harmful elements from the water bodies, namely Cd, Cr, Cu, Co, Fe, Mg and Zn. Element concentrations in the leaves of water hyacinth were higher than petioles of water hyacinth. It confirms that elements are translocated in this part and accumulated in the xylem of plant and translocation of elements from the roots to aerial parts of the plant (Yan et al., 2020). Water hyacinth and water lettuce showed a good ability for absorbing metals,
and with great translocation results in plants. Figures S7 and S8 explain the further information on the effect of gas flow rates on the inertization of elements in each part of water hyacinth (leaves and petioles).

Figures S9 and S10 display the final residue of water hyacinth and water lettuce derived from plasma treatment. Material from the plasma post-treatment shows the transformation of material to bio-char. The higher set up of flow rate and microwave power indicated the complete transformation because of the presence of black color. Figures 9 (a–f) and 10 (a–f) show the removal efficiency of elements from water lettuce and water hyacinth, where the flow rate and microwave power were 6 SLM, 9 SLM, 12 SLM and 800 W, 1000 W and 1200 W, respectively. The result showed that the inertization of some elements, such as As, Cd, Co, Cr, Cu, Ni, and Zn in the final residues of water hyacinth and water lettuce from the plasma post-treatment can be obtained at 60%–80%, where the higher set up of microwave power at 1200 W and 12 SLM. The removal of Cd from the dry water hyacinth can be obtained by nearly 100%. Tables S2–S7 show a final concentration of water hyacinth and water lettuce in dry samples and wet samples from each part of plants. Dry samples of water hyacinth and water lettuce indicate a better result on the detection of elements because of their smaller particle sizes that make it easier to be extracted compare to the slurry samples. Thus, the detection of elements concentration is higher compared to the slurry. Inertization of elements is associated with the ionization because the metastable atoms collide with elements, resulting in the inertization of elements with the e– reactions (Mattox, 2010; Sanito et al., 2020). Inertization of harmful elements in plasma technology is related to the bond breakage and the sputtering atoms from the penning ionization. It occurs because of the interaction of reactive species (Bonizzoni and Vassalo, 2002; Heberlein and Murphy, 2008; Benedikt, 2010; Sanito et al., 2020; Sanito et al., 2022a; Sanito et al., 2022b). Thus, inertization occurs and the concentration of elements in the sample decreases.

3.4. Comparison on the inertization of elements with different carrier gases

Figure 11 shows information associated with the inertization of elements compared to nitrogen and argon as the carrier gases. From this study, the using of argon as the carrier gas showed greater results compared to nitrogen. Removal efficiencies of As, Cd, Cr, Cu, Pb, Ni, Zn can be obtained by 82.85%, 93.25%, 86.66%, 88.03%, 85.85%, 80.27%, and 74.92%, respectively, with the using argon as the carrier gas in the treatment of water hyacinth. Conversely, with nitrogen gas, removal efficiencies of As, Cd, Cr, Cu, Pb, Ni, Zn can be obtained by 68.06%, 95.40%, 28.30%, 89.33%, 84.59%, 77.22%, and 68.14%, respectively. The highest removal was Cd (95.40%), which is 2.2% higher than argon as the carrier gas. Interestingly, inertization of other elements, such as As, Cr, Pb, Ni, and Zn, exhibited better removal efficiencies, ranging from 74.92% to 88.03%. Thus, for degrading elements from water hyacinth, argon indicates better results. All elements of water lettuce, namely, As, Cd, Cr, Cu, Pb, Ni, and Zn can be degraded by 95.01%, 95.04%, 28.30%, 89.33%, 89.33%, 84.59%, 77.22% and 68.14%, respectively, comparing with the use of nitrogen. All the inertizations were higher than the using of nitrogen gas. Argon gas indicates the high inertization, which is probably it is associated with the ionization from the plasma jet. Electrons absorb energy from an electric field and transfer it to plasma components, resulting in ionization, which is important for degrading contaminants (Benedikt, 2010; Sanito et al., 2020, 2022a). High energy electrons can be generated from the metastable argon’s atoms (Tian et al., 2019). The kinetic energy of free electrons causes the high free
electrons that creates excite argon (Zhang et al., 2020). Thereby, the argon indicates better results in degrading elements.

3.5. Hydrogen and methane production from plasma pyrolysis

Percentages and concentrations of H₂, CH₄, CO, and CO₂ from water hyacinth and water lettuce are outlined in Tables S8 and S9, respectively, and Figure 12a and 12b show the production rate of gases, namely H₂, CH₄, CO, CO₂, and VOCs from water lettuce and water hyacinth, respectively. Furthermore, Figure S10 gives the illustration of calibration curves of H₂, CH₄, CO and CO₂, respectively. Nitrogen was used as the carrier gas with the setup at 9 SLM, considering the fast reaction and cost-less issue (Gomez et al., 2009; Sanito et al., 2020). Meanwhile, microwave power was controlled at 1000 W (Li et al., 2014a). In first 2 min, H₂ concentrations from water hyacinth and water lettuce were 9500 ppm and 12,812 ppm, respectively. In addition, the concentration of CH₄ from water hyacinth and water lettuce were 459 ppm and 457 ppm, respectively. High temperature from an atmospheric-pressure microwave plasma reactor play a major role in the pyrolysis cellulose (Yang et al., 2007) where the temperature of plasma almost reached 1000 °C (Sanito et al., 2020). Thereby, the total gas concentrations of water hyacinth and water lettuce were 16,848 ppm and 22,565 ppm, respectively. The accumulation of gas concentration in water lettuce was higher 5717 ppm, compared to the water hyacinth. In 4 min, the concentrations of H₂, CO, CO₂, CH₄ from water lettuce were 9433 ppm, 5151 ppm, 1339 ppm, and 319 ppm, respectively. In addition, H₂, CO, CO₂ concentrations of water hyacinth were 8542 ppm, 4082 ppm, and 1680 ppm, respectively, except CH₄, was 744 ppm (4.89%), which was 425 ppm (0.72%) higher than water lettuce. Despite the concentration of gases decreased in 4 min, the percentage of the H₂ had the highest percentage ranging from 56.17% to 58.10% in water hyacinth and water lettuce compared to others. Ramzan et al. (2022) stated that the higher temperature in pyrolysis plays a major role to increase H₂ yield. VOCs compositions from water lettuce and water hyacinth were 11.279 ppm–20.051 ppm (0.049%–0.1%) and 6.484 ppm–157 ppm (0.038%–1.032%), respectively. Thereby, it can be concluded that the biomass from water lettuce and water hyacinth thus can be recommended for producing the gases.

From the plasma pyrolysis, the highest concentration of gas from water lettuce and water hyacinth is H₂. The composition of H₂ from water hyacinth and water lettuce were 56.28% (18,042 ppm) and 57.30% (22,255 ppm), respectively. However, CH₄ compositions of water hyacinth and water lettuce were 3.75% (1203 ppm) and 2% (776 ppm), respectively, which are, respectively, 52.53% (16,839 ppm) and 55.3% (21,479 ppm) less than results of H₂. Thus, CH₄ productions from water hyacinth and water lettuce are lower compared to the H₂. Interestingly,
Pyrolysis also generated the other gases, such as CO₂ and CO. Concentrations of carbon dioxide from water hyacinth and water lettuce were 3833 ppm (8.72%) and 3612 ppm (11.29%), respectively. CO concentrations of water hyacinth and water lettuce were 9025 ppm (28.15%) and 12,392 ppm (31.90%), respectively. Lin et al. (2014b) confirmed that the H₂ production can obtained by 67.33% with the setup 1000 W from banyan leaves. The compositions of H₂ from water lettuce and water hyacinth were 57.35% and 56.58%, respectively, which are, respectively, 9.98% and 10.75% less than banyan leaves. However, total H₂ concentrations of water lettuce and water hyacinth were 22,255 ppm and 18,042 ppm, respectively. Conversely, the total concentration in ppm of banyan leaves from the study of Lin et al. (2014b) was not mentioned.

Figure 9. Removal efficiency of elements from water lettuce (a). Dry water lettuce with flow rate 6 SLM (b). Dry water lettuce with flow rate 9 SLM, (c). Dry water lettuce with flow rate 12 SLM, (d). Fresh water lettuce with flow rate 6 SLM, (e). Fresh water lettuce with flow rate 9 SLM, (f). Fresh water lettuce with flow rate 12 SLM.
The percentages of H₂ from water hyacinth and water lettuce were higher than wheat straw, waste wood, rice straw, corn stalk bale with value of 43.70%, 44.40%, 50.67%, 23.70%, respectively (Huang et al., 2010; Zhao et al., 2010; Efiika et al., 2012; Zhang et al., 2020). Thus, it can be recommended that water hyacinth and water lettuce are useful for H₂ production. All the comparison studies of pyrolysis from biomass are shown in Table 2.

Water hyacinth and water lettuce pyrolysis also generated 3620 ppm (11.35%) of CO₂ and 9025 ppm (28.30%) of CO, and 12,392 ppm (31.93%) of CO and 3383 ppm (28.30%) of CO₂, respectively. Huang et al. (2010) stated that pyrolysis of rice straw generated CH₄ with fraction at 7.42%. Zhao et al. (2010) confirmed that 6.7% of CH₄ can be produced from microwave pyrolysis. The mechanism of cellulose conversion for generating H₂ is associated with the reaction of lignocellulose. During the reactions, oxidation and conversion occur (Lemmens and Elslander, 2007). Thus, transformation of material occurs, causing the production of gas.

Figure 10. Removal efficiency of elements from water hyacinth (a). Dry water hyacinth with flow rate 6 SLM (b). Dry water hyacinth with flow rate 9 SLM, (c). Dry water hyacinth with flow rate 12 SLM, (d). Fresh water hyacinth with flow rate 6 SLM, (e). Fresh water hyacinth with flow rate 9 SLM, (f). Fresh water hyacinth with flow rate 12 SLM.
According to Lin et al. (2014a), H₂ gas was generated from the secondary gas-phase reaction via the hydrogen atoms. As a result, the H₂ gas can be produced from the reactions. Cellulose and hemicellulose are the materials that provide the most H₂ composition in biomass pyrolysis, especially crops (Wu et al., 2013; Huang et al., 2016). In addition, the lower structures of lignin in plants give a high result as a bioenergy source (Rezania et al., 2015a, 2015b). However, due to the amorphous structure and little inherent physical strength, CO and CO₂ can be generated from the pyrolysis of biomass (Yang et al., 2007; Li et al., 2013). Further studies for reducing CO₂ and CO concentrations are suggested with the addition of catalyst during plasma pyrolysis of biomass (Efiaka et al., 2012; Wu et al., 2013). Reactions of conversion pathway from the biomass, for example cellulose, is described as follows (Lemmens and Elslander, 2007; Gorling et al., 2013; Huang et al., 2016).

Active cellulose is recognized as an intermediate in pyrolysis. An oxygenated hydrocarbon is generated from active cellulose. Then, active cellulose pathway reactions produce levoglucosan, hydroxymethylfurural and char. Secondary char is formed from the repolymerization of anhydrosugars (levoglucosan) (Shen et al., 2013; Lin et al., 2013). Each reaction generates H₂ and CO. The higher temperature of the carbon gasification is responsible for changing the reaction pathways (Li et al., 2014a). The presence of high CO₂ and CO fractions in products because of the carbon gasification is not reacted properly in the pyrolysis. According to Dominguez et al. (2007), the reaction temperature affects the decrease of CO₂ formation. Further investigation should be performed with the setup of plasma parameters for tackling CO₂ and CO issues from plasma pyrolysis because of the presence of high temperature reactions. An illustration of the reactions pathway of water hyacinth and water lettuce is described in Figure 13.

Huang et al. (2013) stated that the gas product should be quenched for generating the high reaction temperatures in the plasma reactors. Moreover, radicals combine with the functional groups in the breaking process. Also, high temperatures from the plasma are responsible for degrading cellulose because during the pyrolysis, it requires high energy. In this study, the radicals from the reactive species in plasma reacts with C-H, C=C, and C=O for breaking processes, respectively, which are respectively, produce H₂, CO, and CO₂ gases.

### 3.6. Volatile organic compounds (VOCs) analysis

Table 3 gives the information on volatile organic compounds (VOCs) from the pyrolysis of the water hyacinth and water lettuce. In this study, benzyl chloride, ethylbenzene, m,p-xylene, and others were not detected from the pyrolysis. However, some contaminants, such as propylene, chloromethane, butanone, benzene, toluene, ethylbenzene, were detected from the plasma pyrolysis. The highest concentration of VOCs from water hyacinth was 119.20 ppm of propylene, which was lower 95.71 ppm of water lettuce. The concentration of propylene from water hyacinth can be obtained at 23.49 ppm. Unfortunately, pollutants, such as benzene and toluene can be detected at nearly 1.21 ppm and 5.2 ppm. Kim and collaborators (2016) stated that decomposition of lignin produces aromatic compounds, such as benzene, toluene and naphthalene. Bashir...
et al. (2018) confirmed that the presence of aromatic compounds is due to the high temperature pyrolysis of biomass and cellulose materials. In this study, benzene, toluene, ethylbenzene, and m.p-xylene concentrations from water hyacinth were 1.21 ppm, 5.82 ppm, 1.23 ppm, and 0.31 ppm, respectively. From water lettuce, the concentrations of benzene, toluene, ethylbenzene, and m.p-xylene from water hyacinth were 1.28 ppm, 0.45 ppm, 0.066 ppm, and 0.019 ppm, respectively. Sanito et al. (2020) stated that the compositions of lignin from water hyacinth and water lettuce were 7.03% and 3.47%, respectively, which were the lowest among cellulose and hemicellulose of whole plants. Thereby, it can be understood that the pyrolysis of water hyacinth and water lettuce via an atmospheric-pressure microwave plasma reactor generates lower concentrations of benzene, toluene, ethylbenzene, and m.p-xylene.

Figure 12. Gas production of H₂, CO, CH₄, and CO₂ from water lettuce and water hyacinth, respectively. Total VOCs concentration also presented in this figure (a). Gas production rate of water lettuce, (b). Gas production rate of water hyacinth.
respectively. Thus, carbon from biomass with the lower composition of lignin can be used as recycling materials via an atmospheric-pressure microwave plasma reactor.

Despite H₂ and CH₄ are generated from plasma pyrolysis, VOCs from the pyrolysis should be treated carefully from the installation system for trapping the pollutants. Huang et al. (2011) found that approximately 3.5% of total VOCs can be detected from the burning of biomass. According to Li et al. (2014), biomass burning, such as crop and wood residues, contributes significantly to VOC emissions. Tomul et al. (2020) confirmed that bio-sorbent derived from biomass contains of volatile compounds with value of 77.9%. Wang et al. (2014) stated that the VOCs emission factors of straw burning can be obtained by approximately 6.99 g/kg. Compared to straw burning, VOCs from wood-burning have a lower concentration at 0.98 g/kg. In this aspect, the installation of a scrubber is suggested for reducing contaminants (Cubas et al., 2014), especially from biomass. Thereby, the elimination of VOCs during the

Table 2. Comparison of pyrolysis for generating hydrogen (H₂) and methane (CH₄).

| Systems                          | Biomass                  | Microwave power | Flow rate | Carrier gas | Compositions | References         |
|---------------------------------|--------------------------|-----------------|-----------|-------------|--------------|--------------------|
| Atmospheric-pressure microwave plasma reactor | Banyan (Ficus sp.) leaves | 800 W           | 12 SLM    | N₂          | H₂ 35.05%    | Not analyzed       |
|                                 |                          |                 |           |             | CH₄ 35.05%   | Lin et al., (2014a) |
| Atmospheric-pressure microwave plasma reactor | Banyan (Ficus sp) leaves | 900 W           | 12 SLM    | N₂          | H₂ 51.85%    | Not analyzed       |
|                                 |                          |                 |           |             | CH₄ 51.85%   | Lin et al., (2014a) |
| Atmospheric-pressure microwave plasma reactor | Banyan (Ficus sp.) leaves | 1000 W          | 12 SLM    | N₂          | H₂ 67.33%    | Not analyzed       |
|                                 |                          |                 |           |             | CH₄ 67.33%   | Lin et al., (2014a) |
| Atmospheric-pressure microwave plasma reactor | Spirulina algae         | 800 W           | 12 SLM    | N₂          | H₂ 90.26%    | Not analyzed       |
|                                 |                          |                 |           |             | CH₄ 90.26%   | Lin et al. (2014b)  |
| Atmospheric-pressure microwave plasma reactor | Spirulina algae         | 900 W           | 12 SLM    | N₂          | H₂ 89.57%    | Not analyzed       |
|                                 |                          |                 |           |             | CH₄ 89.57%   | Lin et al. (2014b)  |
| Atmospheric-pressure microwave plasma reactor | Spirulina algae         | 1000 W          | 12 SLM    | N₂          | H₂ 88.97%    | Not analyzed       |
|                                 |                          |                 |           |             | CH₄ 88.97%   | Lin et al. (2014b)  |
| Microwave reactor               | Wheat straw              | Not Mentioned   | 3 L/min – 1 | N₂          | H₂ 43.70%    | Not analyzed       |
|                                 |                          |                 |           |             | CH₄ 7.90%    | Zhao et al. (2010)  |
| Screw Kiln Reactor              | Waste wood               | Not Used        | Not mentioned |           | H₂ 44.40%    | Not analyzed       |
|                                 |                          |                 |           |             | CH₄ 44.40%   | Efika et al. (2012) |
| Microwave induced pyrolysis     | Rice straw               | Not Used        | Not Used  | Not Used    | H₂ 50.67%    | Not analyzed       |
|                                 |                          |                 |           |             | CH₄ 7.42%    | Huang et al. (2010) |
| Conventional and Microwave      | Coffee hulls             | Not Used        | Not Used  | Not Used    | H₂ 9.28%     | Not analyzed       |
|                                 |                          |                 |           |             | CH₄ 9.28%    | Dominguez et al. (2007) |
| Microwave pyrolysis             | Corn stalk bale          | 0.668 kW/1 kg H² 6/kW kg⁻¹ | 5 L min⁻¹ | N₂          | H₂ 23.7%     | Not analyzed       |
|                                 |                          |                 |           |             | CH₄ 6.7%     | Zhao et al. (2010)  |
| Atmospheric-pressure microwave plasma reactor | Water hyacinth (Eichhornia crusgatesa) | 1000 W | 9 SLM | N₂ | H₂ 56.28% | 3.75% | This study |
| Atmospheric-pressure microwave plasma reactor | Water lettuce (Pistia stratiotes) | 1000 W | 9 SLM | N₂ | H₂ 57.30% | 2% | This study |

Figure 13. Reaction pathway of cellulose transformation from water hyacinth and water lettuce.
production of H₂ and CH₄ should be considered in further studies of plasma pyrolysis.

4. Conclusion

This research demonstrated that inertization of elements can be obtained via plasma pyrolysis as well as H₂ and CH₄ from water hyacinth and water lettuce. Also, Total concentration of VOCs was assessed from gas exhaust. Elements (metals and metalloids) in water hyacinth and water lettuce can be eliminated via plasma pyrolysis. Pyrolysis generates H₂ and CH₄ with percentages of more than 56% and 1.99%, respectively. Also, the total VOCs concentration of water hyacinth and water lettuce was less than 100 ppm, indicating the less polluted gas emission from the plasma pyrolysis of water hyacinth and water lettuce. Treatment of phytoremediation plants via plasma technology can be suggested to fulfill the concept of circular economy due to its potential not only for inertization of elements but also produce the H₂ and CH₄ with the lower concentration of VOCs. Future studies should be conducted on reducing the VOCs, improving CH₄ fractions, and reducing CO₂ and CO fractions with the addition of the catalysts.

Declarations

Author contribution statement

Raynard Christianson Sanito: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.
Cindy Lidwina: Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.
Hsi-Hsien Yang: Conceived and designed the experiments; Contributed reagents, materials, analysis tools or data.
Ya-Fen Wang: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

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Data availability statement

The authors are unable or have chosen not to specify which data has been used.

Declaration of interest’s statement

The authors declare no conflict of interest.
Additional information

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