Research article

Bio-oil and char obtained from cassava rhizomes with soil conditioners by fast pyrolysis

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ABSTRACT

We studied the properties of the bio-oil and char from fast pyrolysis of cassava rhizomes in a free-fall reactor, catalyzed by adding various soil conditioners (or improvers) as catalysts to the reactor at 500 °C and 200 g/h feed rate. Four conditioners were evaluated - granular TPI pH11 soil conditioner, volcanic rock, TPI pelletized organic fertilizer and super dolomite - and added at 50% (w/w) of the rhizomes. Bio-oil and gas yields decreased, whereas char yields increased, yielding bio-oil 57–59%, gas 14–20% and char 23–28%. With the catalysts, the bio-oil higher heating value increased, while that of char decreased: for cassava rhizomes with volcanic rock the bio-oil high heating value increased from 19.4 to 23.6 MJ/kg, whereas rhizomes with dolomite led to an increased viscosity from 27 to 34 × 10⁻³ mm²/s.

1. Introduction

Today, the world is experiencing a crisis with high oil prices and diminishing oil reserves. Many countries have focused on renewable energy and invented new alternative fuel sources with potential and adequate quantity [1]. Synthetic oil from biomass is currently generating strong interest, because it provides energy and can be used in vehicles. The raw biomass also comes from many agricultural residues, therefore it is plentiful and using it reduces waste. In addition, it is environmentally friendly, because it has the potential to reduce pollutants, e.g. SO₂, nitrogen oxides (NOₓ) and others [2].

Fast pyrolysis decomposes particles, calcined in the range of 400–600 °C [3]. The main products are bio-oil, char and non-condensing gases [1, 4]. The characteristics of bio-oil depend on many factors, including the type of biomass, particle size, reactor type and pyrolysis temperature [5]. Generally the bio-oil will be dark brown, with high viscosity and similar properties to fossil fuels [6], but to use it in an engine fuel, it is necessary to improve its properties. One improvement emulsifies it with diesel and biodiesel at 20–50% by volume, decreasing the viscosity and improving stability [7, 8]. Catalysts also improve the properties: ZSM-5 [9], dolomite [10], natural zeolite [11], zinc oxide [12], kaolin [13] and potassium carbonate [14] have been reviewed by Jahirul et al. [15], Pattiya [16] and Ochoa et al. [17]. One function of the catalysts is assisting heat transfer to biomass, thus catalysts providing high surface areas are effective. Catalysts also crack primary organic vapors to form secondary products and remove oxygen content and acids, aldehydes and ketones.

In addition, the char can also be used as fuel or for other applications, for example as a metal reductant soil amender [18], as activated carbon for removal of toxins from water [19] and biocarbon electrodes [20]. Char can take many forms, depending on the feed biomass, catalysts used and conditions: it has been reviewed extensively by Ocha et al. [21].

The pyrolysis gas mainly consisted of H₂ and CO [15]. It also contains small fractions of CO₂, N₂, H₂O, alkanes, alkenes and alkynes, for example CH₄, C₂H₄, and C₃H₆, depending on the biomass and pyrolysis conditions [22, 23].

In fast pyrolysis, typically 20–30% by weight of the product is char. A typical process flow is shown in Figure 1. Generally, biomass is held in a storage tank and fed to the pyrolysis chamber or reactor. Some shards (biomass or catalyst) leaving the reactor are filtered by a cyclone that removes large particles. Finer pyrolysis products can be filtered by glass wool in a hot vapor filter set. However, glass wool filters cannot filter super fine char, which will remain in the bio-oil, reducing its stability. The char, essentially carbon, obtained can be processed into household fuel rods and used as a soil conditioner. Pattiya [24] found that the char...

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from fast pyrolysis of cassava rhizomes and stems was 55–65% C, ~3% H, ~1% N and 6–10% O, by weight. Kim et al. [25] found that the char also contained Zn, Na, Mg, Al, Si, Cl and K - beneficial to plant growth. When applied, it changed the soil physical structure, allowing it to drain water and air well. Further acidity was reduced and the char became a place to store microorganisms and humus.

Here, we focused on improving and upgrading the products from fast pyrolysis with several soil conditioners, i.e. granular pH11 plus soil conditioner (L11), volcanic rock (VR), TPI pelletized organic fertilizer (OF) and super dolomite (SD). We envisaged that our work would help farmers to construct small scale pyrolysis plants, that converted waste to energy and useful bioproducts, i.e. the char, which has been demonstrated to be useful as an additional soil conditioner, thus we chose a set of conditioners, that have already been shown to be effective catalysts. Since they were inexpensive and readily available - near the source of the agricultural waste - they did not introduce any exotic new materials. Further, since our system should be as simple as possible, any additional steps to recover or reactivate the catalysts would defeat one of our aims, so the catalysts should be able to be collected in the char and used without further processing. Therefore existing solid conditioners would meet this requirement.

2. Materials and methods

2.1. Biomass feedstock

The cassava rhizomes used were derived from Kasetsart-50 varieties from Non Daeng Baan, Bua Ban, Yang Talat, Kalasin, Thailand (16.49301’N, 103.42594’E). It was ground until particles passed a 0.5 mm diameter sieve. It was dried to reduce moisture (105 ± 5 °C, 3 h). The input was approximately 50% by weight of cassava rhizomes and soil improvement. Characteristics of cassava rhizomes were measured - moisture content following ASTM E1756-01, fraction of volatile matter (ASTM E872-82), ash content (ASTM E1755-01), the fraction of fixed carbon was calculated from the differences as.

Fixed carbon (dry basis) = 100 – Volatile – Ash.

Elemental analysis, for C, H, N and O, followed EN45014. The higher heating value was measured by a bomb calorimeter, ESSOM, Art 2060/2070, following DIN 51900. Table 1 shows the properties of cassava rhizomes used here, compared to previous research, which shows the typical variations, expected for a natural product.

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| Analysis | This study | Rueangsan et al. [28] | Boonsombut et al. [29] |
|----------|------------|----------------------|----------------------|
| Proximate analysis | | | |
| Moisture content, wt % | 8.3 | 6.1 | 6.6 |
| Volatile, wt % | 77.7 | 80.5 | 76.1 |
| Ash, wt % | 4.1 | 3.0 | 3.8 |
| Fixed carbon a, wt % | 18.2 | 10.4 | 13.5 |
| Ultimate analysis | | | |
| Carbon (C) | 51.6 | 53.6 | 43.2 |
| Hydrogen (H) | 7.6 | 7.7 | 6.0 |
| Nitrogen (N) | 1.3 | 1.3 | 1.2 |
| Oxygen (O b) | 39.5 | 37.4 | 45.5 |
| Higher heating value, MJ/kg | 23.7 | 15.4 | 15.5 |

a Fixed carbon (dry basis) = 100 – Volatile – Ash.

b Oxygen = 100 – Carbon–Hydrogen–Nitrogen.

Table 2 lists specifications of the soil conditioners - granular pH11 plus conditioner (L11), volcanic rock (VR), TPI pelletized organic fertilizer (OF) and super dolomite (SD). The soil improvements were purchased from TPI Polene Bio-organics Co. Ltd. Bangkok, Thailand. The L11 conditioner is, as its name suggests, a high pH (dissolved in an equal mass of water, it will record a pH ≥ 11) additive, mostly CaO, with a small

| Analysis | L11 | VR | OF | SD |
|----------|-----|----|----|----|
| Calcium Oxide (%) | 45.54 | 44.11 | 53.84 |
| Magnesium Oxide (%) | 2.47 | 21.99 |
| Moisture (%) | 2.54 | 0.42 | 15.72 |
| pH | 11.67 | 11.73 | 8.80 |
| Loss on Ignition (%) | 37.75 | 33.67 |
| Calcium Carbonate Equivalent | 89.69 | 88.09 |
| Organic Matter (%) | 29.10 |
| Total Nitrogen (%) | 1.71 |
| Total P2O5 (%) | 1.29 |
| Total K2O (%) | 1.16 |
amount of MgO, designed to increase the pH of acid soils. Dolomite (SD), mostly CaMg(CO3)2, has a similarly high pH ~12, and a similar function: there are numerous reports [10, 15, 26, 27] showing that it can increase pyrolysis yields. Volcanic rock (VR) has a highly porous structure presenting a large surface – a characteristic of many effective catalysts: it also contains quantities of zeolites and another member of the same family, ZSM-5, has been extensively used as a catalyst [16]. The organic fertilizer (OF) was mostly organic material, with guaranteed, but small (>1%), N, P and K contents, thus potentially beneficial if used as an enhanced soil conditioner.

2.2. Fast pyrolysis experiments

The flow in our free-fall reactor is illustrated in Figure 1: it was used in our previous work and images are found there [28]. The components of the fast pyrolysis system are shown in Figure 2. Biomass was kept in a hopper and fed by an N2 gas flow and a screw feeder, into the reactor. The 7.7 mm diameter, 1.2 m long, 304 stainless steel reactor was wrapped in a 2 kW circular heating coil. Temperature through the length of the reactor was monitored with type-K thermocouples and set at 500 °C by a PID controller. A water cooling jacket was fitted at the top and a char pot, at the bottom, captured char, while vapor and entrapped particles flowed to the cyclone. Pyrolysis occurred within the heated reactor, without O2, pyrolysis products were delivered to cyclones 1 and 2, which blocked chars. A hot filter kept at 420 °C captured very small char particles that escaped the cyclones. Vapor transiting the filter was liquefied by a 30 °C water condenser. The liquid was retained in bio-oil pot 1. An 14kV electrostatic precipitator captured vapor that passed the condenser: bio-oil captured at this point was labelled ‘heavy bio-oil’. Any remaining vapor was captured in three ice-buckets, kept at 10 °C and labelled ‘light bio-oil’. A cotton wool filter captured any vapor remnants. Three replicates were measured for each condition: means and standard deviations are reported.

2.3. Mass balance

Fast pyrolysis produces bio-oil, char and gas. Chang et al. [30] derived the yield of bio-oil from the condenser and the used biomass, whereas Pattiya et al. [31] calculated yields from water condenser, dry-ice and acetone condenser and cotton wool filter versus the input biomass and subtracted moisture in the input and solids in the bio-oil. We calculated the pre- and post-experimental mass difference of bio-oil pot 1 and 2 and char pot 1 and 2 versus mass of biomass consumed. Gas yield was calculated from the difference as shown in Equations (1) – (3).

\[
Y_{\text{bio-oil}} = \frac{\sum W_{\text{bio-oil}}}{W_{\text{biomass}}} \times 100\% (1)
\]

\[
Y_{\text{char}} = \frac{\sum W_{\text{char}}}{W_{\text{biomass}}} \times 100\% (2)
\]

\[
Y_{\text{gas}} = 100 - Y_{\text{bio-oil}} - Y_{\text{char}} (3)
\]

where

\[
Y_{\text{bio-oil}} = \text{Bio-oil yield (wt %)}
\]

\[
Y_{\text{char}} = \text{Char yield (wt %)}
\]

\[
Y_{\text{gas}} = \text{Yield of non-condensable gases (wt %)}
\]

\[
W_{\text{bio-oil}} = \text{Total bio-oil mass from water condensing unit and electrostatic precipitator (g)}
\]

\[
W_{\text{char}} = \text{Total mass of char from char pot and cyclone (g)}
\]

\[
W_{\text{biomass}} = \text{Mass of biomass consumed (g)}
\]

Figure 2. Annotated image of the fast pyrolysis unit.
2.4. Bio-oil analysis

The heavy bio-oil from the bio-oil pot 1 was analysed for water, solids and ash content, pH, heating value, density and viscosity. Three replicates were run [32].

pH value: pH was measured with a Denver UB-10 pH meter. Before every measurement, it was checked against standard buffers at pH 4, 7 and 10.

Density: A known volume of bio-oil was weighed at 15 °C, following ASTM D4052. The beaker used had a volume of 10 ml. 5 ml of bio-oil was poured into the beaker, the weight recorded and compared with the beaker volume to determine the bio-oil density.

Viscosity: Following ASTM D445 and ASTM D446, kinematic viscosity, in centistokes (mm²/s or cSt) was measured with a Cannon-Fenske Opaque viscometer, at 40 °C. We poured 12 ml bio-oil into the tube and measured the time for the oil to flow through the glass tube immersed in water at 40 °C, then multiplied the time by the tube calibration factor, determined by calibrating with a liquid of known viscosity.

Ash content: Following ASTM D482, we placed 3 g bio-oil in a ceramic cup, weighed before and after heating at 775 ± 25 °C for 24 h. The difference was the ash content.

Solids content: We used vacuum filtration, following ASTM D7579. We mixed 50 ml of methanol and dichloromethane at a ratio of 1:1 [33], ~3 g bio-oil added to the solvent and shaken well. Then, Whatman No.3 filter paper, capable of capturing solid particles down to 6 μm, was baked at 105 °C for 15 min and placed on a vacuum filter unit. The dissolved bio-oil was pulled through the filter paper by vacuum. After that, the filter paper was baked at 105 °C for 30 min and compared with the initial weight to determine the solid content.

Higher Heating Value (HHV): About 1 g sample was weighed, placed in a combustion cup and placed in the bomb filled with O₂. The cup was attached to the calorimeter bomb and the sample ignited with an electric wire. During combustion, heat generated inside the bomb cup and was transferred to the water in which the cup was immersed. The measured rise in water temperature was used to determine the heat of combustion. The bomb was calibrated from the temperature rise from combustion of a known mass of pure dry benzene.

Morphology of char samples was observed using Scanning Electron Microscopy, SEM, Leo 1450 VP, Zeiss, Germany. Atomic force microscopy was used to characterize deposited film surface topographies - semi-contact mode with the scanning area and 0.4Hz scan frequency.

| Product       | CR | CR + L11 | CR + VR | CR + OF | CR + SD |
|---------------|----|----------|---------|---------|---------|
| Bio-oil       | 60.6 ± 0.4 | 57.1 ± 0.3 | 59.7 ± 0.8 | 59.6 ± 0.4 | 57.6 ± 1.0 |
| Char          | 18.0 ± 0.6 | 28.1 ± 2.0 | 25.9 ± 2.9 | 20.0 ± 5.4 | 22.9 ± 1.0 |
| Non-condensible gas | 21.4 ± 0.6 | 14.8 ± 1.6 | 14.6 ± 2.5 | 20.4 ± 0.4 | 19.5 ± 0.7 |

3. Results and discussion

The yields and analyses of bio-oil, char and gas from cassava rhizomes, pyrolyzed with several readily available and inexpensive soil improvers.

3.1. Product yield

Generally, using the soil improvers led to lower yields of bio-oil and non-condensable gas, but the char yield increased. Yields for the oil, gas and char for the L11, VR, OF and SD soil conditioners are shown in Table 3. Oil yields compare favourably with those from other agricultural waste, e.g. Biswas et al. [34] – 28–47% by weight from corn cobs, wheat straw, rice straws and rice husks; Pidtasang et al. [32] – 50–60% by weight from eucalyptus bark catalyzed with alcohols; Boer et al. [35] – 55% by weight from sugarcane bagasse.

3.2. Bio-oil properties

Table 4 shows that bio-oil from rhizomes with the soil conditioners had higher HHVs, which is consistent with several other studies, e.g. fast pyrolysis of hardwoods using ZSM-5 and Kaolin clay [36]. In particular, the volcanic rock contained significant amounts of zeolites or related compounds, which have been shown to effectively ‘crack’ long chain polymers to smaller molecules [16] and led to a significant increase in HHV. For all four soil conditioners, the thermal properties of bio-oil fuels were improved.

3.3. Char properties

Char obtained from the fast pyrolysis of the rhizomes had a dark black color: char from the L11, VR and SD catalysts had a slightly different color and structure characteristics. Measured properties of the char are listed in Table 5.

3.4. Char morphology

Images of the char obtained from the cassava rhizomes, is shown in Figure 3 (CR). The morphology changed significantly when the catalysts were introduced, as seen in images marked L11, VR, OF and SD. Generally the char appears to have formed about particles of the catalysts, but examples of several types of char structures are evident. Catalyst surface coverage by the char is generally considered a disadvantage and many techniques to avoid catalysts deactivation have been described [16, 17, 37]. However, we note that since we considered the char might be a useful soil conditioner, so that the catalysts we used – all chosen as economic and readily available to farmers – were, in themselves, useful conditioners, and ‘consuming’ them in the char was not a disadvantage: the low cost catalyst would be replaced in the biomass feed.

Table 3. Yields of bio-oil, char and non-condensable gas.

| Product       | CR | CR + L11 | CR + VR | CR + OF | CR + SD |
|---------------|----|----------|---------|---------|---------|
| Bio-oil       | 60.6 ± 0.4 | 57.1 ± 0.3 | 59.7 ± 0.8 | 59.6 ± 0.4 | 57.6 ± 1.0 |
| Char          | 18.0 ± 0.6 | 28.1 ± 2.0 | 25.9 ± 2.9 | 20.0 ± 5.4 | 22.9 ± 1.0 |
| Non-condensible gas | 21.4 ± 0.6 | 14.8 ± 1.6 | 14.6 ± 2.5 | 20.4 ± 0.4 | 19.5 ± 0.7 |

Table 5. Char properties.

| Property       | CR | CR + L11 | CR + VR | CR + OF | CR + SD |
|----------------|----|----------|---------|---------|---------|
| Density (kg/m³) | 600 ± 100 | 800 ± 100 | 700 ± 100 | 800 ± 100 | 900 ± 100 |
| Volatile matter (wt %) | 36.5 ± 0.5 | 30.7 ± 0.6 | 33.8 ± 0.5 | 34.4 ± 0.6 | 34.4 ± 0.6 |
| Ash content (wt %) | 19.0 ± 0.6 | 28.5 ± 0.5 | 26.6 ± 0.6 | 24.6 ± 0.5 | 30.4 ± 0.5 |
| Fixed carbon (wt %) | 43.2 ± 0.9 | 38.3 ± 0.3 | 37.5 ± 0.7 | 38.8 ± 0.7 | 33 ± 0.8 |
| HHV (MJ/kg) | 24.6 ± 0.3 | 20.6 ± 0.4 | 21.6 ± 0.3 | 23.2 ± 0.2 | 20.2 ± 0.2 |
3.5. Char vs catalyst used

In addition, the catalysts increased the amount of ash, which was the highest for dolomite at 30.4% by weight. Consequently the added catalysts decreased the amount of volatile matter.

3.5.1. Density

The measured char densities are also consistent with deposition of the catalyst into it: from rhizomes alone, we observed the lowest density, 600 kg/m³, rising to 700 kg/m³ with volcanic rock (VR), which has densities ranging from <1000 kg/m³ to 3300 kg/m³ – depending on which components are measured: for the L11 catalyst, char density was 800 kg/m³, consistent with a density for CaCO₃ of 2700–2950 kg/m³, similarly dolomite (SD) char was slightly denser at 900 kg/m³, but dolomite has a similar density range, 2600–2900 kg/m³. The organic matter (29% w/w) in the OF catalyst led to the fibrous nature of the char (see Table 3, OF) and the slightly lower char density, 800 kg/m³. However, it contains a large amount of moisture (16% w/w based on the manufacturer’s assay, but specified as <30%). Thus it is likely to vary considerably from sample to sample, as most of the moisture will be driven out at the 500 °C pyrolysis temperature – as seen in the higher variation in char yield for OF shown in Table 3.

3.5.2. Char HHV

Char HHV values also correlated with the organic matter in the feed: highest for rhizomes alone at 24.6 MJ/kg, but slightly lower for OF. However, with mostly inorganic catalysts (L11, VR and SD), large amounts of the catalyst were trapped in the char, leading to significantly lower HHVs (20.2–21.6 MJ/kg). This was consistent with the ash content of the char, which increased in the order: CR < OF < VR < L11 < SD, where the inorganic material of VR, L11 and SD appeared in the ash. Similarly, fixed carbon was significantly higher for the rhizomes alone (CR, 43% w/w) and lowest with dolomite (SD, 33% w/w), with the others lying in the 37–39% range.

4. Conclusion

Fast pyrolysis of cassava rhizomes with several soil conditioners as catalysts in a free-fall reactor at 500 °C overall showed that the catalysts decreased the yield of bio-oil and gas, but the char yield increased. However, the soil additives increased the bio-oil calorific value from 19.4 MJ/kg, without catalyst, to 21.3 MJ/kg with L11, 23.6 MJ/kg with VR, 21.2 MJ/kg with OF and 20.5 MJ/kg with SD. Whilst yield of bio-oil decreased slightly, from 60% to 57–60%, the char yield increased significantly from 18% to 23–28%, although the HHV values decreased, consistent with the reduction of fixed carbon and increase in ash content, caused by catalyst entrained in the collected char.

In future work, we expect that this work will develop into a simple practical system with dual aims: exploiting the large volumes of agricultural waste to both produce energy and benefit soils, using materials, the soil conditioners, that are already available and cheap and present no potential hazards.
Declarations

Author contribution statement

Koson Rueangsan, Pakkip Kraisoda, Adcha Heman, Homhuan Tasarod & Monchawan Wangklangkool: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Somsuk Trisupakitti & John Morris: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.

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Data availability statement

Data will be made available on request.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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