Cogent Engineering

ISSN: (Print) (Online) Journal homepage: https://www.tandfonline.com/loi/oaen20

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To cite this article: Koson Rueangsan, Adcha Heman, Pakkip Kraisoda, Homhuan Tasarod, Keyoon Duanguppama, Somsuk Trisupakitti & John Morris (2023) Bio-oil production via fast pyrolysis of cassava residues combined with ethanol and volcanic rock in a free-fall reactor, Cogent Engineering, 10:1, 2156054, DOI: 10.1080/23311916.2022.2156054

To link to this article: https://doi.org/10.1080/23311916.2022.2156054

Rueangsan et al., *Cogent Engineering* (2023), 10: 2156054
https://doi.org/10.1080/23311916.2022.2156054

MATERIALS ENGINEERING | RESEARCH ARTICLE

Bio-oil production via fast pyrolysis of cassava residues combined with ethanol and volcanic rock in a free-fall reactor

Koson Rueangsan[1], Adcha Heman[1], Pakkip Kraisoda[1], Homhuan Tasarod[2], Keyoon Duanguppama[2], Somsuk Trisupakitti[3]* and John Morris[4]

Received: 31 March 2022
Accepted: 29 November 2022

*Corresponding author: Somsuk Trisupakitti, Department of Chemistry, Rajabhat Maha Sarakham University, Thailand
E-mail: somsukrmu@gmail.com

Reviewing editor:
Ian Philip Jones, Metallurgy & Materials, The University of Birmingham, United Kingdom

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Abstract: Pyrolysis of waste biomass to produce usable energy has the potential to, in part, alleviate the consumption of limited fossil fuel resources. We describe bio-oil production via fast pyrolysis of cassava residues, a mostly wasted byproduct of cassava crops. The waste biomass was combined with two readily available additives—volcanic rock and ethanol—in a free fall reactor to generate bio-oil, char and syngas. Using cassava stems as the raw feed-stock we tested pyrolysis reaction temperatures in the range 450–500 °C in a free fall reactor using a N_2 flow. Analysis of the pyrolysis products should little variation in this range, so analyses for the effects of ethanol and volcanic rock as additives were tested at 500 °C. The bio-oil yield ranged between 58 and 60%, char represented 17–19% and gas 21–24%. With volcanic rock, the higher heating value was significantly higher at 23.6 MJ/kg compared to ~19 MJ/kg for cassava alone or added ethanol. Readily available, inexpensive, naturally occuring zeolites in the volcanic rock led to significant extra degradation of the biomass, under the same experimental conditions, leading to this improvement.

Subjects: Agriculture & Environmental Sciences; Biotechnology; Mechanical Engineering Design; Materials Science; Technology; Clean Tech

Keywords: cassava residues; bio-oil; free fall reactor; ethanol; volcanic rock

1. Introduction

The oil crisis has led to the realization that fossil fuel supplies will decline in the near future, which stimulated every nation to find new renewable energy sources. Biomass is one potential energy source, that can replace fossil fuel, especially in agricultural countries, where excessive amounts of biomass waste or residue are readily available. Pyrolysis can transform biomass into bio-oil and other valuable products: it involves the thermal decomposition of the input feed stock, at elevated temperatures, in an inert atmosphere, such as a vacuum gas (PAC, 2007); it changes the chemical composition and is irreversible. At high temperatures and anaerobic conditions, the (usually organic) feed stock is decomposed into three main components. It is one of the processes involved in charring wood, starting at 200–300 °C (Kramer et al., 2022). In general, pyrolysis of organic materials produces some volatile products, sometimes referred to as "syngas", and leaves a solid residue enriched in carbon, char. Extreme pyrolysis, which leaves mostly carbon as the residue, is called carbonization. Bio-oil is a major product—also known as pyrolysis oil, bio-oil, crude oil, wood oil, liquid from wood smoke or pyroligneous acid—containing many oxygenated compounds, formed by thermal decomposition and rapid degradation of polymer of cellulose, hemicellulose and lignin. Pyrolysis is used heavily in the chemical industry, for example, to produce ethylene, many forms of carbon, and other chemicals from petroleum, coal, and even wood; its potential has been studied for decades now, with aspirational applications which would convert biomass into syngas and biochar, waste plastics back into usable oil, or noxious waste into safely disposable substances discussed by Goodacre and Kell (1997). Recent reviews have extended these applications (Ochoa et al., 2020; Yogalakshmi et al., 2022), including, for example, sewage sludge (Seiple et al., 2017).

Igneous rocks in Thailand are mainly plutonic, volcanic and volcaniclastic rocks. Hypabyssal rocks are known in some places. Igneous rocks, which are widespread in Thailand, have been reviewed by Nutalaya (1973), Thanasuthipitak (1978) and Bunopas (). Among them, granites are the most common constituents, whereas intrusive rocks of intermediate, mafic, ultramafic composition and volcanic rocks are subordinate. Volcanic rocks are also found in most parts of Thailand and can be broadly grouped into the Permo-Triassic, Chiang Mai-Chiang Rai Volcanic Belt, the Permo-Triassic to Jurassic with some minor Late Tertiary Ko Chang-Tak-Chiang Khong Volcanic Belt and the Late Cenozoic Basalt (Department of Mineral Resources, Ministry of Natural Resources and Environment, 2016).

Ethanol, C_2H_5OH or ethyl alcohol, is a volatile, flammable, colorless liquid; it is naturally produced by the fermentation of sugars, from cassava, sugar cane and other plants, by yeasts or via petrochemical processes. It is widely used in foods, in medicine, as an antiseptic and disinfectant, and in science and industry, as a solvent. Ethanol is also used as a clean-burning fuel source (Goettemoeller & Goettemoeller, 2007).

Cassava is extensively farmed in Thailand and many other countries; its roots are a source of edible starch and can be converted, by fermentation, to ethanol for fuel and other uses. Thus there is an ample supply of little exploited residues after the starch-rich roots have been harvested.

1.1. Fast pyrolysis

Because of the potential to turn waste into useful energy, fast pyrolysis has been extensively studied. In a recent review, Yogalakshmi et al. (2022) reviewed lignocellulosic biomass pyrolysis focussing on the decomposition products, e.g., furfural and a variety of phenols, and briefly discussed reaction conditions (temperature, heating rate, residence time and particle size). Ochoa et al., 2020) discuss in detail the mechanisms for char formation. Optimum fast pyrolysis temperatures typically range from 450 °C to 550 °C (Shrivastava et al., 2021), with char predominating at lower temperatures (Mishra & Mohanty, 2020). Catalysts, commonly zeolites, have been also used extensively to optimize yields, see, Wu et al., 2022)'s review. Volcanic rocks are a significant source of a wide class of zeolites (Colella, 2005), so are a readily available and inexpensive source of catalysts.

The potential use otherwise troublesome waste, a wide variety of agricultural wastes have been studied—see reviews listed above. For cassava waste, Suttibak et al. (2012) reported fast pyrolysis of cassava rhizomes, in a fluidised-bed fast pyrolysis reactor unit, with a hot vapour filter: the optimum pyrolysis temperature was 472 °C, with a bio-oil yield of 63 wt% on dry biomass basis. The bio-oil components were water (18 wt%), solids (0.9 wt%) and ash <0.01 wt%: density was 1100 kg m^{-3}, pH 3.1 and the higher heating value (HHV) was 26.9 MJ/kg. Their values represent a reference for our work.

Focussing on readily available materials, cassava waste biomass, volcanic rock and ethanol, we measured bio-oil production from a thermal process, using nitrogen gas in a free fall reactor at 500 °C, and also measured the physical and chemical properties of the bio-oil.

To effectively convert biomass waste into bio-fuel energy, the fuel potentials of the biomass oils and their composition and properties as well as bio-oil conversion techniques are needed.

2. Material and methods

2.1. Biomass feedstock

The biomass used was residues from cassava farming in Kalasin, Thailand, dried, ground and sieved, to select particles between 0.2 and 0.5 mm diameter, and dried in an oven at 105 °C overnight (Rueangsan et al., 2018).

2.2. Fast pyrolysis experiments

Our fast pyrolysis machine was a free-fall reactor, described by Rueangsan et al. (2022) and shown in Figure 1. It had a biomass storage tank, from which biomass was fed by an auger, driven by a 12 V DC motor, at 200 g/h. The reactor was fabricated from a 304 stainless steel 16 mm diameter tube, 1.2 m long, heated at 10 °C/s. With an N_2 flow rate of 4 L/min, the residence time for particles in the reactor tube was ~3.6 s. Temperature was controlled between 450 and 550 °C. Initial char from decomposition was captured in char pot 1 and further trapped by a cyclone in char pot 2. Fine char particles are trapped with a hot steam fiber filter set. The cyclone set and hot steam filter were controlled at 420 °C. The water condensing unit was set to 30 °C. Pyrolysis vapor was then condensed as the "bio-oil" and stored in bio-oil pot 1, while excess pyrolysis vapor was further trapped by an electrostatic precipitator, set at 14 kV, from which bio-oil was collected in bio-oil pot 2. Product yields were determined gravimetrically and properties are analyzed.

Figure 1 shows the reaction chamber where the biomass is rapidly heated in the absence of air, producing gases, vapours and char. Typically, 60–75 wt.% of the feedstock was converted into oil. This research procedure refers to pyrolysis offers the possibility of de-coupling (time, place and scale), easy handling of the liquids and a more consistent quality compared to any solid biomass with fast pyrolysis a clean liquid is produced as an intermediate suitable for a wide variety of applications. The rotating cone was a key feature of this reactor: biomass particles at room

Figure 1. Schematic diagram of the fast pyrolysis unit.

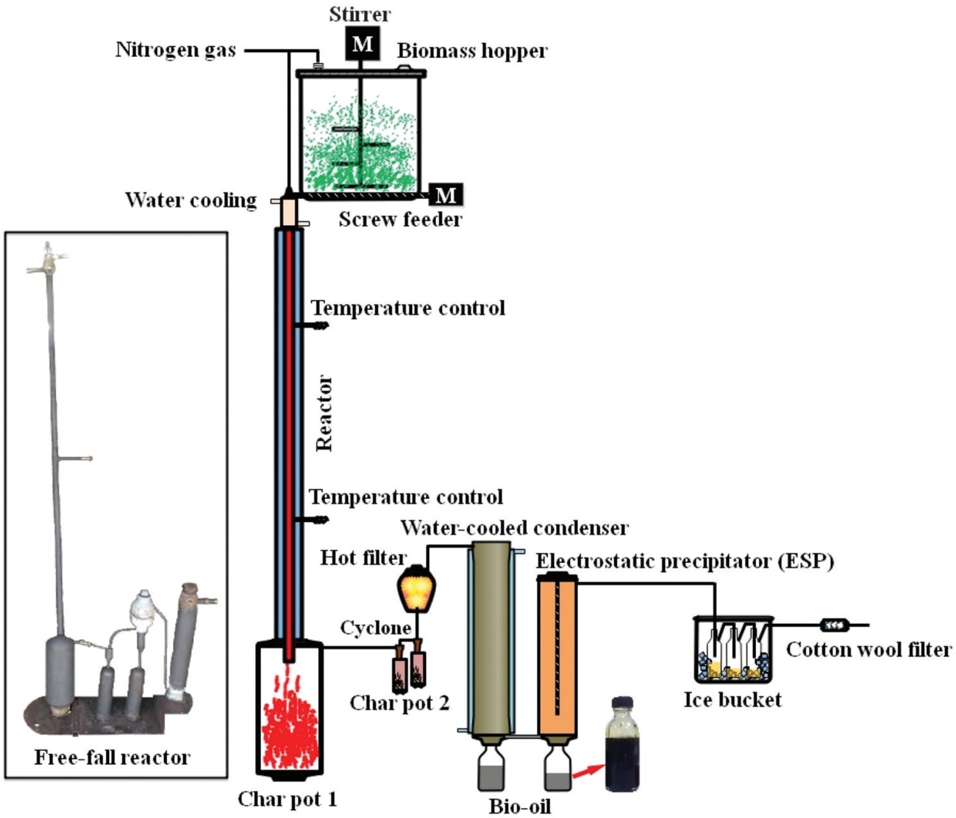


Table 1. Pyrolysis conditions of cassava residue

Parameters	Cassava residue	Cassava residue mixed volcanic rock	Cassava residue mixed ethanol
Pyrolysis temperature	450–550 °C	500 °C	500 °C
Biomass feed rate	200 g/h	200 g/h and 500 g of volcanic rock in hot filter	180 g/h of cassava residue mixed 20 g of ethanol

temperature and hot volcanic ash particles were introduced near the bottom of the cone, where the solids were mixed and transported upwards by rotation of the cone. This ensured rapid heating and a short gas phase residence time. A process flow diagram for fast pyrolysis process is given below. Experimental conditions are listed in Table 1. Triplicate runs were made for each experiment. Reproducibility was estimated as the standard deviation (S.D) of each pyrolysis temperature experiment.

2.3. Mass balance

Figures 3 and 4 report yields of bio-oil yield, char and non-condensable gas, derived by calculating the mass difference before and after the bio-oil experiment in Sets 1 and 2 against the mass of the char in storage pot 1 and 2 to the mass of biomass used. The gas yield was calculated by the difference from the measured products (Rueangsan et al., 2017).

2.4. Bio-oil analysis

The bio-oil products collected from the heavy bio-oil were analyzed for solids and ash content, pH, density, high heating value, viscosity and stability. Each analysis was performed in triplicate, following Rueangsan et al. (2018).

3. Results and discussion

The products gained from biomass fast pyrolysis consist of three parts: bio-oil, char, and gas. The bio oil was a dark brown viscous liquid, see, Figure 2.

3.1. Effect of pyrolysis temperature

The key factor for the proportion of pyrolysis products is the pyrolysis temperature. We investigated pyrolysis temperatures of 450, 500 and 550 °C. The yields of the different products are shown in Figures 3 and 4. Temperature directly influenced the yields: a low temperature produced a high volume of char. On the other hand, at higher temperatures, gas production increased.

Pyrolysis temperature was the main factor affecting fast pyrolysis products. Previous work suggests temperatures in the range 300–600 °C (Pattiya et al., 2012; Yogalakshmi et al., 2022). Our data (Figure 3) showed that ~500 °C led to a high volume of bio-oil. An appropriate

Figure 2. Bio-oil formed in this research.

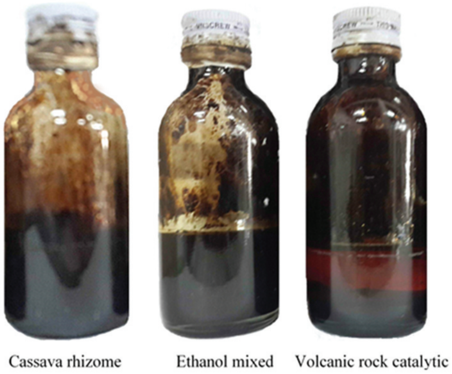


Figure 3. Effect of pyrolysis temperature: bars are labelled with the temperature and the yield of the indicated component at that temperature.

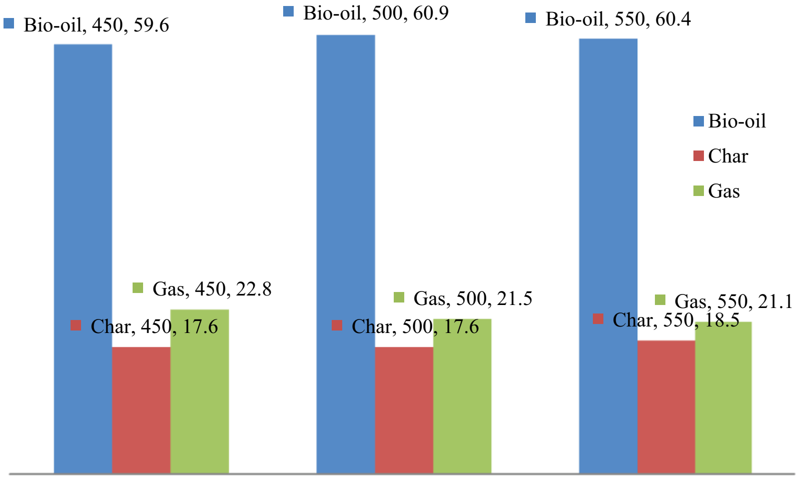


Figure 4. Effect of ethanol and volcanic rock.

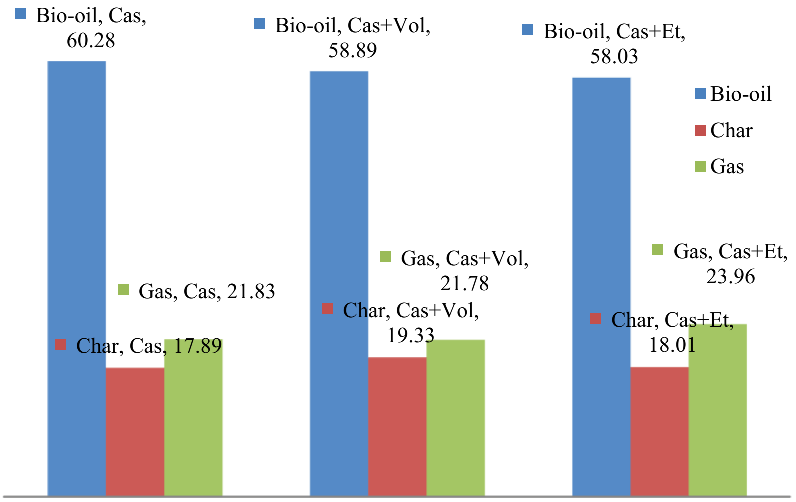


Table 2. Effect of pyrolysis temperature on bio-oil properties

Properties	Temperature (^{o}C)		
	450	500	550
pH	3.07 ± 0.1	3.06 ± 0.1	3.02 ± 0.1
Density (kg/m^3)	1110 ± 4	1118 ± 5	1110 ± 5
Viscosity (cSt)	35.0 ± 1.3	33.9 ± 1.2	36.3 ± 1.5
Stability	0.6 ± 0.1	0.5 ± 0.1	0.5 ± 0.1
Ash content	0.4 ± 0.1	0.3 ± 0.1	0.4 ± 0.1
Solid content	0.6 ± 0.1	0.6 ± 0.1	0.5 ± 0.1
HHV (MJ/kg)	18.4 ± 0.2	19.1 ± 0.2	19.8 ± 0.3

temperature helps transfer heat to the biomass, resulting in faster combustion and evaporation, and thus, increasing bio-oil yield. The bio-oil properties were measured from specific analysis of bio-oil viscous phase, because this was the most abundant and usable part; they are shown in Table 2. Bio-oil pH was ~3, in the pH 2–3 range of a typical bio-oil (IEA Bioenergy, 2022). Solids in bio-oil were ~0.5% by weight, within a normal range of 0.2 to 1 wt% (Czernik & Bridgwater, 2004; Lehto et al., 2014). Product density did not differ significantly with temperature, and fell into range of bio-oils in ASTM D7544—between 1100 and 1300 kg/m^3 (Oasmaa & Peacocke, 2010). The higher heating values also did not differ with temperature. Reported bio-oil higher heating values range from 12–28 MJ/kg, which can be used for oil-grade fuel (Elkhalifa et al., 2022). The viscosity at 500 °C was the lowest. Viscosity is a key property to judge usability: an improper viscosity may cause problems with injection systems in engine combustion chambers (Pattiya et al., 2012). The yields and properties of bio-oil shown in Figure 3 and Table 2 at three temperatures. A 500 C pyrolysis temperature led to optimum parameters, so it was used in the following experiments with additives.

Figure 3 reports the effect of pyrolysis temperature on the yield and composition of bio-oils obtained from the pyrolysis. The tests were conducted at reactor wall temperatures of 450 °C, 500 °C and 550 °C. Due to the relatively low heat transfer rates achieved between the reactor wall and the biomass particles, the temperature of the solid residue obtained bio-oil yields were close to those reported for fluidized bed reactors. The maximum oil yield was obtained at a reactor wall temperature of 600 °C.

3.2. Effect of mediators—ethanol and volcanic rock

Yields and properties from cassava roots, as the biomass, and two types of mediators, volcanic rock and ethanol, are shown in Figure 4 and Table 3. The yield of bio-oil slightly decreased when ethanol was added to the feed. The yield of bio-oil was 58–60 wt%, but ethanol in the feed increased the gas yield. Also, after installing volcanic rock in the heat vapor filter set, the HHV clearly increased.

Figure 4 shows the effect of solvent on the pyrolysis. Alcohols were chosen as they can be readily produced from renewable resources by fermentation of sugar cane and cassava and other common crops. The important fuel properties of blended fuels covering both the binary and ternary systems are provided.

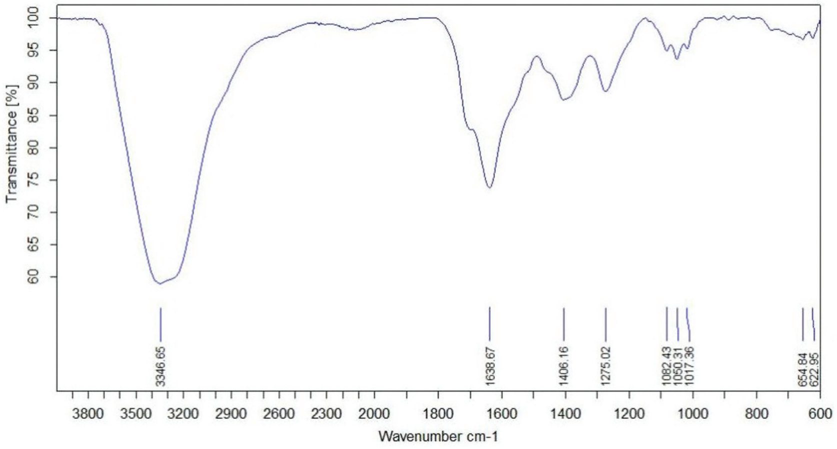
3.3. Functional groups of bio-oil

The functional groups of the bio-oils were analyzed by Fourier Transform Infrared Spectroscopy (FTIR), as shown in Figure 5. All bio-oils contained oxygenated and hydrocarbon groups, identified by broad O-H vibration peaks in the 3200–3600 cm^{-1} region and C = O stretching vibrations in the 1702 to 1638 cm^{-1} region. Alkane (C-H, 2850 to 2962 cm-1) stretchings became more prominent in the volcanic rock samples due to catalytic degradation. Generally, application of volcanic rock led to much wider range of compounds: many new peaks appeared in the “fingerprint” region (<1400 cm^{-1}). The principal active components are zeolites, which have been shown to be good catalysts in many other studies: they facilitate the degradation of the original cellulose, hemicellulose and lignin components of the biomass.

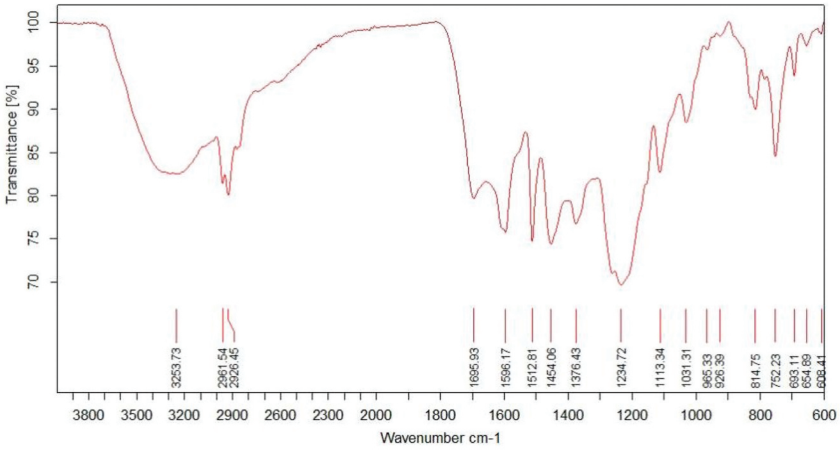
Table 3. Effect of ethanol and volcanic rock on bio-oil properties

Properties	Cassava residues	Cassava residues + volcanic rock	Cassava residues + ethanol
pH	3.1 ± 0.1	3.9 ± 0.1	3.2 ± 0.1
Density (kg/m^3)	1112 ± 5	1092 ± 5	1110 ± 4
Viscosity (cSt)	35.1 ± 1.2	37.8 ± 1.7	35.1 ± 1.2
Stability	0.5 ± 0.1	0.6 ± 0.1	0.3 ± 0.1
Ash content	0.4 ± 0.1	0.5 ± 0.1	0.3 ± 0.1
Solid content	0.6 ± 0.1	0.5 ± 0.1	0.5 ± 0.1
HHV (MJ/kg)	19.1 ± 0.7	23.6 ± 0.2	19.4 ± 0.4

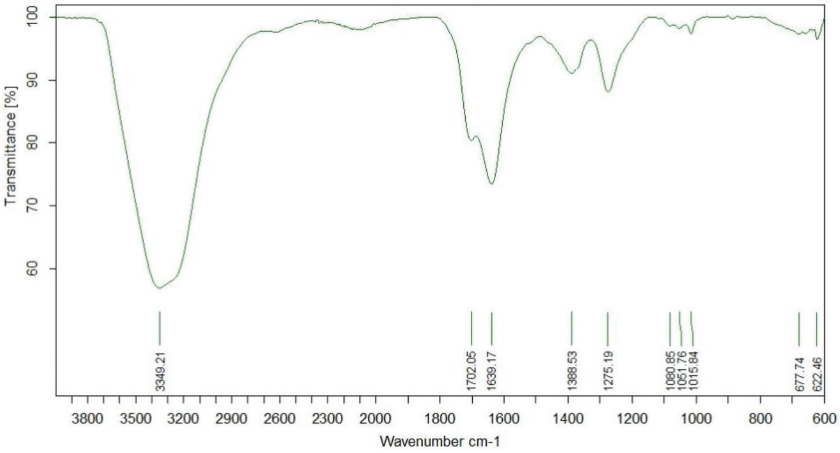
Figure 5. Fourier transform infrared spectrum of bio-oil.



Bio oil from cassava residues



Bio oil from cassava residues with volacnic rock



Bio oil from cassava residues with volcanic rock and ethanol

Further analysis to identify potential useful products which might be separated from the bio-oil is planned.

4. Conclusions

Adding volcanic rock significantly enhanced the yield of bio-oil from fast pyrolysis of cassava residue in a free fall reactor. The volcanic rock was more effective than ethanol as an additive, increasing the HHV from to 23.6 MJ/kg from 19.8 MJ/kg, with little change in other properties, although viscosity increased by as much as 10% (37.8 cSt versus the best result at 500 °C of 33.9 cSt), pH was also higher at 3.9 vs typically 3.1. Zeolites, known as effective catalysts for the breakdown of cellulosic material, in the volcanic rock, notably increased the range of low molecular weight compounds found in the bio-oil increasing the HHV and also increasing pH, by removing acidic groups and increasing the attractiveness of the bio-oil as a fuel by lowering corrosion in engines.

The key contribution of this work was that readily available and potentially cheaper volcanic rock achieved improvemments equal or better than ethanol as an additive.

Noting that volcanic rock is a low cost source of pyrolysis catalysts, we plan to extend this work to identify and characterize the bio-oil products: we hypothesize that they may be economic sources of additional chemicals.

Acknowledgements
We thank the laboratory staff at the Faculty of Engineering and Industrial Technology, Kalasin University. We also thank the Department of Chemistry, King Mongkut's Institute of Technology Ladkrabang for access to their FT-IR machine.

Funding
The authors received no direct funding for this research.

Author details
Koson Rueangsan[1]
ORCID ID: http://orcid.org/0000-0002-3037-8325
Adcha Heman[1]
Pakkip Kraisoda[1]
Homhuan Tasarod[2]
Keyoon Duanguppama[2]
Somsuk Trisupakitti[3]
E-mail: somsukrmu@gmail.com
ORCID ID: http://orcid.org/0000-0002-3865-1285
John Morris[4]
[1] Department of Agricultural Machinery Engineering, Kalasin University, Thailand.
[2] Department of Mechanical Engineering, Kalasin University, Thailand.
[3] Department of Chemistry, Rajabhat Maha Sarakham University, Maha Sarakham Thailand.
[4] School of Industrial Education and Technology, King Mongkut's Institute of Technology Ladkrabang, Thailand.

Disclosure statement
No potential conflict of interest was reported by the author(s).

Citation information
Cite this article as: Bio-oil production via fast pyrolysis of cassava residues combined with ethanol and volcanic rock in a free-fall reactor, Koson Rueangsan, Adcha Heman, Pakkip Kraisoda, Homhuan Tasarod, Keyoon Duanguppama, Somsuk Trisupakitti & John Morris, *Cogent Engineering* (2023), 10: 2156054.

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