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Improvement of Bio-oil Quality through the use of Natural Zeolite Catalysts in the Fast Pyrolysis of Cassava Rhizomes using a Free-fall Reactor

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Abstract

This research investigated the properties of bio-oil derived from cassava rhizomes using natural zeolite catalyst within the hot gas filtration unit of a free-fall reactor. The reactor was controlled at 450, 500, and 550°C, while the condenser unit was maintained at a constant temperature. Cassava rhizome particle, 0.2 to 0.5 mm in size, were input at 200 g/h. Results indicated a maximum bio-oil yield of approximately 60 wt% at 500°C without catalysis. Bio-oil collected from the electrostatic precipitator exhibited higher density and viscosity compared to that from the water-cooled condenser, and these properties were not significantly affected by the catalyst. However, the catalyst enhanced the bio-oil's higher heating value (HHV) by approximately 17%, potentially due to increased hydrocarbon content and decreased oxygen levels, particularly in the electrostatic precipitator-derived bio-oil. These findings suggest the potential of natural zeolite catalysts to improve bio-oil quality.

Keywords: Fast pyrolysis; Free-fall reactor; Natural zeolite; Cassava rhizomes.
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1. Introduction

Fast pyrolysis has garnered significant research interest as a technology for converting biomass into valuable products, notably bio-oil, char, and non-condensable gases. Bio-oil presents potential as an alternative energy source to fossil fuels and as a feedstock for the chemical industry.[1] While bio-oil from fast pyrolysis exhibits properties akin to heavy fuel oil, offering a route to reduce fossil fuel dependence and promote sustainable energy, its high water and oxygen content leads to instability and limits its commercial applicability.[2] This elevated water and oxygen content impedes storage and transportation, concurrently lowering its heating value, rendering it unsuitable for high-energy applications. Enhancing bio-oil's chemical properties to improve stability and heating value is therefore critical for its widespread adoption.

The performance and quality of fast pyrolysis-derived bio-oil are contingent upon several key parameters, including biomass type, particle size, pyrolysis temperature, and reactor configuration.[3] These factors influence bio-oil yield and can be optimized for different biomass feedstocks. For instance, high-moisture biomass necessitates precise temperature control during pyrolysis to yield high-quality bio-oil. The optimal temperature range for maximizing bio-oil yield in fast pyrolysis is typically 400-600°C,[4] thereby enhancing its efficiency and stability.

Beyond temperature control, catalysis plays a crucial role in upgrading bio-oil quality.[5] Catalysts effectively reduce water and oxygen content and increase the concentration of aromatic hydrocarbons, thereby enhancing bio-oil's value and application potential.[5] However, inherent limitations such as high oxygen content, water, solids, ash, and viscosity still result in a lower heating value compared to petroleum fuels,[6] necessitating further property enhancement.

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Synthesized catalysts, such as ZSM-5,[2] have demonstrated significant efficacy in improving bio-oil quality.[7] Furthermore, studies have explored various catalytic materials and application methods to optimize process efficiency and bio-oil quality.[8] Integrating a hot vapor filter within the reactor can enhance the decomposition of cellulose, hemicellulose, and lignin, as catalysts can modify the chemical structure of bio-oil, particularly by reducing water and oxygen content – key factors affecting stability and heating value.[9] Catalysts also promote cracking and deoxygenation reactions of oxygen-rich compounds like carboxylic acids and phenols, thus improving overall bio-oil quality[10] and increasing the proportion of valuable aromatic hydrocarbons[11-16] relevant to the chemical industry.[3]

This research investigates the application of natural zeolite catalysts within the hot gas filtration unit of a free-fall reactor for the fast pyrolysis of cassava rhizomes, with the primary objective of enhancing the quality of the produced bio-oil. Furthermore, this study highlights the utilization of agricultural waste biomass, thereby contributing to the advancement of sustainable and environmentally sound alternative energy practices.

2. Experimental section

2.1 Biomass used in the experiment

This study investigated the application of natural zeolite catalysts within the hot gas filtration unit of a free-fall reactor during the fast pyrolysis of cassava rhizomes to enhance the quality of the produced bio-oil. Furthermore, this research underscores the utilization of agricultural waste biomass, thereby contributing to the advancement of sustainable and environmentally sound alternative energy practices.

2.2 Properties of the catalyst

The natural zeolite catalyst employed in this experiment was activated at 500°C for 4 hours to enhance its catalytic activity, thereby ensuring suitable properties for the fast pyrolysis process. The catalyst exhibited a bulk density of 700 kg/m³ and a particle density of 1165 kg/m³, suggesting its capacity for mass support and transfer. These density values provide insights into the catalyst's physical characteristics. Surface area and porosity analysis revealed a surface area of 14 m²/g, indicative of a substantial surface area beneficial for adsorption, a crucial factor in improving reaction efficiency during pyrolysis. The catalyst possessed a mean pore diameter of 25.4 nm and a total pore volume of 68.3 mm³/g, reflecting its potential for water retention and effective substance distribution. The Si/Al ratio of 12.34 provides information regarding the zeolite's structural characteristics and performance capability within this process.

2.3 Equipment used in the experiment

As depicted in Fig. 1, the free-fall reactor system incorporates a 12V DC motor for continuous and uniform biomass agitation during pyrolysis. A biomass storage tank precedes the reactor, which is constructed from 304 stainless steel with a 16 mm internal diameter and a height of 1.20 m. This reactor facilitates biomass heating and efficient pyrolysis, with the stainless-steel construction providing resistance to high temperatures and process-generated chemicals. A reactor temperature control unit maintains the specified operating temperature for optimal results. The char byproduct is collected in a dedicated char pot. A cyclone unit separates non-condensable gases from the hot vapor stream. Subsequently, a hot filter unit purifies the hot vapors before entering a water-cooled condenser, where the vapors are condensed into bio-oil. An electrostatic precipitator, operating at 14 kV, further removes fine particulates from the vapor stream to enhance bio-oil purity. Finally, the produced bio-oil is stored in a designated bio-oil storage tank for subsequent analysis.

The experimental conditions involved a biomass feed rate of 200 g/h in a nitrogen gas flow rate at 4 L/min. The reactor, cyclone, and hot gas filter temperatures were maintained at 450, 500, and 550°C, respectively, while the water-cooled condenser was set at 30°C. Precise temperature control is a critical parameter for optimizing bio-oil production efficiency.

2.4 Analysis of yield

The yield analysis for bio-oil and char was based on the mass difference of the initial biomass compared to the respective product mass post-experiment. This calculation provides an accurate evaluation of pyrolysis process efficiency and enables yield comparisons across different conditions. The non condensable gas yield was determined by the difference between the total yield and the yields of other products, as detailed in Eq. (1)-(3).[17] These equations facilitate the efficient quantification of product yields.

$$Y_{bio-oil} = \left[\frac{\sum W_{bio-oil}}{W_{biomass}}\right] \times 100\% \tag{1}$$

$$Y_{char} = \left[\frac{\sum W_{char}}{W_{biomass}}\right] \times 100\% \tag{2}$$

$$Y_{gas} = 100 - Y_{bio-oil} - Y_{char} \tag{3}$$

where

$Y_{\text{Bio oil}}$ = Bio oil Yield (Percentage by mass)
Y_{char} = Char Yield (Percentage by mass)]
Y_{gas} = Non-condensable Gas Yield (Percentage by mass)
$W_{\text{bio - oil}}$ = The mass of the bio-oil obtained from the water-

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cooled condenser and electrostatic precipitator (grams)
W_{char} = The mass of the char obtained from the char pot and cyclone unit (grams)
$W_{biomass}$ = The mass of the biomass used (grams)

2.5 Analysis of bio-oil properties

2.5.1 The higher heating value (HHV) of the bio-oil was determined using an Art.2060/2070 bomb calorimeter following ASTM D240 standards. This method, detailed in ASTM D240-00 for assessing the heat of combustion of liquid hydrocarbon fuels, involves combusting a weighed fuel sample within a bomb calorimeter pressurized to 3.0 MPa with oxygen. The resulting temperature change during controlled combustion is measured and subsequently used to calculate the heat of combustion in MJ/kg (ASTM D240-00).[18]

2.5.2 The pH of the bio-oil was determined using a UB-10 Denver Instrument pH Meter. This measurement is based on potentiometry, where the acidity or alkalinity of a solution is quantified by measuring the electrical potential resulting from the activity of hydrogen ions (H^+). Variations in pH correspond to changes in this electrical potential, which the pH meter converts into a direct pH reading. Prior to measurement, the pH meter was calibrated using standard solutions of known pH values (*e.g.*, 4, 7, and 10). Subsequently, the pH sensor (electrode) was immersed in the bio-oil sample, and the pH value was recorded once a stable reading was obtained. To ensure accuracy, the electrode was thoroughly cleaned to prevent contamination from other chemicals.

2.5.3 The density of the bio-oil was determined following ASTM D4052 standards. This method involves measuring the mass of a known volume of the liquid sample at a controlled temperature using a calibrated density meter to ensure accuracy. The density (ρ) is calculated using the formula $\rho = m/V$, where m is the mass and V is the volume, with the result typically expressed in g/mL or kg/m³ (ASTM D4052-18).[19]

2.5.4 The solid content of the bio-oil was determined according to ASTM D7579 standards using ethanol as a solvent and Whatman No. 3 filter paper.[20] This method quantifies the insoluble solid content in oils or liquids through solvent extraction and gravimetric analysis. The procedure involves extracting the solids with a solvent (ethanol in this case) and separating them by filtration using Whatman No. 3 filter paper. The residue retained on the filter paper is then dried and weighed. The solid content is calculated by comparing the mass of the dried residue to the initial mass of the bio-oil sample (ASTM D7579-16).[21]

2.5.5 The ash content of the bio-oil was determined according to ASTM D482 standards. This method quantifies the non-combustible residue in oils or fuels by subjecting a weighed sample to combustion in a high-temperature furnace (550°C) until only ash remains. The residual ash is then weighed, and the ash content is calculated as a percentage of the initial sample mass. This analysis provides an indication of the amount of non-combustible solids present, serving as a quality and cleanliness indicator for the product (ASTM D482-17).[22]

2.5.6 The viscosity of the bio-oil was determined according to ASTM D445 standards using a Cannon-Fenske Opaque Viscometer for kinematic viscosity measurement.[20] This method assesses the resistance of a fluid to flow by measuring the time required for a specific volume of the liquid to pass through a calibrated capillary tube under gravity at a controlled temperature. The kinematic viscosity is then calculated by multiplying the measured flow time by the viscometer's calibration constant, yielding results typically expressed in centistokes (cSt). Dynamic viscosity can subsequently be calculated by multiplying the kinematic viscosity by the fluid's density (ASTM D445-17).[23]

2.5.7 The stability of the bio-oil was assessed according to the method outlined by Oasmaa et al. (2011),[24] which involves heating the bio-oil at 80°C for 24 hours to evaluate property changes under elevated temperature exposure. The procedureentailed preparing a 50 mL bio-oil sample and subjecting it to the specified heating duration. Subsequently, key properties, such as viscosity or color, were measured and recorded both before and after the heating period. The differences observed in these properties serve as indicators of the bio-oil's thermal stability.

3. Results and discussion

3.1 Yield of bio-oil, char and gas

Table 1 illustrates the fast pyrolysis of cassava rhizomes at 450, 500, and 550°C, revealing a maximum bio-oil yield of 60 wt% at 500°C under non-catalytic conditions. The introduction of natural zeolite catalysts induced chemical alterations in the bio-oil compared to the non-catalyzed process. Across the tested temperature range, the use of natural zeolite catalysts resulted in a reduced bio-oil yield, averaging approximately 3 wt%, a trend consistent with observations in non-catalytic fast pyrolysis of cassava stems, rhizomes, and eucalyptus bark.[25,26] This decrease in bio-oil yield may be attributed to the catalyst promoting the decomposition of less desirable compounds. Furthermore, the reduced bio-oil yield with natural zeolite catalysts could also be a consequence of increased non-condensable gas production, potentially due to secondary decomposition reactions of pyrolysis vapors. This process led

Table 1: The yield of the products from the fast pyrolysis process.

Product	Temperature (°C)	Product Yield (%)	
		Cassava Rhizomes	Use of Natural Zeolite Catalyst
Bio-oil	450	59.6±0.6	58.1±0.4
	500	60.9±0.7	57.9±0.6
	550	60.4±0.5	58.4±0.6
Char	450	17.6±0.6	19.6±0.6
	500	17.6±0.7	19.2±0.5
	550	18.5±0.5	19.0±0.5
Gas	450	22.8±0.6	22.3±0.8
	500	20.9±0.5	22.9±0.6
	550	21.1±0.6	22.6±0.6

Table 2: Properties of bio-oil. Analysis Temperature (°C).

Analysis	Temperature (°C)	Cassava Rhizomes		Use of Natural Zeolite Catalyst		Standard ASTM D7544[27]	
		Condenser	ESP	Condenser	ESP	Grade D	Grade G
pH	450	3.5 ± 0.2	3.8 ± 0.1	4.4 ± 0.1	4.0 ± 0.2	-	-
	500	3.5 ± 0.1	3.9 ± 0.1	4.2 ± 0.3	4.0 ± 0.2	-	-
	550	3.4 ± 0.1	3.8 ± 0.1	4.0 ± 0.3	3.9 ± 0.3	-	-
+Density (kg/m^3)	450	1.11 ± 1.7	1.21 ± 1.4	1.11 ± 1.3	1.28 ± 1.4	1.1–1.3	1.1–1.3
	500	1.10 ± 1.2	1.31 ± 1.1	1.12 ± 1.2	1.25 ± 1.2		
	550	1.11 ± 1.2	1.34 ± 1.2	1.12 ± 1.2	1.28 ± 1.2		
Viscosity (cSt)	450	22.4 ± 0.3	44.5 ± 0.2	22.7 ± 0.3	38.2 ± 0.4	≤ 125	≤ 125
	500	21.9 ± 0.2	48.2 ± 0.5	22.0 ± 0.2	41.9 ± 0.6		
	550	23.7 ± 0.2	49.3 ± 0.9	23.1 ± 0.3	42.1 ± 0.4		
Stability	450	0.61 ± 0.2	0.65 ± 0.4	0.62 ± 0.1	0.66 ± 0.2	-	-
	500	0.55 ± 0.1	0.66 ± 0.3	0.56 ± 0.1	0.65 ± 0.2		
	550	0.53 ± 0.1	0.60 ± 0.4	0.56 ± 0.1	0.55 ± 0.3		
Ash Content (%)	450	0.45 ± 0.2	0.28 ± 0.3	0.42 ± 0.2	0.32 ± 0.3	≤ 0.15	≤ 0.25
	500	0.42 ± 0.2	0.24 ± 0.2	0.52 ± 0.3	0.35 ± 0.3		
	550	0.42 ± 0.2	0.24 ± 0.2	0.48 ± 0.2	0.34 ± 0.3		
Solid Content (%)	450	0.65 ± 0.2	0.46 ± 0.1	0.81 ± 0.2	0.48 ± 0.1	≤ 0.25	≤ 2.5
	500	0.66 ± 0.3	0.56 ± 0.1	0.77 ± 0.2	0.47 ± 0.2		
	550	0.58 ± 0.2	0.55 ± 0.2	0.58 ± 0.2	0.45 ± 0.2		
HHV (MJ/kg)	450	18.52 ± 0.2	19.25 ± 0.1	21.8 ± 0.2	24.66 ± 0.1	≥ 15	≥ 15
	500	18.56 ± 0.1	19.55 ± 0.1	20.0 ± 0.2	25.89 ± 0.1		
	550	19.44 ± 0.1	19.81 ± 0.1	21.8 ± 0.1	25.24 ± 0.1		

to an increased non-condensable gas yield, averaging 20-22 wt%. In contrast, the char yield remained relatively stable at approximately 19 wt%, similar to the non-catalytic experiment, suggesting that the catalyst primarily influenced the bio-oil and gas yields.

3.2 Properties of bio-oil

The analysis of bio-oil properties obtained from both water-cooled condenser and the electrostatic precipitator, encompassing pH, density, viscosity, stability, ash content, solid content, and higher heating value, is crucial for assessing bio-oil quality and its suitability for diverse industrial applications. These properties were measured and compared between the two collection methods, as each can differentially affect the resulting bio-oil characteristics. Evaluating these properties provides insights into the bio-oil's

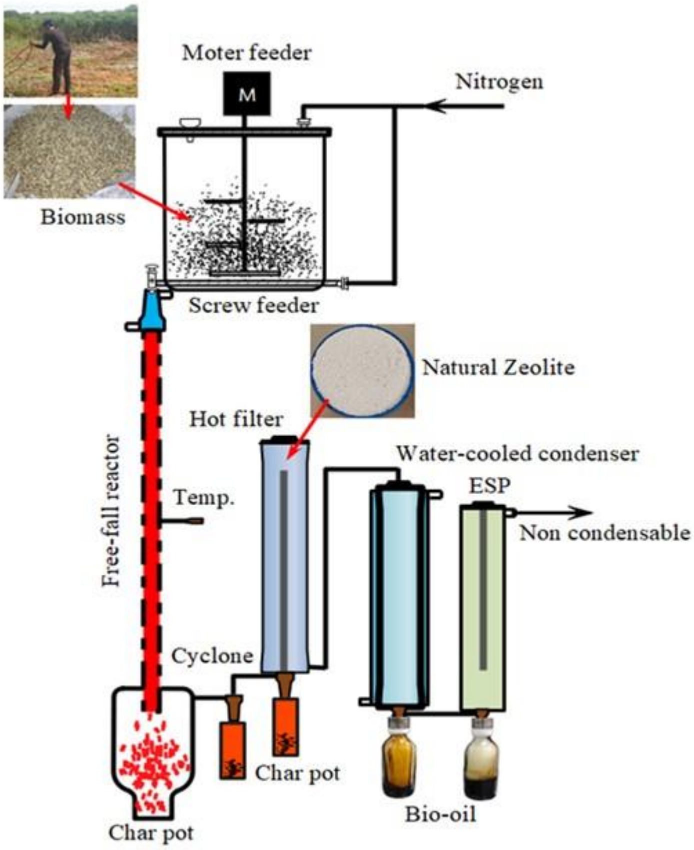

Fig. 1: Free-Fall Reactor.

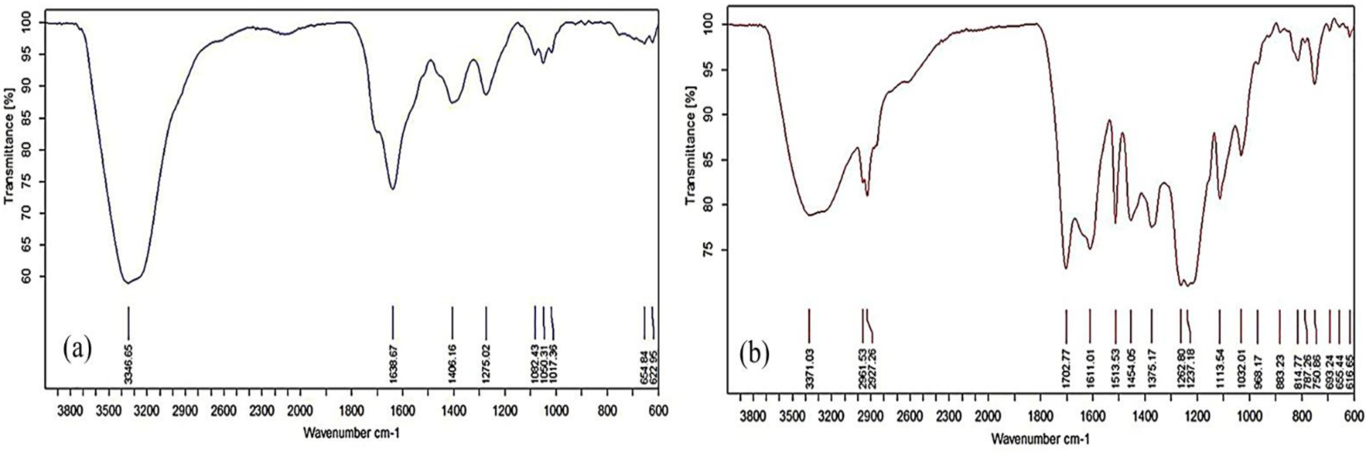

Fig. 2: FTIR Spectra of (a) Bio-oil from cassava rhizome and (b) Bio-oil with the use of natural zeolite catalyst.

stability and long-term usability. Detailed results of these property analyses are presented in Table 2.

3.3 Functional groups of bio-oil

As illustrated in Fig. 2, Fourier Transform Infrared Spectroscopy (FTIR) analysis revealed the presence of various functional groups in the bio-oil. The broad absorption band in the 3200-3600 cm^{-1}. regions indicates the presence of oxygen-containing (O-H) groups, reflecting moisture content and the potential for chemical reactivity. The presence of carbonyl groups (C=O) in the 1702-1638 cm^{-1}. region suggests the formation of reactive carbon species and chemical structural transformations within the bio-oil. Notably, the stretching of alkene (C-H) groups in the 2850-2962 cm^{-1}. range is more pronounced in bio-oil produced with natural zeolite catalysts, indicating the formation of key bio-oil components and increased compositional complexity, as further evidenced by the spectral region below 1400 cm^{-1}. These findings suggest that the natural zeolite catalyst facilitates the breakdown of cellulose, hemicellulose, and lignin, thereby enhancing the conversion of less desirable components into potentially valuable compounds during the pyrolysis process.

4. Conclusion

The incorporation of natural zeolite catalysts within the rapid pyrolysis hot vapor filter system resulted in a decrease in bio-oil yield, reaching a minimum of 57 wt% at 500°C. This reduction may be attributed to the catalyst's influence on the bio-oil's chemical structure. The bio-oil collected from both water condensation and electrostatic precipitator systems exhibited a pH range of 3.6-4.0, indicating acidity that could impact its commercial applications. The density ranged from 1.10 to 1.28 kg/m³, and the viscosity varied from 22.7 to 43.8 cSt, with bio-oil from the electrostatic precipitator displaying higher viscosity, potentially affecting its flow properties in fuel systems. The stability was approximately 0.60. Ash content ranged from 0.29 to 0.45 wt%, and solid content ranged from 0.50 to 0.86 wt%, with the electrostatic precipitator yielding bio-oil with lower solid content, which can enhance product cleanliness and quality. While the overall bio-oil properties were not drastically altered, the use of natural zeolite catalysts significantly increased the higher heating value (HHV) by approximately 17% (23.23 MJ/kg). This enhanced HHV signifies greater energy content per unit mass, potentially leading to more efficient energy production.

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Conflict of Interest

There is no conflict of interest.

Supporting Information

Not applicable.

CRediT Statement

Koson Rueangsan: Conceptualization, Formal analysis, Investigation, Supervision, Validation, Visualization. **Pakkip Kraisoda:** Data curation. **Homhuan Tasarod:** Investigation, Software, Writing. **Sayun Phansomboon:** Methodology, Project administration. **Piyachat Wiriyaampaiwong:** Resources. **Nuttapan Promsampao:** Validation. **Somsuk Trisupakitti:** Writing – original draft and review & editing.

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