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DATA NOTE

Physicochemical Pretreatment on Lesser-Known Shrub Biomass for Bio-Pellet Production

[version 1; peer review: 2 approved with reservations]

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Abstract

Abstract*

Rising demand for sustainable energy has renewed interest in forest biomass, yet shrubs remain underutilized as their wood has lower strength, durability, and dimensions, relegating it to low-value uses like firewood. Nonetheless, shrubs grow rapidly, are easy to cultivate, and show high ecological adaptability, making them promising bioenergy feedstocks where woody biomass is limited or unevenly distributed. This study aimed to generate a comprehensive dataset evaluating whether physicochemical pretreatment (solvent-based extraction followed by torrefaction) can improve key fuel properties of bio-pellet feedstock from four lesser-known shrub species (*Lagerstroemia speciosa*, *Ganophyllum falcatum*, *Myrsine avenis*, and *Mischocarpus sundaicus*), with particular emphasis on reducing volatile matter that commonly remains high even after torrefaction and can prevent compliance with Indonesian quality standards. Branch particles (20–40 mesh; 0.41–0.82 mm) collected in South Sulawesi, Indonesia, were extracted at a 1:7 solid-to-liquid ratio using distilled water, ethanol, hexane, or sequential ethanol+hexane at 25 °C (6 h; or 3 h + 3 h sequential) and 50 °C (2 h; or 1 h + 1 h sequential), washed until no solvent odor remained, oven-dried (100 °C, 24 h), and torrefied uniformly at 200 °C for 10 min. Moisture content, weight loss (after extraction and after torrefaction), total weight loss, yield, volatile

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Approval Status ? ?

	1	2
version 1 16 Apr 2026	? view	? view

1. **Wasoh Helmi**, University Putra Malaysia, Serdang, Malaysia
2. **Sarah Augustina**, National Research and Innovation Agency (BRIN), Bogor, Indonesia

Any reports and responses or comments on the article can be found at the end of the article.

matter, and gross calorific value were determined and summarized as means with standard deviations (for moisture and volatile matter) across all species–solvent–temperature combinations. The dataset indicates that solvent extraction coupled with torrefaction can reduce volatile matter for the most shrubs species, but values remain relatively high and do not yet meet SNI 8675:2018 thresholds for *M. avenis*, underscoring the need for further optimization. Despite limitations in extraction conditions, torrefaction settings, solvent-removal verification, and replicates, the dataset provides evidence to guide process optimization and supports broader utilization of shrub biomass for renewable energy applications.

Keywords
Bio-pellet, Calorific value, Chemical extraction, Shrub, Torrefaction, Volatile matter, Yield

This article is included in the Energy gateway.

This article is included in the Plant Science gateway.

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Author roles: Pangestu KTP: Conceptualization, Funding Acquisition, Methodology, Supervision, Writing – Original Draft Preparation; **Yunianti AD**: Project Administration, Validation, Writing – Review & Editing; **Baharuddin B**: Methodology, Resources, Writing – Review & Editing; **Rahma SA**: Investigation, Resources, Visualization; **Fahira L**: Investigation, Resources, Visualization; **Marwanto M**: Formal Analysis, Validation, Writing – Review & Editing; **Kutana AN**: Data Curation, Formal Analysis, Writing – Review & Editing

Competing interests: No competing interests were disclosed.

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Introduction

The growing global demand for sustainable energy sources has renewed attention toward biomass as a versatile and renewable resource.[1] Among various forms of forest-derived biomass, shrubs represent a particularly underutilized yet abundant vegetation group. Although morphologically similar to trees, shrub wood generally possesses lower strength, limited durability, and smaller dimensions, restricting its use to low-value applications such as firewood. However, shrubs offer notable advantages, such as rapid growth, ease of cultivation, and high ecological adaptability.[2] It positions them as promising candidates for bioenergy development,[3,4,5] especially in regions where woody biomass resources are limited or unevenly distributed.

Despite their potential, previous studies on bio-pellets derived from lesser-known shrub species have shown that key fuel properties, particularly calorific value and volatile matter content, frequently fall below the quality requirements established in the Indonesian National Standard (SNI 8675–2018: Biomass pellets for energy).[5,6] While thermal pre- and post-treatment through torrefaction has been shown to improve energy density, volatile matter concentrations remain high and continue to hinder combustion efficiency and emissions performance.[3] This condition underscores the need for additional pretreatment steps capable of extracting low-energy compounds prior to thermal processing.

To address this challenge, the present study generated a comprehensive dataset investigating the effectiveness of physicochemical pretreatment, specifically solvent-based extraction (chemical treatment) followed by torrefaction (physical treatment) in modifying the fuel properties of four lesser-known shrub species (*L. speciosa*, *G. falcatum*, *M. avenis*, and *M. sundaicus*).[7] The data were produced to evaluate how different solvents (distilled water, ethanol, hexane, and ethanol–hexane) and extraction temperatures influence moisture content, weight loss, yield, volatile matter content, and calorific value. By systematically comparing these conditions across species, the dataset aims to clarify which pretreatment strategies most effectively reduce volatile compounds while maintaining or enhancing the energy potential of shrub biomass. Enhancing the quality of these materials not only increases their suitability as renewable fuel sources but also promotes the broader utilization of shrub vegetation in sustainable energy systems. The dataset thus provides foundational evidence for optimizing chemical extraction–torrefaction processes and informs future efforts to establish shrubs as viable feedstock for renewable energy applications.

Materials and methods

Study site, materials, and sample collection

Four lesser-known shrub species, including *Lagerstroemia speciosa* (Local name: Bungur), *Ganophyllum falcatum* (Locong), *Myrsine avenis* (Latte), and *Mischocarpus sundaicus* (Terasa) were selected as biomass feedstocks. The plant materials were taxonomically identified by botanist Nasri, and voucher specimens (Voucher No. WAN0005748/*L. speciosa*, No. WAN0021952/*G. falcatum*, No. WAN0017752/*M. avenis*, and No. WAN0008828/*M. sundaicus*) have been deposited in the Herbarium Wanariset, located in Kutai Kartanegara Regency, East Kalimantan Province, Indonesia (−0.997047, 116.976286).

Branches were collected from the Educational Forest Area of Hasanuddin University, Maros Regency, South Sulawesi, Indonesia (−5.005145, 119.767224). Sample preparation through to pretreatment was conducted at the Laboratory of Forest Products Utilization and Processing, Faculty of Forestry, Hasanuddin University, Makassar, Indonesia (−5.130221, 119.485481). Subsequent property testing was performed at the Environmental and Forestry Laboratory, Faculty of Forestry, Hasanuddin University, Makassar, Indonesia (−5.130277, 119.484054), and the gross calorific value analysis was carried out at the Chemical Service Laboratory of the Center for Poultry and Livestock Assembly and Testing, Bogor, Indonesia (−6.663145, 106.857261).

Sample preparation

Collected shrub branches including the bark were cleaned and sun-dried to air-dry condition (moisture content <12%). The material was chopped using a flaker machine. It was then comminuted with a hammer mill to produce smaller particle, and sieved using a wire mesh to obtain particles retained between 20 and 40 mesh (0.41–0.82 mm). Only this fraction was used in subsequent experiments.

Physicochemical pretreatment overview

The pretreatment comprised a chemical extraction (soaking) followed by a physical extraction (torrefaction). Chemical extraction was designed to remove volatile and other low-energy constituents prior to thermal conditioning. Torrefaction then provided a uniform thermal step for all samples. A process flow is provided in the study's method schematic (Figure 1).

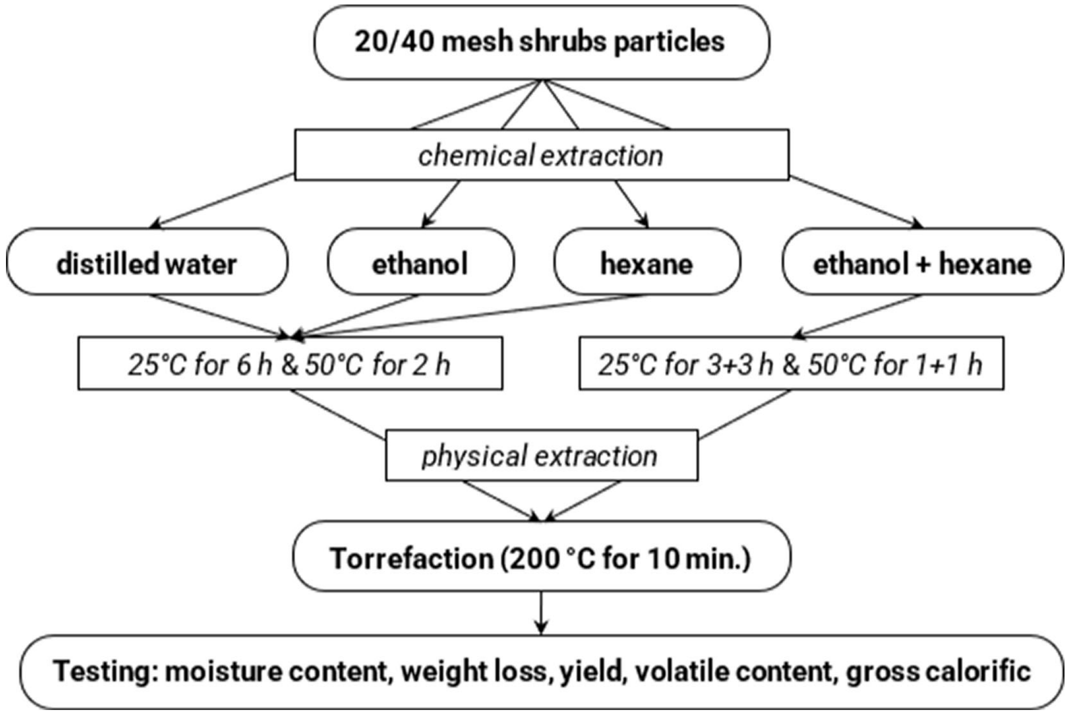

Figure 1. A schematic study methods of physicochemical treatment on shrub biomass.

Chemical extraction

For each run, 100 g (oven-dry basis) of shrub particles were placed in an Erlenmeyer flask and contacted with solvent at a 1:7 (wt/wt) solid-to-liquid ratio. Four solvent conditions were tested, including distilled water, ethanol, hexane, and a sequential ethanol + hexane treatment. Extractions were conducted at 25 °C for 6 h (room temperature) and at 50 °C for 2 h (elevated temperature kept below solvent boiling points). To limit evaporation, especially at 50 °C, flasks were covered with aluminum foil. Suspensions were manually stirred every hour for approximately 30 s to promote mass transfer. For the sequential ethanol + hexane condition, soaking was performed consecutively in ethanol then hexane, with 3 h + 3 h at 25 °C and 1 h + 1 h at 50 °C. After extraction, slurries were vacuum-filtered and the retained solids were washed repeatedly with 80 °C distilled water until no solvent odor remained. This practical criterion was used to confirm solvent removal. The solids were then dried in a Memmert UN55 oven at 100 °C for 24 h.

Physical extraction (torrefaction)

After the chemical extraction, the dried-extracted samples were wrapped in aluminum foil and torrefied at 200 °C for 10 min in an oven. This temperature was aligned with that used in previous studies,[3] while the duration was shortened to improve the efficiency without significantly reducing the calorific value. All samples received the same torrefaction setting, without additional controlled variables in this step.

Properties measurement

After pretreatment, the following properties were determined, including moisture content (MC), weight loss (WL) (separately after chemical extraction and after torrefaction), total weight loss, yield, volatile matter (VM), and gross calorific value. MC, WL, yield, and VM were assessed at the Environmental and Forestry Laboratory, Faculty of Forestry, Hasanuddin University (Makassar), while gross calorific value was measured at the Chemical Service Laboratory of the Center for Poultry and Livestock Assembly and Testing (Bogor). Summary data are reported for each shrub species under each solvent and temperature condition (Table 1–4 and Figure 2–8).

Data calculation and aggregation

Mass-based indicators were computed from recorded sample masses before extraction, after extraction, and after torrefaction. In keeping with standard mass-balance relationships reflected in the dataset structure.

$$\text{WL after chemical extraction}\,(\%) = \frac{m_0 - m_1}{m_0} \text{ x } 100$$

Table 1. Moisture content of shrubs pre- and post-physicochemical treatment.

Shrub Species	Raw/Solvent	Chemical Extraction at 25 °C		Chemical Extraction at 50 °C	
		MC (%)	SD	MC (%)	SD
L. speciosa	Raw	6.81*	0.85	-	-
	Distilled water	6.57	0.16	6.55	0.06
	Ethanol	5.97	0.06	6.07	0.05
	Hexane	5.49	0.07	6.00	0.09
	Ethanol + Hexane	6.16	0.07	6.14	0.05
G. falcatum	Raw	9.29*	0.85	-	-
	Distilled water	6.09	0.06	6.65	0.09
	Ethanol	6.46	0.02	6.10	0.24
	Hexane	6.45	0.05	6.22	0.03
	Ethanol + Hexane	6.34	0.07	6.20	0.09
M. avenis	Raw	9.44*	0.30	-	-
	Distilled water	5.81	0.11	5.76	0.09
	Ethanol	6.11	0.04	5.95	0.04
	Hexane	5.55	0.05	5.75	0.03
	Ethanol + Hexane	6.00	0.07	6.13	0.07
M. sundaicus	Raw	8.70*	0.96	-	-
	Distilled water	6.63	0.11	6.54	0.04
	Ethanol	6.57	0.06	6.64	0.03
	Hexane	5.78	0.04	6.32	0.07
	Ethanol + Hexane	6.60	0.06	6.26	0.07

Note: *moisture content of raw shrubs prior to chemical extraction, MC represents moisture content, and SD denotes standard deviation.

$$\text{WL after torrefaction}\,(\%) = \frac{\mathrm{m_1} - \mathrm{m_2}}{\mathrm{m_1}}\ \text{x}\ 100$$

$$\text{Total WL}\,(\%) = \frac{\mathrm{m_0} - \mathrm{m_2}}{\mathrm{m_0}}\ \text{x}\ 100$$

$$\text{Yield}\,(\%) = 100 - \text{Total WL}$$

where m_0 is the oven-dry mass before extraction, m_1 the mass after extraction/drying, and m_2 the mass after torrefaction. The manuscript presents means with standard deviations for MC and VM, and tabulates WL and yield per condition, enabling direct cross-checks.

Dataset validation

It was noted several scope limitations, including: (i) extraction conditions were restricted to two temperatures and relatively short durations, which may not reflect broader operational envelopes; (ii) although VM was reduced for the most shrub species, values remain relatively high and do not yet meet SNI 8675:2018 quality thresholds for *M. avenis*; (iii) for each species–solvent–temperature combination, the extraction was performed once on a single sample (no technical or biological replicates). This lack of replication limits statistical power, precludes robust uncertainty quantification beyond the reported descriptive values, and warrants caution when generalizing treatment effects; and (iv) experiments were conducted under controlled laboratory settings, so scale-up and field variability were not

Table 2. Weight loss and yield of shrubs following physicochemical treatment.

Shrub Species/Solvent	WL After Chemical Extraction (%)		WL After Torrefaction (%)		Total WL After Treatment (%)		Yield (%)	
	25 °C	50 °C	25 °C	50 °C	25 °C	50 °C	25 °C	50 °C
L. speciosa								
Distilled water	15.90	17.94	1.05	0.45	16.95	18.38	83.05	81.62
Ethanol	17.96	18.56	0.15	0.21	18.11	18.77	81.89	81.23
Hexane	11.58	11.48	0.50	0.90	12.08	12.38	87.92	87.62
Ethanol + Hexane	18.02	16.80	0.33	1.04	18.35	17.85	81.65	82.15
G. falcatum								
Distilled water	13.56	13.62	0.14	0.57	13.70	14.18	86.30	85.82
Ethanol	14.04	14.28	0.15	0.11	14.18	14.39	85.82	85.61
Hexane	11.84	11.93	0.17	0.16	12.00	12.09	88.00	87.91
Ethanol + Hexane	12.91	13.85	0.41	0.50	13.32	14.35	86.68	85.65
M. avenis								
Distilled water	14.40	16.15	0.29	0.25	14.69	16.40	85.31	83.60
Ethanol	15.41	16.73	0.47	0.06	15.88	16.79	84.12	83.21
Hexane	11.04	11.87	0.57	0.11	11.61	11.98	88.39	88.02
Ethanol + Hexane	15.99	15.86	0.38	0.81	16.36	16.66	83.64	83.34
M. sundaicus								
Distilled water	14.29	16.45	0.83	0.42	15.13	16.87	84.87	83.13
Ethanol	15.01	15.59	0.75	0.19	15.76	15.78	84.24	84.22
Hexane	12.32	12.81	0.77	0.14	13.09	12.95	86.91	87.05
Ethanol + Hexane	14.28	17.08	1.17	0.73	15.45	17.81	84.55	82.19

Note: WL represents weight loss.

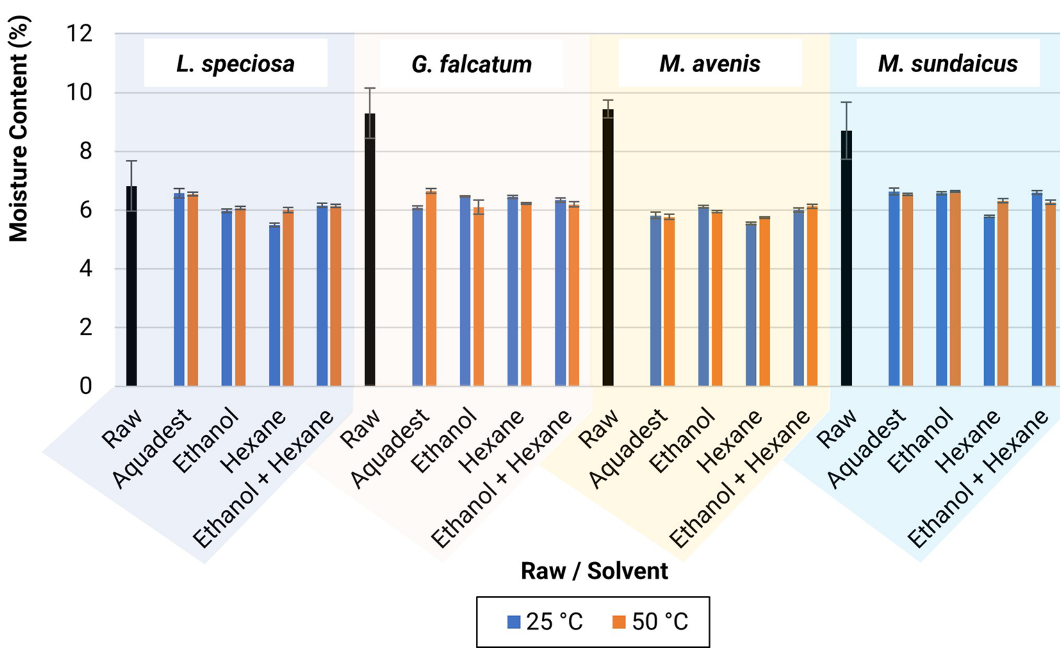

Figure 2. Moisture content of shrubs before (raw) and after physicochemical treatment under various chemical extraction solvents and temperature conditions.

Table 3. Volatile matter content in shrubs following physicochemical treatment.

Shrub Species	Solvent	Chemical Extraction at 25 °C		Chemical Extraction at 50 °C	
		VM (%)	SD	VM (%)	SD
L. speciosa	Distilled water	81.01	0.29	80.42	0.44
	Ethanol	79.06	0.07	79.03	0.63
	Hexane	78.87	0.18	78.84	0.19
	Ethanol + Hexane	79.20	0.14	79.20	0.41
G. falcatum	Distilled water	81.49	0.45	79.34	0.32
	Ethanol	78.29	0.21	78.32	0.11
	Hexane	78.20	0.34	78.08	0.17
	Ethanol + Hexane	78.44	0.21	78.52	0.32
M. avenis	Distilled water	85.61	0.31	83.85	0.25
	Ethanol	83.18	0.19	83.30	0.23
	Hexane	82.07	0.13	82.00	0.19
	Ethanol + Hexane	83.30	0.20	83.52	0.40
M. sundaicus	Distilled water	81.69	0.41	80.21	0.24
	Ethanol	79.59	0.25	79.66	0.09
	Hexane	78.95	0.22	79.10	0.60
	Ethanol + Hexane	79.49	0.15	79.64	0.14

Note: VW represents volatile matter content, SD denotes standard deviation

Table 4. Calorific value of shrubs following physicochemical treatment.

Shrub Species	Solvent	Gross Calorific Value (kcal/kg)	
		Chemical Extraction at 25 °C	Chemical Extraction at 50 °C
L. speciosa	Distilled water	4349	4342
	Ethanol	4363	4298
	Hexane	4366	4356
	Ethanol + Hexane	4386	4373
G. falcatum	Distilled water	4330	4386
	Ethanol	4277	4331
	Hexane	4309	4338
	Ethanol + Hexane	4329	4380
M. avenis	Distilled water	4404	4328
	Ethanol	4372	4366
	Hexane	4416	4397
	Ethanol + Hexane	4420	4337
M. sundaicus	Distilled water	4260	4280
	Ethanol	4207	4233
	Hexane	4300	4220
	Ethanol + Hexane	4262	4269

represented. Additionally, the torrefaction step was fixed at a single set-point (200 °C, 10 min), and solvent removal was verified pragmatically by odor rather than by residual-solvent analytics. These choices simplify the protocol but leave

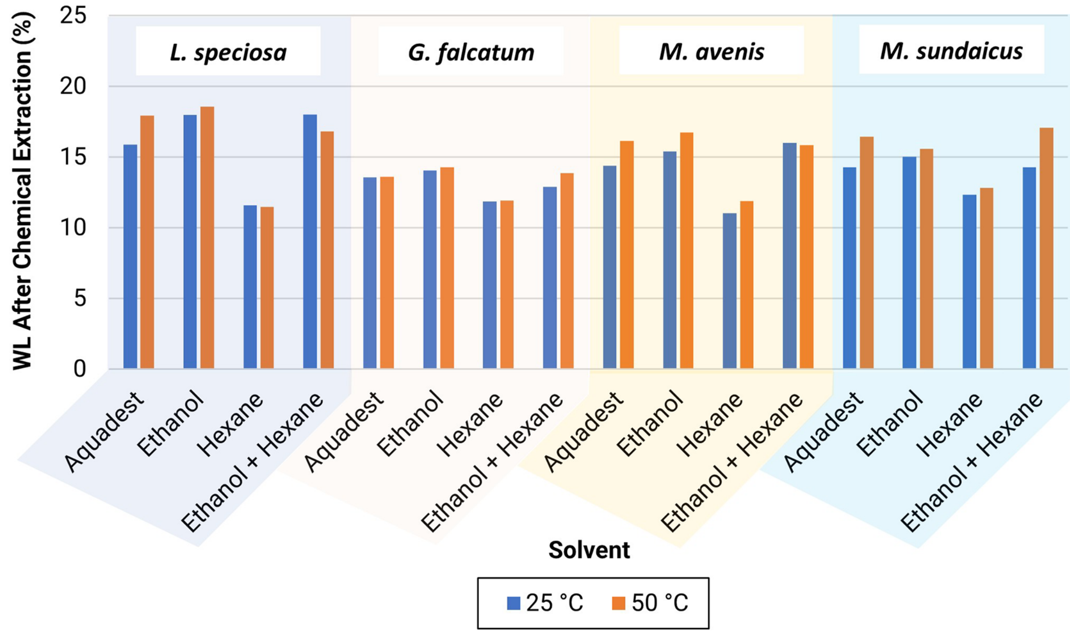

Figure 3. **Weight loss (WL) of shrub after chemical extraction under different solvent and temperature conditions.**

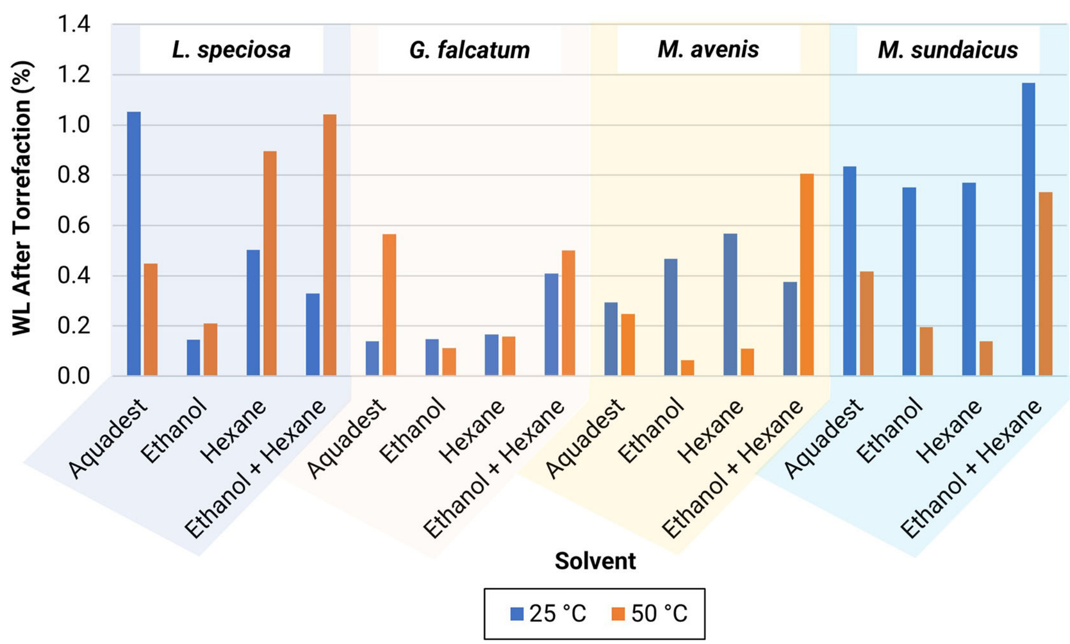

Figure 4. **Weight loss (WL) following the torrefaction of chemically extracted shrubs under different solvents and temperature conditions.**

limited room to quantify instrument-level uncertainty or solvent residues. Users should consider these constraints when extrapolating the dataset to industrial contexts.

For secondary analyses or replication, users should preserve the documented particle size, solid–liquid ratio, solvent sequence and durations (for ethanol + hexane), temperature controls, and the drying/torrefaction set-points to maintain

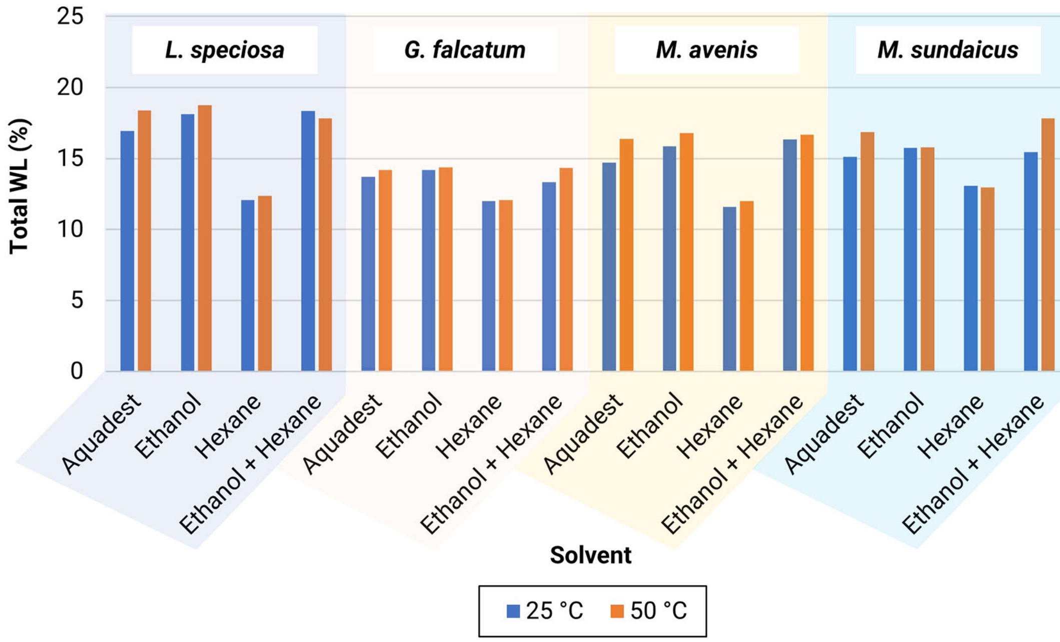

Figure 5. **Total weight loss (WL) of shrubs following the physicochemical treatment.**

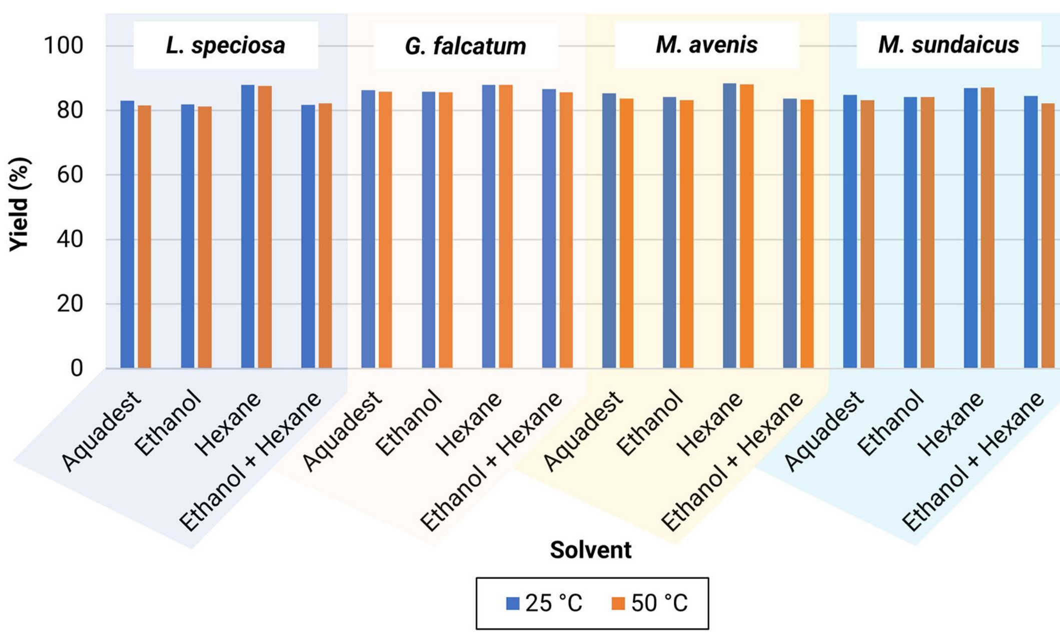

Figure 6. **Total yield of shrubs following the physicochemical treatment.**

comparability with the published tables and figures. Deviations in any of these parameters will directly affect WL, Yield, VM, and calorific value outcomes. The DOI-hosted files provide the necessary inputs to re-calculate all derived metrics and to propagate uncertainty using the reported SDs.

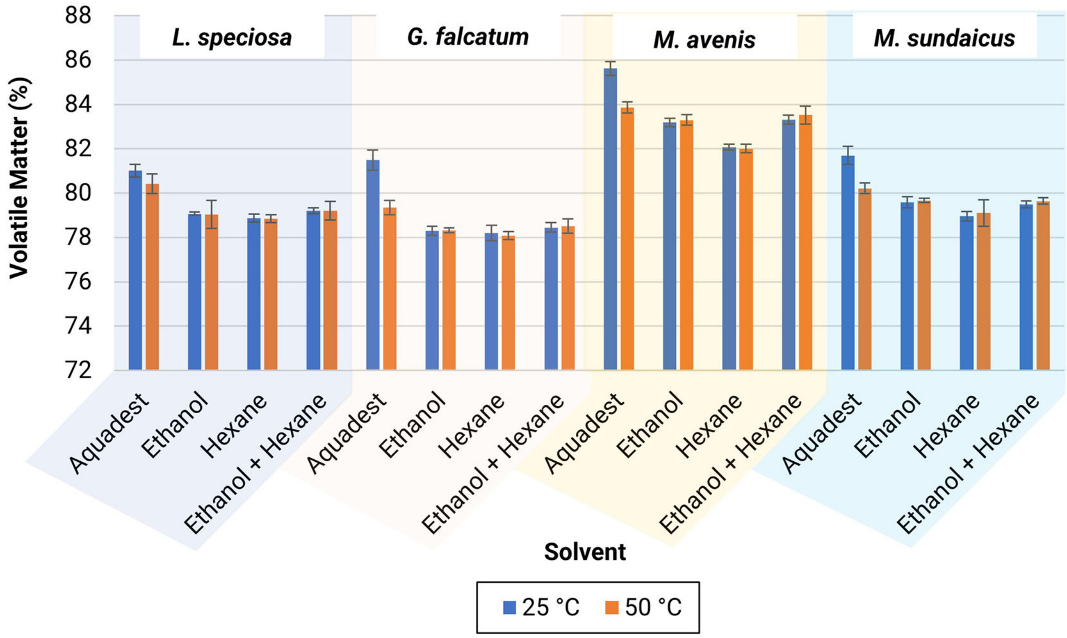

Figure 7. Volatile matter content of shrubs following the physicochemical treatment.

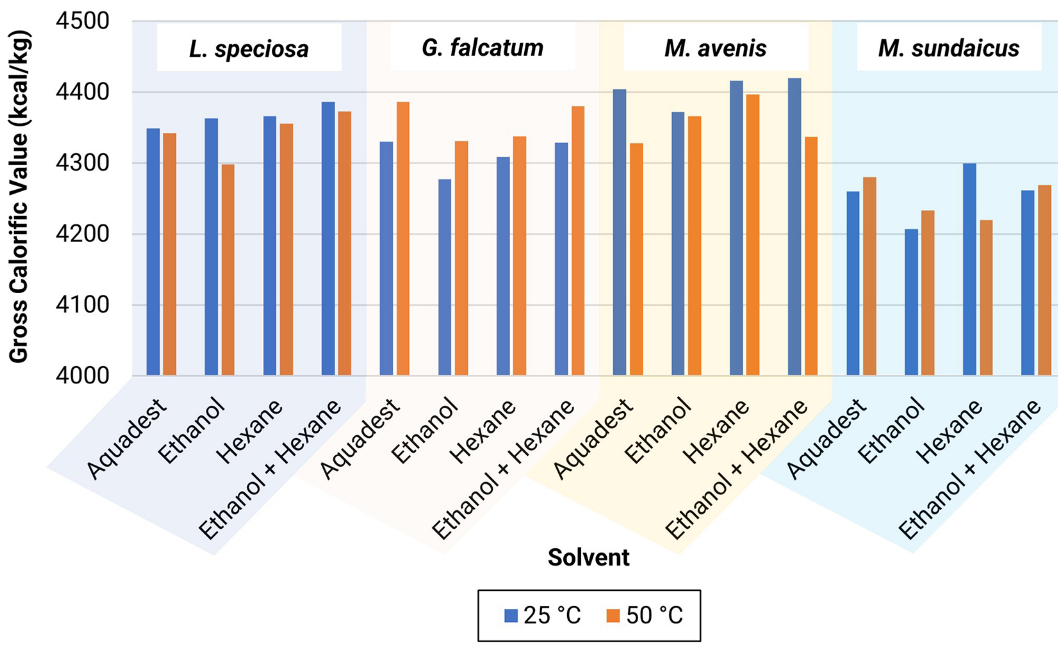

Figure 8. Gross calorific value of shrubs following the physicochemical treatment.

Ethical considerations

This study did not involve human participants, animals, or data sourced from social media. The authors affirm adherence to the publisher's ethical requirements for F1000Research, and, given the absence of human or animal subjects, no institutional review board (IRB) or animal care and use committee (IACUC) approval was required.

Data availability

Mendeley Data: Physicochemical Pretreatment on Lesser-Known Shrub Biomass for Bio-Pellet Production. https://doi.org/10.17632/s9g65ht6hm.4.[7]

This project contains the following underlying data:

- Dataset_DIB.xlsx

Data are available under the terms of the Creative Commons Attribution 4.0 International (CC BY 4.0).

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Current Peer Review Status: ? ?

Version 1

Reviewer Report 15 July 2026

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?

Sarah Augustina
National Research and Innovation Agency (BRIN), Bogor, Indonesia

The data note that was submitted has the potential to make a significant contribution to the field; however, there are substantial deficiencies that must be addressed before it can be proceeded to the next stage. In addition, the english writing (grammar and style) and formatting setting in this data note must be improved to meet the journal's standards.

Introduction:
1. Please provide detailed information on the four lesser-known shrub species used in this study, including their botanical characteristics, availability, and relevance to biopellet production.
2. Please include a review of previous studies on the modification and utilization of shrub biomass for biopellet production, highlighting their key findings and limitations.
3. Please clearly identify the research gap addressed by this study, particularly in relation to previous work on solvent-based extraction. The rationale for combining solvent extraction with torrefaction is not sufficiently explained. The authors should clarify why this integrated approach is necessary and how it is expected to improve biomass properties or pellet performance compared with existing methods.

Method:
1. Why did the authors use different extraction methods, particularly in the ethanol–hexane solvent system?
2. Please provide a justification for the selected ratios, including any supporting references or preliminary studies that guided their selection.

Data set validation:
1. Please explain the underlying mechanism responsible for this phenomenon and discuss the factors that may have contributed to the observed results.
2. Please justify the use of odor analysis as a method for verifying solvent removal. Since odor assessment can be subjective, the authors should discuss its reliability and, if possible, provide supporting measurements or references to validate this approach.
3. Although a separate Results and Discussion section may not be required for a data note article,

the manuscript would benefit from a more comprehensive description of the dataset, including key characteristics, quality-control procedures, limitations, and potential applications.

Is the rationale for creating the dataset(s) clearly described?

Yes

Are the protocols appropriate and is the work technically sound?

Yes

Are sufficient details of methods and materials provided to allow replication by others?

Partly

Are the datasets clearly presented in a useable and accessible format?

Yes

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: Lignocellulose/biomass modification

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.

Reviewer Report 06 July 2026

https://doi.org/10.5256/f1000research.196734.r495628

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?

Wasoh Helmi
University Putra Malaysia, Serdang, Selangor, Malaysia

Title:
Physicochemical Pretreatment on Lesser-Known Shrub Biomass for Bio-Pellet Production

Specific Comment

Keywords: How did the authors determine "yield" as a keyword? The term appears to be too general and may not effectively improve the discoverability of the article. Please consider replacing it with a more specific and descriptive keyword.

Table 2 & Table 4: Why were the measurements not performed in replicate? The authors have provided a justification; however, it is not sufficiently convincing. Please provide a more detailed rationale for the lack of replication and discuss its potential impact on the reliability of the reported results.

Table 1: The results for the raw material subjected to chemical extraction at 50 °C are missing. Please clarify whether these experiments were performed. If so, please include the corresponding data or provide a justification for their omission.

Table 2: The standard deviations (SDs) for weight loss and yield are absent. The current justification does not adequately explain why these values were omitted. Please provide a more robust explanation and clarify whether these parameters were determined from replicated measurements.

Figure 2: The moisture content of the raw material exhibits a substantially larger error bar than the other samples, indicating greater variability. Please explain the possible cause of this observation and discuss whether it may have affected the reliability of the measurements.

Figure 5: The total weight loss appears to be very similar across all treatments. Please explain the reason for this observation and discuss whether it is expected based on the treatment conditions.

General Comment

Please do not use the em dash (—) in equations or mathematical expressions throughout the manuscript. Instead, use the standard minus sign (-), as appropriate.

Please provide a scientific justification for the use of the sequential ethanol–hexane extraction. The rationale should be supported by relevant literature and should explain the choice of these solvents based on their polarity, solubility characteristics, and other pertinent physicochemical properties, as well as their influence on the extraction of the target compounds.

The term **"volatile content"** is used in the flowchart, whereas **"volatile matter"** is used throughout the manuscript. Please ensure consistent terminology throughout the manuscript. If referring to proximate analysis, **"volatile matter"** is the more commonly accepted term.

The term **"Aquadest"** is used in the graph, whereas **"distilled water"** is used in the table. Please use consistent terminology throughout the manuscript. Furthermore, the term **"Aquadest"** does not appear elsewhere in the text. As **"distilled water"** is the standard term used in international scientific publications, I recommend using this term consistently throughout the manuscript.

The solid-to-liquid ratio is reported as **1:7 (wt/wt)**. Please clarify how this ratio was determined. If the solvent was measured by volume rather than by mass, **wt/vol (w/v)** may be a more appropriate notation. Please ensure that the reported units accurately reflect the experimental procedure.

The manuscript states that a sequential solvent treatment using ethanol + hexane was The manuscript states that a sequential solvent treatment using **ethanol + hexane** was performed. Please clarify how the transition between ethanol and hexane was carried out. Specifically, describe whether the ethanol was removed before the hexane treatment, how the sample was handled between the two extraction steps, and whether any drying or intermediate washing procedures were performed.

The manuscript states that *"To limit evaporation, especially at 50 °C, flasks were covered with aluminum foil."* Please clarify whether covering the flasks with aluminum foil was sufficient to minimize solvent evaporation throughout the extraction period. Provide a more detailed description of the measures taken to prevent solvent loss and discuss whether solvent evaporation could have affected the solid-to-liquid ratio, extraction efficiency, or reproducibility of the results.

Is the rationale for creating the dataset(s) clearly described?

Yes

Are the protocols appropriate and is the work technically sound?

Yes

Are sufficient details of methods and materials provided to allow replication by others?

Yes

Are the datasets clearly presented in a useable and accessible format?

Yes

Competing Interests: No competing interests were disclosed.

Reviewer Expertise: Food Biotechnology, Fermentation, Bioremediation

I confirm that I have read this submission and believe that I have an appropriate level of expertise to confirm that it is of an acceptable scientific standard, however I have significant reservations, as outlined above.

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