



# Article Energy Potential of Oil Palm Empty Fruit Bunch (EFB) Fiber from Subsequent Cultivation of Volvariella volvacea (Bull.) Singer

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**Copyright:** © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). Abstract: EFB and EFB-based mushroom compost (SMC) from Volvariella volvacea cultivation is a promising energy feedstock because it has adequate nutrient quality. The biochemical methane potential (BMP) and calorific value (CV) of this biomass are investigated. Other analyses such as proximate, compositional, and final analysis; thermogravimetric analysis (TGA); and Fourier transform infrared spectroscopy (FTIR) are also performed. The biomass samples consist of two types of EFB, namely fibers (F) and pellets (P) and SMC from the subsequent cultivation of Volvariella volvacea, with samples FS and PS from the first cultivation and FS2 and PS2 from the second cultivation. P produces the highest biological efficiency (BE) of 28% compared to 9.83% for F. Subsequent cultivation with FS and PS then produces only 2.9 and 6.83% of BE. A higher amount of methane is measured in samples P and PS2, while better biodegradability is observed in PS2 and FS2, suggesting that subsequent cultivation is a good pretreatment of the substrate for anaerobic digestion (AD). CV is highest in F (20.57 MJ/kg), followed by P (19.06 MJ/kg), which is comparable to commercial wood pellet. Samples F, FS, and FS2 have higher ash content, which is due to higher mineral content. The cellulose composition is reduced to almost 50% during cultivation due to fungal metabolism, which is also evidenced by FTIR analysis. TGA analysis revealed that EFB-based SMC exhibits higher weight loss during combustion compared to EFB, which reduces its thermal properties. SMC of EFB is a high potential biomethane feedstock, but not recommended as a fuel pellet.

Keywords: empty fruit bunch; spent mushroom compost; bioenergy; biomass pellet; biomethane

## 1. Introduction

Biomass is a promising renewable energy source as it is abundant and derived from various sources such as agricultural waste, wood and grass [1]. The global biomass resources contributed to more than 467 EJ of energy conversion potential per year [2]. Palm oil is the most widely grown edible oil in the world, with a market share of nearly 30% [3]. It is widely used for cooking, frying, and baking as it has a stable cooking point. The industry contributes to a high percentage of solid waste from the plantation and production process. The most significant percentage term is the empty fruit bunch (EFB) which is typically discarded after harvest. It is estimated that about 20 million tons of EFB are generated as waste from plantations in Malaysia every year [4]. EFB can be converted into cellulosic ethanol, a form of biofuel that does not compete with land use for food production [5].

The use of EFB for *Volvariella volvacea* cultivation is gaining attention in Malaysia and Indonesia due to its abundance as a byproduct from the local oil palm industry [6]. Another problem encountered in mushroom cultivation is the spent mushroom compost (SMC) generated at the end of cultivation. About 5–10 kg of SMC is generated from 1 kg of mushroom harvested [7]. In Malaysia, the standard practice for SMC management is either dumped in the soil or burnt [8,9]. Only 28% of the SMC is recycled into agriculture activities as organic fertilizer or soil conditioner [9]. As for the SMC from *Volvariella volvacea* cultivation, there has been no report on recycling in Malaysia. While reports of SMC utilization from other species of mushroom is also considered scarce in Malaysia. The use of SMC as energy feedstock has attracted many researchers and industry players as it is abundant and available at zero cost. At the same time, clean energy (SDG 7) can potentially be generated from mushroom industry waste [10]. Moreover, this move supports SDG 12 for responsible production and consumption in the mushroom industry.

Lignocellulosic biomass is an economically feasible source of heat and power mainly by combustion. The use of SMC as a fuel pellet has many environmental and economic advantages as a cheap, clean, and renewable energy source. In a report by Ryu et al. (2008) [11], SMC from button mushroom is suitable for pellet production as a mixture with coal waste. It exhibits property for use in gasifiers and power plants. This study investigates the potential use of SMC from subsequent Volvariella volvacea cultivation as biomass fuel. Characterization of biomass samples in terms of calorific value, thermal properties, compositional analysis, and chemical properties is carried out to determine the various elements of the biomass for their suitability as biomass fuel. Moreover, biogas production using SMC as a substrate is a good alternative for recycling and waste reduction. Mamimin et al. (2021) [12] reported that the SMC of EFB from V. volvacea cultivation gave a better biogas yield than EFB. This is expected since SMC is a simplified substrate due to naturally occurring degradation by fungi [13]. Furthermore, the type of lignocellulosic biomass influences the degradation rate. A similar trend is also noted by Purnomo et al. (2018) [14], who indicated that semi-wet fermentation using EFB based SMC resulted in optimal biogas production.

This article aimed to demonstrate the possibility of zero waste cultivation of *Volvariella volvacea* using an EFB pellet. Subsequent cultivation of *V. volvacea* using EFB and EFB based SMC will act as the pre-treatment for the substrate of anaerobic digestion. Biochemical methane potential analysis is used as a tool for this investigation as it provided insightful analysis on the potential yield of methane and biodegradability of the biomass [15]. In addition, the characterization of the biomass samples is to prove its potential as energy feedstock and provide an alternative for dumping issues. In the end, the integration between the palm oil industry and mushroom industry can be a benchmark for zero waste management and circular economy [16]. The whole experimental work is shown in Figure 1.



Figure 1. Evaluation of biomass samples for energy potential using various analyses.

### 2. Materials and Methods

## 2.1. Chemicals and Reagents

All chemicals, solvents, and reagents used in this study are of analytical grades provided from Sigma-Aldrich. The chemicals used in this study are listed as below:

Dipotassium phosphate ( $K_2HPO_4 \cdot 3H_2O$ ), sodium phosphate monobasic dihydrate ( $NaH_2PO_4 \cdot 2H_2O$ ), ammonium chloride ( $NH_4Cl$ ), calcium chloride ( $CaCl_2 \cdot 2H_2O$ ), magnesium sulphate ( $MgSO_4 \cdot 7H_2O$ ), iron (III) chloride ( $FeCl_3 \cdot 4H_2O$ ), sodium selenite pentahydrate ( $Na_2SeO_3 \cdot 5H_2O$ ), cobalt (II) chloride hexahydrate ( $CoCl_2 \cdot 6H_2O$ ), nickel(II) chloride hexahydrate ( $NiCl_2 \cdot 6H_2O$ ), mangan chloride ( $MnCl_2 \cdot 4H_2O$ ), ethylenediaminetetraacetic acid (EDTA), cuprum chloride ( $CuCl_2 \cdot 2H_2O$ ), acid hydrochloride ( $HCl_36\%$ ), zinc chloride ( $ZnCl_2$ ) resazurin, boric acid ( $HBO_3$ ), yeast extract, ammonium molybdate ( $(NH_4)_6Mo_7O_2 \cdot 4H_2O$ ), sulfuric acid ( $H_2SO_4$ ).

### 2.2. Biomass Samples

Samples of EFB fibers labeled F were prepared at the Malaysian Palm Oil Board laboratory (MPOB), Bangi, Selangor facilities. The source of EFB pellet labeled P is from USSB Sdn. Bhd., a local supplier. The SMC of P and F, designated as FS and PS, respectively, are produced from the cultivation of *Volvariella volvacea* in a mushroom house located in Negeri Sembilan. These SMCs then were used for the second cultivation of the similar fungus and the second SMCs were collected as samples FS2 and PS2, respectively. The samples' physical properties, including moisture and bulk density, are measured according to a standard procedure [4]. Digital calipers were used to measure the diameter and length of the samples. The biomass was ground to a smaller size between 0.1–0.2 mm depending on the analysis requirement.

#### 2.3. Volvariella volvacea Cultivation

Indoor cultivation was carried out using F and P samples as a substrate. The dry substrate was weighed and recorded before adding rice bran, organic fertilizer, and CaCO<sub>3</sub> at 8%, 7%, and 5% (w/w) concentrations, respectively. The compost mixture was thoroughly mixed with water to achieve suitable moisture content between 65% and 70%. The spawning process was initiated by adding the loosened spawning block with approximately 100 g to around 2 kg of compost in the plastic bag. These plastic bags then were tightly sealed and arranged in the closed pile to prevent air access, ready for 10-day incubation. After 10 days, the compost bags were opened and placed in the rack and ready for the fruiting phase. Humidity in the mushroom house was maintained at 70-80% by water misting and watering the floor of the mushroom house. The fruiting bodies were plucked at the egg stage, weighed, and recorded. For subsequent cultivation, spent mushroom compost (FS and PS) from the first cultivation are collected. Then, these substrates were steam sterilized for 5–6 h at 80 °C. They are then allowed to cool before mixing with the same mixture at a similar ratio to the first cultivation. The same steps as for the first cultivation were then repeated. All cultivation samples were done in triplicate. The mushroom was harvested, cleaned, counted and weighed every day. Biological efficiency (BE) was measured by dividing the weight of fresh yield over a substrate in percentages.

## 2.4. Solid Analysis

All solid analysis follow American Public Health Association, American Water Works Association, & Water Environment Federation (1999). Standard methods of total solids (TS) and volatile solids (VS) were carried out following the Standard Method 2540 B and 2540 G [17]. TS was determined by weighing the samples in an evaporating dish and placing them in an incubator set at 105 °C for 24 h. The samples were then burned in a muffle furnace at a temperature of 550 °C for 1 h to determine volatile organic components, with the resulting post-combustion residue or ash content as the inorganic content. Analyses of the sample were done in triplicate.

#### 2.5. Fiber Morphology and Composition Analysis

All fibers were observed in a bright field using a USB digital microscope with a built-in camera (China). A plug-in digital viewer software was used for computer observation. The microscopic appearance of the biomass samples was observed, while the diameter size of the samples were measured using digital calipers. The biomass samples were subjected to compositional analysis to quantify the concentration of cellulose, hemicellulose, and lignin. The analytical procedures followed the modified Chesson-Datta method based on acid digestion and gravimetry method [18]. Analyses of the samples were done in triplicate.

#### 2.6. Calorific Value

Calorific value (CV) is an important parameter for determining the energy content of the materials. CV was defined as the absolute value of the specific heat (enthalpy) of combustion of materials; in joules. It is calculated per unit mass of the materials or biofuel when burned in oxygen at constant pressure at 0.1 Mpa under such conditions that all the water of the reaction products remains as vapors [19]. CV is also reported as HHV or high heating value, which is calculated by unit conversion. CV was determined using a bomb calorimeter, IKA WORKS Model C5000. For combustion in this apparatus, 1 g of samples was placed in the crucibles.

#### 2.7. Ultimate Analysis

The ultimate analysis was performed using the Model 2400 CHNS elemental analyzer (Perkin Elmer, USA). Biomass samples of 1–2 mg were used and placed in the auto-sampler system of the instrument. Combustion initiated in the furnace at a temperature of >1800 °C. After combustion in an oxygen-rich environment, the producing gases were released and carried by a helium flow past a copper-filled layer through a gas chromatography (GC) column. The combustion gases are separated and detected by a thermal conductivity detector (https://resources.perkinelmer.com/lab-solutions/resources/docs/BRO\_2400\_SeriesII\_CHNSO\_Elemental\_Analysis.pdf, accessed on 1 August 2021). The software calculates the resultant gases based on the initial weight and shows C, H, N, and S in percentage. The oxygen content was calculated from the difference between the cumulative percentage composition of C, H, N, and S and 100%. Analyses of the samples were done in triplicate.

#### 2.8. Biomethane Potential Assay

Biochemical methane potential (BMP) was used to determine substrate conversion to methane via the AD process. The inoculum used is an anaerobic sludge taken from an active anaerobic digester in Serting Hilir Palm Oil Mill, Negeri Sembilan. While the substrate in this experiment is the biomass samples. Following Zhang et al. (2014) [20] procedure, other components in this test are phosphate buffer, macronutrients, and micronutrients. Before the experiment, the inoculum was degassed for five days. The mixture was filled in 120.0 mL of serum bottle with a working volume of 80.0 mL. All BMP tests for blank and samples were done in triplicate. The sample group consists of inoculum, biomass and media components, while the blank groups of assay performed without the biomass. Inoculum and substrate were mixed based on the volatile solid content at the ratio 2:1 [21,22]. A pressure meter measured the daily production of biogas based on the pressure changes in the headspace of serum bottles. The BMP tests were stopped when the pressure changes were stable, indicating that the substrate was depleted. The gas produced is collected and analyzed in a gas chromatograph. The calculation for BMP analysis is

$$BMP mL CH_4/g VS = \frac{\text{net CH}_4 \text{ of sample } (CH_{4sample} - CH_{4blank})}{g VS \text{ of sample}}$$
(1)

### 2.9. Theoretical BMP and Biodegradability

For theoretical BMP (TBMP) and biodegradability, the calculation follows the Boyles equation, modified from the Buswell method [23]. The measurement was based upon the assumptions that the experimental condition is optimum in which ideal microbial condition; total substrate digestion; complete mixing and constant temperature; substrate composition limited to only C, H, O, N, and S; and output in the form of CH, CO, and NH [23]. The formula is shown below:

Theoretical BMP mL CH<sub>4</sub>/g VS (TBMP) = 
$$\frac{22400 (n/2 + a/8 - b/4 - ac/8)}{12n + a + 16b + 14c}$$
 (2)

In which C = n, H = a, O = b, N = c.

Biodegradability (BD) = 
$$\frac{BMP \times 100\%}{TBMP}$$
 (3)

## 2.10. Fourier Transform Infrared (FTIR) Spectroscopy Analysis

FTIR analysis of the samples was done using Spectrum 100 Spectrometer Perkin Elmer, to determine changes in the component. FTIR spectra of each sample were recorded in the range of 4000–600 cm<sup>-1</sup> at 4 cm<sup>-1</sup> resolution and averaged over 32 scans. Before analysis, the samples were ground into powder and kept at a low temperature of 22 °C. This analysis uses a simple and fast qualitative technique using standard IR spectra. It helps identify the functional group(s) of the samples' components, which can provide insightful observation of compositional changes or degradation [24].

## 2.11. Thermogravimetry Analysis (TGA)

The thermogravimetry technique using Perkin Elmer (USA) equipment is applied to determine the thermal properties of the biomass sample [25]. Samples were ground in powder form and conditioned at indoor laboratory temperature  $24 \pm 2$  °C for 24 h. The samples were scanned in the condition that the heating rate is set at 10 °C/min in a nitrogen atmosphere with a gas flow of 20 mL/min from 30 °C to 600 °C [26].

#### 2.12. Statistical Analysis

Data for mushroom yield and BMP were analyzed using IBM SPSS Statistic 26. Means comparison with LSD and Tukey's significant test at a level of  $\alpha = 0.001$  was performed to determine the significant difference between the treatments.

#### 3. Results and Discussion

#### 3.1. Yield Performance, Physical Properties, and Compositional Analysis of the Biomass Sample

The yield performance of *V. volvacea* cultivation was summarized in Table 1. Cultivation took place in the mushroom house for 30 days. It was observed that the first harvest of fruiting bodies is as early as day 14 for spent compost (FS), followed by samples F, PS, and P, which appear on days 16, 17, and 19, respectively. It is believed that the first cultivation improved the biodegradability of the substrate and accelerated the development of fruiting bodies in the spent substrate compost. The values with a different superscript letter in a column are significantly different at (*p*-value < 0.001). P gave the highest yield of  $567 \pm 58.4$ with biological efficiency (BE) of 28%. This is a significant difference compared to F samples, with an increase in yield of about 188.5%. The highest number of fruiting bodies was observed in the P samples, 32 or three times more than the others. This is followed by F, PS, and FS. It is also observed that subsequent cultivation with PS gave a better yield and BE than FS. Overall, the total yield and B.E for subsequent cultivation using pellet (P and PS) are 702.46 and 34.76%, while fiber samples (F and FS) are 254.34 g and 12.73%, respectively. EFB pellet recorded better B.E of *Volvariella volvacea* compared to paddy straw (25.3%), whole bunch EFB (7.06%) and cotton waste (16.71%) [27–29].

Samples	Yield (g)	BE (%)	DFFH	No. FB	HD
F	$196.67 \pm 7.81$ b	9.83	$16\pm1.2$	$10 \pm 1.2$	$5\pm1.3$
Р	$567.33\pm58.4$ c	28.00	$19\pm0.5$	$32\pm0.7$	$5\pm1.5$
FS	$57.67\pm20.1~\mathrm{a}$	2.90	$14\pm0.0$	$6\pm2.0$	$3\pm0.0$
PS	$135.33\pm13~{}_{bc}$	6.76	17 + 1.5	$10\pm2.0$	$4\pm0.0$

 Table 1. Profile of mushroom yield using different samples.

HD = harvesting day; BE = biological efficiency; DFFH = day for first harvest; FB = fruiting bodies. Mean with the same subscript in the column are not significantly different at p < 0.001.

Table 2 shows the physical properties of the biomass samples. The P sample had a higher density compared to the F sample, which was expected since the fiber samples are still in aggregate form, which reduces the compactness. The F sample is slightly longer due to the nature of the biomass produced by the screw pressing and primary grinding process. The P sample is an EFB pellet that underwent a further process compared to the F sample, where it was cut smaller to obtain a denser structure. The length distribution for the F, FS, and FS2 samples is more than 50 mm, while the P, PS, and PS2 samples are in the range of 3–18 mm. The length of this biomass was determined by the production method. Cellulose, hemicellulose and lignin are the main components of natural fibers. The distribution of these compounds in the fibers varies depending on the species, growth conditions, climatic effect, age of the plants, and the test method used [26,30]. In the samples, changes were observed in the EFB fibers after cultivation, which were attributed to the metabolism of the fungi. As expected, more cellulose was detected in the P and F samples, with both samples having about 45% cellulose. The concentration is within the normal range of previous researchers who reported a range of 13.75–59.70% [31–33]. On the other hand, for FS and PS, the samples contained 36.42% and 34.65% of cellulose, respectively. However, these results are relatively high compared to Triyono et al. (2019) [28], who reported that the spent EFB fiber of V. volvacea contains an average  $23.51 \pm 2.32\%$  cellulose, but comparable to a study by Apetorgbor et al. (2015) [34] who found 40.6% of the similar sample. About 50% of cellulose from the initial concentration remains in the samples after subsequent cultivation of V. volvacea. The percentage of hemicellulose has remained fluctuating in all the samples. In contrast, the lignin content is relatively high even after subsequent V. volvacea cultivation. This is to be expected as V. volvacea lacks lignin-degrading enzymes [35]. Figure 2 presents the morphology of the samples under  $200 \times$  magnification. The pellets used in this study were originally in intact form but were crushed for mixing in compost for the cultivation. The diameter of the intact form of the pellet was 8.85 mm. For FS2 (0.16–0.28 mm) and PS2 (0.21–0.23 mm), the fiber diameter was slightly thinner than P (0.30–0.42 mm) and F (0.33–0.71 mm). For the PS and FS samples, the differences in diameter were minimal compared to the original samples (P and F). This study shows that the subsequent cultivation decreased the diameter of the samples.

Sample	Moisture (%)	Length (mm)	Diameter (mm)	Bulk Density (kg m <sup>-3</sup> )	Cellulose (%)	Hemicellulose (%)	Lignin (%)
F	4.21	>50	0.33-0.71	0.0948	$45.22 \pm 1.44$	$18.03\pm2.12$	$35.9\pm5.24$
FS	13.5	>50	0.19-0.44	ND	$36.42 \pm 2.08$	$22.31 \pm 1.85$	$36.38 \pm 4.31$
FS2	15.3	>50	0.16-0.28	ND	$23.41 \pm 3.78$	$24.94 \pm 1.54$	$33.51 \pm 1.75$
Р	8.73	3–18	Loose = 0.30–0.42 Intact = 8.85	0.615	$45.37\pm2.96$	$16.87\pm3.24$	$31.33\pm2.61$
PS	50.1	3–18	0.23-0.29	ND	$34.651\pm3.35$	$10.67 \pm 4.5$	$42.84 \pm 2.62$
PS2	62.5	3–18	0.21-0.23	ND	$27.3\pm0.38$	$13.02\pm4.3$	$35.29\pm0.02$

 Table 2. Physical properties and compositional analysis of the biomass sample.

ND = Not determined.



Figure 2. Morphology of the different biomass samples under a digital microscope (100× magnification).

## 3.2. Proximate Analysis and Calorific Value

Table 3 shows the proximate and ultimate analysis of the obtained biomass samples. CV was highest for F (20.57 MJ/kg), followed by the P (19.06 MJ/kg). Both data are comparable to the standard for commercial wood pellets (>16) [4]. In contrast, the SMC samples recorded lower CV mostly due to the higher moisture content, which also affected other parameters. However, these data were similar to the previous report of other agriculture residues such as spent compost lark (16.62 MJ/kg), rice husk (16.8 MJ/kg), and sweet sorghum bagasse (18.57 MJ/kg) [36–38]. Moisture content is higher in the spent compost samples due to the nature of the cultivation process that required high water availability in the substrate in the range of 60–70%. Although the samples were left to dry prior to analysis, entrapped moisture was still present, especially in the SMC pellet samples (PS and PS2). High ash content was observed in both EFB fiber (11.92%) and pellet samples (10.8%) which was also higher than the previous study by Nasrin et al. (2017) [4], who reported 7.55%. However, the data is comparable to Sumardi et al. (2020) [39] that recorded 10.78% of ash content. This is probably caused by impurities in handling the raw material at the source and the transport chain. Dirt, sand, and soil could be the contaminant that contributes to this increment [4,40]. The spent compost was found to have higher ash content compared to the original substrate. This was expected because other compounds such as calcium carbonate, rice bran, and organic fertilizer were added to the mushroom compost, while the mushroom compost only partially degraded during the cultivation process. Moreover, the spent EFB is also temporarily stored in the container in an open space before processing, which increases the probability of contamination. Higher ash content in the samples may contribute to the lower CV as ash is a non-combustible matter which will not contribute to heat release during the combustion process [41,42]. Utilizing the biomass with high ash content can contribute to fouling and slagging [43]. For P and F samples, comparable data to Chandrasekaran et al. (2012) [44] were obtained for ultimate analysis. While data for EFB based SMC in this study show some differences compared to similar samples by Mamimin et al. (2021) [12]. The differences were expected due to the sample's condition and cultivation method. C and N show a downtrend in all samples after the cultivation process except for FS, which yields higher C than F samples. The transformation of the substrate into fruiting bodies likely contributes to this downtrend. Finney et al. [45] reported that commercial pellet produces higher efficiency in generating heat and power during combustion than the pellet plus SMC, although the latter produce self-sustaining energy. The higher moisture content of SMC is also addressed in various studies. A common strategy to overcome the issue is by mixing it with other materials such as coal tailings, cardboard, and sawdust to reduce it and eliminate carbon loss, which eventually increases the calorific value [46].

Samples/		Ultima	te Analysis (%)				Proximate	Analysis (%)		HHV
Reference	С	Н	Ν	$O^1$	S	Ash	FC	VM	МС	(MJ/kg)
F	$39.26 \pm 1.2$	$6.15\pm0.11$	$1.25\pm0.01$	53.34	$0.016\pm0.0$	$11.92 \pm 1.2$	$22.98 \pm 2.41$	$65.10 \pm 1.11$	$3.80\pm0.5$	20.57
FS	$42.04\pm0.56$	$6.09\pm0.09$	$0.72\pm0.02$	51.15	$0.00\pm0.0$	$19.11 \pm 2.11$	$25.04 \pm 2.34$	$55.85\pm\overline{1.44}$	$13.50\pm0.45$	16.77
FS2	$35.19\pm0.87$	$4.68\pm0.27$	$1.25\pm0.03$	58.88	$0.23\pm0.0$	$17.84 \pm 1.75$	$25.88 \pm 3.11$	$56.28 \pm 1.98$	$15.30\pm2.11$	15.06
Р	$42.59 \pm 1.31$	$5.40\pm0.02$	$1.01\pm0.08$	51.00	$0.055\pm0.0$	$10.80\pm0.97$	$21.30 \pm 2.77$	$67.90 \pm 1.45$	$10.10\pm0.76$	19.06
PS	$41.65 \pm 1.21$	$5.11\pm0.22$	$0.6 \pm 0.01$	52.64	$0.11\pm0.01$	$8.02\pm0.87$	$46.49 \pm 2.55$	$45.49\pm0.77$	$50.10\pm2.51$	15.53
PS2	$35.00 \pm 0.77$	$3.95\pm0.09$	$0.99\pm0.03$	60.05	$0.13\pm0.0$	$9.36\pm0.71$	$54.64 \pm 3.77$	$36.00 \pm 1.98$	$62.50\pm3.11$	12.6
EFB pellet [44]	42.99	6.19	0.64	50.11	0.08	-	-	-	-	-
EFB based SMC [12]	51.00	6.40	0.65	40.20	0.00	-	-	-	-	-

Table 3. Proximate analysis and elemental analysis of the EFB and SMC samples.

C = carbon; H = hydrogen; N = nitrogen; O<sup>1</sup> = oxygen, calculated by difference; S = sulphur; FC = fixed carbon; VM = volatile matters; MC = moisture content; HHV = High heating value.

#### 3.3. TGA Analysis

In general, thermal degradation occurs in three steps: (i) moisture reduction; (ii) oxidative pyrolytic decomposition of cellulose and hemicellulose; and (iii) oxidative pyrolytic decomposition of lignin and char [47]. Figures 3 and 4 depict the TGA curves of fiber-based and pellet-based samples, respectively. For the fiber-based samples (F, FS, and FS2), the curves of TG produce expected trends of EFB samples according to previous research [24,48–50]. It starts with moisture evaporation which contributes to a small weight loss of about 8.37% at approximately 120 °C. This is followed by the thermal decomposition of hemicellulose before prolonged to cellulose compounds that produced volatile products at 250–340 °C and 340–430 °C, respectively. The highest in weight loss was observed in FS2, followed by FS and F. This result could be due to the different initial concentrations of cellulose and hemicellulose in each sample. Abdullah et al. (2011) [24] previously reported a similar trend of TG curves of washed and unwashed EFB. The washed EFB sample has a higher weight loss than the unwashed sample. In this study, the cultivation process partially reduces the concentration of cellulose and hemicellulose compounds observed in FS and FS2 samples. The F samples contain a higher concentration of the respective compound. The final weight of 27.39% was determined in F samples much higher compared to only 7.61 and 5.91 in FS2 and FS samples, respectively. The subsequent Volvariella volvacea cultivation might contribute to the trend that appeared in the TG curves of fiber. The same trend was observed for thermal degradation in the pellet samples. Moisture evaporation, cellulose, and hemicellulose degradation and lignin decomposition followed a similar pattern. Samples PS2 recorded a final weight of 6.00%, while PS and P samples weighed 8.99% and 19.77%, respectively. The higher weight loss in the spent compost samples of pellet and fiber is due to the lower cellulose and hemicellulose composition in the latter samples compared to the pellet and fiber of EFB. It is reported that the difference in lignin decomposition in treated and untreated EFB contributed to different final weights of the samples [48]. The findings suggest that fungal metabolism during cultivation contributes to the final weight of the samples during thermal degradation of the biomass samples. In relation to the objective of the work, the results show that the EFB samples (F and P) have better biomass fuel quality based on thermal degradation than EFB based SMC.

In general, various organic wastes can be opted for methane production, such as animal manure, municipal solid waste, agricultural residues, and industrial waste [51]. This study focused on agricultural residues, specifically the spent mushroom compost. A summary of the BMP analysis of the samples was provided in Table 4. The biomethane production of the biomass samples was monitored for 15 days, and the BMP and TBMP yields in the samples varied significantly. The values with different superscript letters in a column are significantly different (p < 0.05). As shown in Figure 5, the cumulative biogas production recorded the highest accumulation in sample P, followed by F and PS2. Samples P and F produce biogas earlier than the other samples. It was observed that the spent samples of first crop produced lower methane yield. Lower BMPs were also reported for fiber samples. The spent fiber samples (FS and FS2) produced only

17.2 and 19.75 (mLCH $_4$ /gVS), respectively. The difference in methane yield between the samples is related to the nutrient composition and the treatment applied. The best results in methane production were obtained for samples P and PS2, 41 and 47.6 (mLCH<sub>4</sub>/gVS). The data from P contrasts with the expected methane from TBMP, which predicted as much as 169.68 (mLCH<sub>4</sub>/gVS) could be produced. However, probably due to the poor biodegradability of these lignocellulosic compounds and their recalcitrant structure, the actual yield of BMP is lower [52]. However, for PS2 samples, TBMP and BMP are about 97% similar, which is expected as the' biodegradability of the samples increases. This finding is supported by Cordoba et al. (2016) [53], who reported that spent sawdust from *Gymnopilus pampeanus* cultivation increased methane yield by 62% compared to sawdust. Mamimin et al. (2021) [12] also found that EFB based SMC from Volvariella volvacea cultivation is a good substrate for the production of methane in solid-state anaerobic digestion with a maximum yield of  $281.1 \pm 7.2$  (mLCH<sub>4</sub>/gVS). Moreover, the spent compost further increases the yield to 405 (mLCH<sub>4</sub>/gVS) when used as a co-substrate with 5% (v/w) palm oil mill effluent. During the cultivation process, biomass hydrolysis increases, and subsequent cultivation further improves the process, as shown by the yield and percentage of methane [52]. This was observed in the second cultivation samples (FS2 and PS2), which showed higher biodegradability, indicating that the second cultivation is a good substrate pre-treatment for better methane yield. Overall, the methane yield recorded from this study is quite low as it is a preliminary study. Nevertheless, it shows a promising result for EFB based SMC from subsequent cultivation. A full application in an anaerobic digester is suggested to investigate the full potential of biomass for biomethane production.



Figure 3. TG curves of biomass samples (F, FS, and FS2) at different heating rates.



Figure 4. TG curves of biomass samples (P, PS, and PS2) at different heating rates.

Samula	Initial Load	BMP	TBMP		Biogas			
Sample	(gVS/ L)	$(mLCH_4/gVS)$	(mLCH <sub>4</sub> /gVS)	BD (%)	Total Vol (mL)	CH <sub>4</sub> (%)	CO <sub>2</sub> (%)	
Blank	ND	ND	ND	ND	170	ND	ND	
F	$32.5\pm2.11$	$28.15\pm0.77~\mathrm{a}$	$101.4 \pm 0.11$ b	28.2	349	50	50	
FS	$27.92\pm0.92$	$17.2\pm0.56$ a	$141.28 \pm 0.37$ c	12.17	260	50	50	
FS2	$28.1\pm0.77$	$19.75\pm1.22$ a	$44.08\pm0.75~\mathrm{a}$	44.8	234	75	25	
Р	$33.95 \pm 1.11$	$41.00 \pm 0.39$ b	$169.68 \pm 0.55$ d	24.1	444	50	50	
PS	$17.4 \pm 1.75$	$18.39 \pm 0.21$ a	$130.87 \pm 0.56$ c	14.05	208	78	22	
PS2	$18 \pm 1.22$	$47.60 \pm 0.22$ h	$49.00 \pm 0.12$	97	306	63	37	

Table 4. Biomethane potential analysis of the biomass samples.

BMP = biomethane potential; TBMP = theoretical biomethane potential; BD = biodegradability; ND = not detected. Mean with the same subscript within the column are not significantly different at p < 0.001.



Figure 5. Cumulative biogas volume.

Figures 6 and 7 provide the FTIR graph for fiber-based samples and pellet-based samples, respectively. While Table 5 depicted the comparison between standard band with bands from all biomass samples. For fiber-based samples, bands 1740.3 and 1736.5 appear in samples FS and FS2, respectively, which are not detected in F. These bands belong to aldehyde, suggesting cellulose metabolism occurred [54]. Another peak in FS and FS2 was 874.73 and 873.68 respectively was subjected to Alkenes (vinylidene) [53]. Vinylidene is known to be a transient molecule. It is possibly contributed by the ongoing bioconversion process throughout the cultivation phase. While for pellet-based samples, band 1244.05 belongs to aryl alkyl ether C-O in P, disappear in PS and PS2 samples. It is expected that the degradation process contributed to this phenomenon [55]. While in PS and PS2 samples, bands belong to aromatic rings 712.95 and 712.99, respectively detected [53]. However, some of the bands in this region are hard to interpret and belong to an unknown compound. Overall, the FTIR analysis provides insight into evidence of the biodegradation of the substrate throughout the cultivation phase.



Figure 7. FTIR analysis of P, PS, and PS2 samples.

Table 5.	Possible com	pound and bond	represented b	y FTIR	peaks of the sam	ples	[54,55].
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Absorption Band F	Absorption Band FS	Absorption Band FS2	Absorption Band P	Absorption Band PS	Absorption Band PS2	Standard Band	Possible Compounds/ Chains Rationale	Possible Bond
3376.88	3291.5	3376.39	3356.38	3284.36	3281.18	3350	Alcohol	О-Н
ND	ND	ND	2928.57	2928.57	ND	2930	Methylene	C-H
ND	1740.3	1736.53	ND	ND	ND	1730	Aldehyde	C=O
1648.09	1647.12	1646.87	1649.56	1649.35	1643.17	1640	Amide/Alkenes	C=C
1424.05	1418.63	1414.92	1428	1416.58	1413.68	1425	Lignin and wood	C-H
1244.73	1216.96	1217.26	1244.05	ND	ND	1200/1244	Ester/aryl alkyl ether	C-O/C-O-C
1036.15	1046.98	1044.15	1047.87	1044.66	1057.08	1100	Ether/alcohol	C-O
ND	874.73	873.68	899.77	872.93	873.49	890	Alkenes (vinylidene)	C-O
ND	ND	ND	ND	712.95	712.99	700	ND	RING

With regard to the objective of this article, the summary of strengths, weaknesses, and measures taken for the application of EFB based SMC for energy conversion is shown in Table 6.

SMC Application	Strength	Weakness	Measures to Overcome
Biogas	<ul> <li>Good substrate—simplified content but with adequate quality.</li> <li>Direct use.</li> <li>Better yield compared to other lignocellulose waste.</li> <li>Spent compost after second cultivation produces a better yield of methane and higher biodegradability.</li> </ul>	- Mechanical problems might occur in actual operation when using SMC as a substrate, such as clogging in the pipeline.	<ul> <li>Ongoing work for the application of the biomass in a digester.</li> <li>Optimization of the process parameter.</li> <li>Considering a suitable design of digester to fit the usage of a solid substrate.</li> </ul>
Fuel pellet	<ul> <li>Abundance at low cost.</li> <li>Easy management of waste.</li> <li>Greener solution.</li> </ul>	<ul> <li>Low calorific value (12.6 and 15.06 MJ/Kg) compared to commercial.</li> <li>High thermal degradation compared to untreated EFB, thus lowering the quality of biomass pellet.</li> <li>Required further processes such as mechanical grinding and drying.</li> <li>Logistic management due to different locations of the resource.</li> </ul>	<ul> <li>Upgrading facilities in a mushroom farm for integration as a biomass plant.</li> <li>Use as low-to-medium quality biomass pellets, such as gasifiers or boilers in the palm oil plant.</li> </ul>

Table 6. Strength, weakness and measures to overcome EFB based SMC for energy conversion.

## 4. Conclusions

In conclusion, EFB pellet is a suitable substrate for the cultivation of *Volvariella volvacea* and gives a better yield of 28% (BE) compared to fibers (9.83%). Based on the proximate and TGA analysis and other characterizations, EFB-based SMC was found to be a potential co-substrate for pellet fuel or can be used as a low-grade pellet fuel. The combustion potential of this biomass showed a lower HHV of 15.06 and 12.6 MJ/Kg, respectively, compared to EFB producing 20.57 and 19.06 MJ/Kg, respectively. Moreover, the BMP studies showed that the subsequent cultivation of *Volvariella volvacea* using EFB was a good pretreatment of the substrate before AD for biogas conversion. Further work can focus on optimizing the operating parameters of AD using EFB-based SMC. An in-depth analysis of material properties using different equipment could provide insightful information for the actual application. The use of this biomass as a blend for pellet production is also worth exploring. Ultimately, the use of EFB as a substrate for mushroom cultivation and SMC to support sustainable production has proven to be successful.

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## Abbreviations and Symbols

Explanation
Anaerobic digestion
Biodegradability
Biomethane potential
Carbon, hydrogen, nitrogen, sulfur, and
oxygen
Calorific value
Day for the first harvest
Harvesting day
Empty fruit bunch
Fourier-transform infrared spectroscopy
Fiber
Fruiting body
Fixed carbon
Spent mushroom compost of EFB fiber
Second spent mushroom compost of EFB fiber
High heating value
Moisture content
Spent mushroom compost
Theoretical biomethane potential
Thermogravimetry analysis
Thermogravimetry
Volatile matters

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