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USE OF CACAO POD HUSKS UNDER DIFFERENT PRETREATMENTS FOR BIOENERGY PRODUCTION

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The current study sought to determine the potential of cacao pod husks for energy use. Biogas potential, the kinetics of anaerobic digestion at bench scale, and physicochemical characterisation of materials were evaluated for cacao pod husks (CPH), depectinised cacao pod husks (CPH-D), and cacao pod husks treated by *Pleurotus* spp. (CPH-DF). These materials showed accumulated biogas productions of 314.86 ±4.45 $l \cdot kg^{-1}$ VS^{-1}, 369.49 ±15.98 $l \cdot kg^{-1}$ VS^{-1}, and 342.52 ±10.98 $l \cdot kg^{-1}$ VS^{-1}, respectively. The calorific values in CPH-DF, CPH, and CHP-DF were 17.65 $MJ \cdot kg^{-1}$, 15.43 $MJ \cdot kg^{-1}$, and 17.21 $MJ \cdot kg^{-1}$, respectively. These values indicate that the evaluated materials have a high potential to be used as biofuels.

Keywords: lignocellulosic biomass; waste; solid-state fermentation; anaerobic digestion; *Pleurotus* spp.

The use of biomass for energy purposes is a sustainable alternative to generating cleaner, low-cost, and more accessible energy from renewable resources (Kaniapan et al., 2021). This search for alternative energies is necessary because it is urgent to diversify the energy matrix, lowering our dependence on fossil fuels and greenhouse gas emissions caused by their combustion (Vasco-Correa and Li, 2015). Likewise, the energy obtained from biomass plays an essential role in energy transition in world policies in coming years (Reid et al., 2020), and waste biomasses are also considered a key alternative for the production of bioenergy due to their low cost and continuous availability in Latin American and Asian countries (Clauser et al., 2021). Cacao pod husks are the main waste during the processing; producing one tonne of dry beans generates about 10 tonnes of CPH. This residue is suitable for bioenergy production since it does not compete with food crops, and the industry's commercial attention is on dry cacao beans (Antwi et al., 2019). Cacao cultivation is of high economic importance in the world. In 2021, global grain production was approximately 4.92 million tonnes: Africa 77.4%, America 17.5%, and Asia and Oceania 5.1% (Daymond and Bekele, 2022). The cultivation of cacao (*Theobroma cacao* L.) is of great commercial importance worldwide; however, during fruit processing, many CPH are generated that are disposed of in the same farms without adequate management, constituting an environmental problem. This situation causes leachate, foul odours, and chemical imbalance in the soil and even becomes a potential source for developing pathogenic microorganisms that can affect the crop's health (Barragán et al., 2008; International Cocoa Organisation, 2023; Vriesmann et al., 2011). CPH has a high potential to generate value-added products such as bioplastics, antioxidants, and biofuels such as ethanol (Hennessey-Ramos et al., 2023). CPH has also been studied for the extraction of pectin, which is a polysaccharide that can be used as an additive in the food, pharmaceutical, and cosmetic industries (Hennessey-Ramos et al., 2021). However, this compound only represents 10–20% of CPH weight, leaving 80% of the biomass unused. Therefore, it is necessary to establish a comprehensive use of this depectinised biomass to obtain the second-generation biomolecules and biofuels (Valladares-Diestra et al., 2022). The anaerobic digestion processes for the production of biogas from the degradation of the lignocellulosic fraction of biomass present a positive energy balance, generating sustainable energy and stabilizing the waste into a material with potential use as a soil improver (Aguilera, 2017). Furthermore, farm energy production is an alternative for strengthening local agricultural economies and ensuring energy security in rural areas (Borek and Romaniuk, 2020). Some researchers have concluded that energy recovery, including anaerobic digestion, is the most effective way to use CPH within a sustainability framework (Maleka, 2016). However, applying pretreatment to enhance the biodegradability of the lignocellulosic fraction in CPH may be necessary (Antwi et al., 2019). Among the pretreatments used in lignocellulosic materials, solid state fermentation (SSF) is considered a sustainable process with low water consumption and has recently attracted attention for the transformation of agro-industrial waste into value-added products such as enzymes, biofuels, antioxidants, and other bioactive compounds (Verduzco-Oliva and Gutierrez-Uribe, 2020). Thus, improving the value of energy production through different treatments of waste is essential, due to the biogas can be used in multiple energy scenarios (Jafari, 2023).

Likewise, exploring other exploitation routes for CPH that lead to the generation of value-added products is necessary. In Colombia, although there is a great diversity of fungi that can be used in SSF processes, there are still few studies on the potential of these fungi to obtain value-added products from the degradation of lignocellulosic material such as agro-industrial waste (Dávila et al., 2020). In SSF processes, when hydrolysis of the lignocellulosic fraction and especially lignin occurs, some phenolic compounds are released, and fungi are capable of synthesizing bioactive compounds that can have antitumor, antimicrobial, antioxidant, and even antiviral effects (Verduzco-Oliva and Gutierrez-Uribe, 2020). This research sought to establish a route for the energy use of CPH. In this way, biogas potential, the kinetics of anaerobic digestion at bench scale, and the chemical and physical properties of cacao pods husk (CPH), depectinised cacao pods husk (CPH-D), and cacao pods husk depectinised and fermented by *Pleurotus* spp. (CPH-DF) were evaluated.

Material and methods

Feedstocks

CPH was supplied by SENA, Espinal, Colombia, at a latitude of 4° 10' 10" N, a longitude of 74° 55' 52" W, and an elevation of 348 m. After post-harvest operations, CPH was washed with distilled water and cut into slices to dry at 48 °C for 24 h. They were ground in a professional mill (MF 10 IKA-Werke, Staufen, Germany), sieved through a 500 µm mesh, and stored under refrigeration. CPH-D was obtained from a material depectinisation process using enzymatic hydrolysis. Once the pectin was obtained, the residual material was washed with distilled water and dried at 48 °C for 48 hours. CPH-DF was obtained for solid-state fermentation (SSF); the *Pleurotus* spp. strain was previously activated in wheat seeds. Seeds were previously hydrated to a humidity of approximately 75%, and the material was sterilised in an autoclave at 103.42 kPa, 121 °C, and 30 min. Once the flasks reached room temperature (24 °C), they were inoculated with 5 PDA (Potato Dextrose Agar) discs of *Pleurotus* spp. until complete mycelium spread was achieved. The CPH-D of 200 g with a humidity of approximately 85% was inoculated with 5% of *Pleurotus* spp. spawn, and bags were incubated at 30 °C for 15 days. Once SSF was finished, the material was dried at 48 °C for 24 h. CPH, CPH-D, and CPH-DF were stored and refrigerated at 4 °C until their respective analyses.

Chemical composition of the feedstock

The analysis of the chemical composition of CPH, CPH-D, and CPH-DF was carried out. Protein content was calculated from total nitrogen using conversion factor 6.25 and Kjeldahl method according to NTC 370:2011. The neutral detergent fibre (NDF) analysis was performed according to Van Soest et al. (1991), and acid detergent fibre (ADF) and acid detergent lignin (LDA) in accordance with method 973.18 (Horwitz and Latimer, 2006). The values obtained for NDF, ADF, and LDA were also expressed as hemicellulose, cellulose, and lignin. The total oxidisable organic carbon content was carried out according to NTC 5167:2022. Total solids (TS), fixed solids (FS), and volatile solids (VS) were determined by gravimetry following SM 2540 (APHA, 2005).

Anaerobic digestion

The VDI 4630 standard was used with some modifications for the anaerobic digestion process (VDI 4630:2006). The inoculum was obtained from an active anaerobic digester with effluent from a palm oil plant (POME). According to the physicochemical properties of the inoculum, the appropriate amounts of substrate and inoculum were introduced into each bottle in the ratio of 1 : 3, respectively, based on the volatile solids present in CPH, CPH-D, and CPH-DF. Amber glass bottles with a volume of approximately 1095 ml were used as batch reactors; the established mixing ratio is 600 ml in each bottle, leaving a free space for the biogas head of 495 ml. Each bottle was capped with a rubber cork pierced with a needle to accommodate a three-way pressure control valve (Fig. 3). Nitrogen was used to purge the bottles and ensure anaerobic conditions. The bottles were incubated at 35 °C for 35 days in a water bath, and each bottle was gently shaken daily to measure the pressure with a manometer with a pressure scale between 0 kPa and 137.89 kPa (Sánchez Marín, 2019). Pressure results were used to calculate the biogas yield using the ideal gas equation. To determine the biogas composition, the Multitec® 540 gas measurement equipment was used. The analyses were done in triplicate, and the blanks containing only the inoculum were used. The statistical analysis to determine significant differences in CPH, CPH-D, and CPH-DF biogas production was performed using the Minitab v.17 software (Minitab Inc., State College, PA, USA).

Higher heating value calculation

The higher heating value (HHV) was determined in a Parr 1341 bomb calorimeter following the ASTM D2015:2000 standard; the results were expressed in $MJ \cdot kg^{-1}$. The samples were introduced into a crucible inside the chamber, and an electrical conductor was connected to the sample to cause the combustion of the sample. The energy released is absorbed by the water of the calorimeter and is calculated by differences in temperature and mass (Gallipoliti et al., 2022).

Results and discussion

Physical and chemical characteristics

The results of the physical characteristics in terms of solids and chemical content of the three analysed materials are shown in Fig. 1 and Fig. 2, respectively. Table 1 shows the characterisation of the three materials evaluated. Since we worked with dry material, the contents of both total and volatile solids in the materials are high. The high value of volatile solids in the samples of CPH, CPH-D, and CPH-DF is an advantage given that volatile solids are the matter that is transformed by bacteria during anaerobic digestion, and as for fixed solids, materials with low content are preferred because this fraction is an inert material (Aguilera, 2017). The carbohydrates in CPH, CPH-D, and CPH-DF have a lignocellulosic structure that varies among them, given the treatments to which they were subjected. However, the CPH lignin, cellulose, and hemicellulose results are similar to those reported in other studies (Alvarez-Barreto et al., 2021;

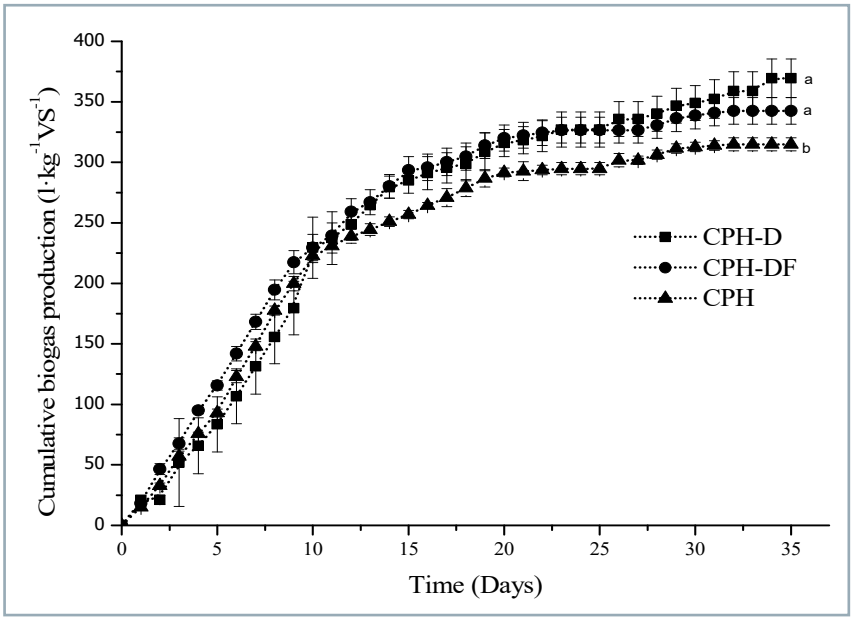


Fig. 1 Cumulative biogas production of the evaluated substrates

Hernández-Mendoza et al., 2021). The C/N ratio of the three analysed CPH, CPH-D, and CPH-DF materials were 59.42 ±3.03, 45.40 ±0.88, and 39.15 ±0.35, respectively.

In general, the C/N ratio of the three materials is well above the acceptable limits for biogas production (Olaya Arboleda and González Salcedo, 2009). A C/N ratio of 20 : 1 to 30:1 is acceptable, and ideal is a ratio of 16 : 1 to achieve a good biogas yield at a constant rate during anaerobic digestion. For this reason, materials with low and high yields must be mixed in appropriate proportions, which is achieved by adjusting the C/N ratio (Corace et al., 2006). However, this study aims to determine CPH, CPH-D, and CPH-DF's biogas potential alone and not co-digest with other materials. The best C/N ratio occurred in the fermented material (CPH-DF, 39.15 ±0.35), which can be explained given that the protein content in this treatment was the highest compared to the protein content of CPH and CPH-D. Although the C/N ratio of the evaluated materials is very high, it is pertinent in future studies to combine any of the evaluated treatments with a nitrogen source and take advantage of high carbon content in the materials. Dahunsi et al. (2019) adjusted the C/N ratio around 20 : 1 in a co-digestion of cacao shells and poultry manure. However, studies have also been conducted directly on cacao pod husk, applying different pretreatments while maintaining high C/N ratios, and biogas production has been possible (Antwi et al., 2019). In materials where it has been possible to reduce the lignin content through pretreatment, this leads to better biogas yields (Yan et al., 2015). The lignin content of CPH was 19.63 ±2.15 g·100 g^{-1}; in CPH-D, it was 28.15 ±1.34 g·100 g^{-1}; and in CPH-DF, it was 25.80 ±1.83 g·100 g^{-1}. The low lignin content in CPH compared to the other two treatments significantly influenced more outstanding biogas production. Although SSF with *Pleurotus* spp. was expected to reduce lignin to lower levels, it is necessary to add manganese ions to the substrate since this would stimulate the production of the manganese peroxidase (MnP) complex, which leads to a higher degree of delignification.

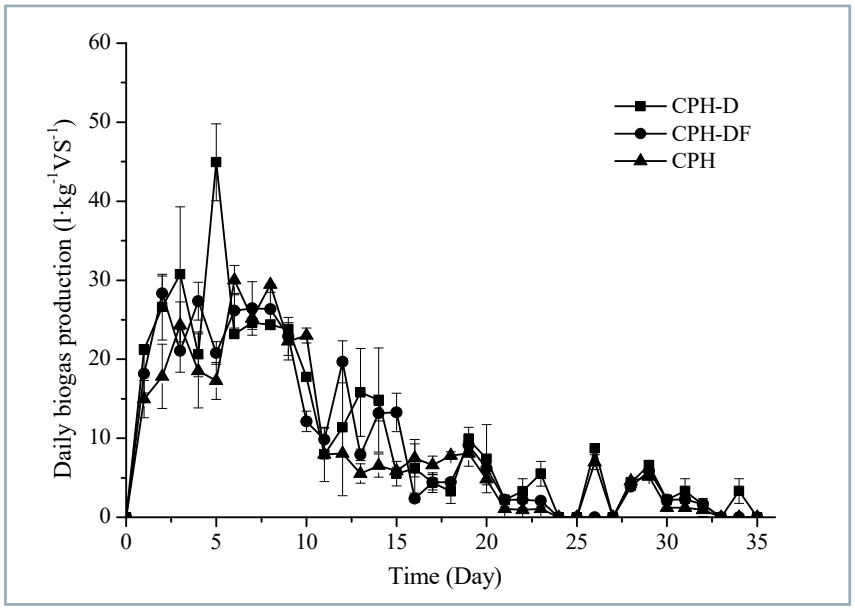


Fig. 2 Daily biogas production of the evaluated substrates

Cumulative and daily biogas production

The test was extended to 35 days because a total decrease in biogas began to be observed during this period. The three materials analysed, CPH, CPH-D, and CPH-DF, presented accumulated biogas production of 314.86 ±4.45 l·kg^{-1} VS^{-1}, 369.49 ±15.98 l·kg^{-1} VS^{-1}, and 342.52 ±10.98 l·kg^{-1} VS^{-1} (Figs 1 and 2). Kinetics in the cumulative production of biogas in the evaluated materials shows a positive effect on biodegradability since transformation of CPH, CPH-D, and CPH-DF to biogas is observed.

In the initial phase of biogas production, a similar trend is observed during the first 3 days in CPH, CPH-D, and CPH-DF. From day 3, CPH decreases biogas production compared to CPH-D and CPH-DF. The most significant biogas production is found between the first 10 days; after this period, production begins to decrease, and biogas is produced gradually until 30 days. Finally, after 30 days, the production of biogas begins to decline. However, measuring the methane content during all days was

Table 1 Physical and chemical properties of CPH, CPH-D, and CPH-DF

Parameter	CPH	CPH-D	CPH-DF
Cellulose (%)	22.81 ±0.69	33.3 ±1.56	29.1 ±0.99
Hemicellulose (%)	12.30 ±3.11	24.6 ±0.85	18.8 ±2.26
Lignin (%)	19.63 ±2.16	28.15 ±1.34	25.80 ±1.84
Nitrogen (%)	0.76 ±0.01	0.83 ±0.04	1.28 ±0.12
Protein (%)	4.73 ±0.09	5.19 ±0.25	7.99 ±0.73
Ashes (%)	10.81 ±0.56	4.07 ±0.03	3.37 ±0.20
Carbon (%)	44.9 ±1.41	37.65 ±1.06	47.00 ±0.72
C/N ratio	59.42 ±3.03	45.40 ±0.89	39.15 ±0.36
Total solids (%)	89.22 ±0.10	96.52 ±0.17	91.47 ±0.00
Volatile solids (%)	83.86 ±0.19	91.85 ±0.63	91.23 ±0.25
Fixed solids (%)	16.14 ±0.19	8.17 ±0.60	8.77 ±0.25
Biogas ($l \cdot kg^{-1}$ VS^{-1})	314.86 ±4.45	369.49 ±15.98	342.52 ±10.98

impossible for technical reasons. It was possible to measure the CPH-DF with the following data: CH4: 68.7%, CO_2: 20.8%, O_2: 4.9%, and H_2S: 89 ppm. The presence of flame was also verified over the days, being positive during the periods evaluated in the three materials (Fig. 3). During the first 15 days, accumulated biogas production for CPH-D, CPH-DF, and CPH was 77%, 84%, and 81%, respectively, and the maximum peaks of biogas production occurred in the first 10 days (Fig. 2). This indicates that some materials have high biodegradability in anaerobic digestion in the first 15 days. However, biogas production continues gradually, showing that some compounds have a slow degradation rate. Other authors reported similar results on CPH (Antwi et al., 2019).

Antwi et al. (2019) found a biogas accumulation of 374.5 $l \cdot kg^{-1}$ VS^{-1} in CPH, while in this experiment, it was 314.86 ±4.45 $l \cdot kg^{-1}$ VS^{-1}. The chemical composition of the substrate and the inoculum can explain the differences.

CPH in co-digestion with other substrates, such as mixing it with poultry manure to increase biogas production, reached up to 633.2±3.1 $l \cdot kg^{-1}$ VS^{-1} (Dahunsi et al., 2019). Of the treatments evaluated, the one that performed best was CPH-D with 369.49 ±15.98 $l \cdot kg^{-1}$ VS^{-1}, and CPH-DF with 342.52 ±10.98 $l \cdot kg^{-1}$ VS^{-1} was in the second place. Although CPH-D and CPH-DF have no significant differences in the accumulated biogas, CPH-D and CPH-DF are significantly different from CPH 314.86 ±4.45 $l \cdot kg^{-1}$ VS^{-1} ($p \leq 0.05$). This difference can be explained because the cellulose and hemicellulose contents in CPH-D and CPH-DF are much higher than in CPH (Table 1). Therefore, it indicates that methanogenic bacteria take advantage of this increase in the lignocellulosic fraction and produce greater biogas than CPH. Another aspect that favours the increase in biogas production in CPH-D and CPH-DF compared to CPH is the C/N ratio since it is much lower in CPH-D and CPH-DF, although the ideal is to maintain ratios between 15 and 20 (Zhang and Sun, 2014). To guarantee an adequate activity of microorganisms, a balance between C/N is necessary since microorganisms need between 15 and 30 times more carbon than nitrogen for their development (Fisgativa et al., 2016). Antwi et al. (2019) worked on hydrothermal pretreatment on CPH between 135 °C and 234 °C to hydrolyse the lignocellulosic fraction, achieving a more significant amount of biogas. They found that the highest biogas yield was 526 $l \cdot kg^{-1}$ VS^{-1} at 150 °C for 15 minutes and the calculated theoretical biogas yield in CPH was 922 $l \cdot kg^{-1}$ VS^{-1}. This pretreatment achieved changes in the contents of the lignocellulosic fraction concerning CPH from 26.10 $g \cdot 100\ g^{-1}$ to 36.85 $g \cdot 100\ g^{-1}$ of cellulose, from 21.00 $g \cdot 100\ g^{-1}$ to 32.16 $g \cdot 100\ g^{-1}$ of lignin, and from 4.82 $g \cdot 100\ g^{-1}$ to 4.41 $g \cdot 100\ g^{-1}$ of hemicellulose. In this way, it is vital to use pretreatments that lead to hydrolysing the lignocellulosic fraction in CPH, significantly increasing the contents of cellulose and hemicellulose and modifying the lignin structure to make the material more exposed to attack by methanogenic bacteria (Antwi et al., 2019). The economic factor is essential when choosing pretreatment, given that pretreatment generates an additional cost versus working with untreated materials. For this reason, pretreatments must achieve a positive energy balance (Rodriguez et al., 2015). In this way, CPH-D, which comes from despectinisation, has a positive effect since a product with high added value (such as pectin) is extracted, and depectinised material with greater levels of cellulose and hemicellulose is used for energy generation. Thus, an alternative use is presented under a circular economy approach.



Fig. 3 Batch reactors to determine biogas potential (a); Ignition test (b)

Higher heating value (HHV)

The calorific value in CPH-DF was the highest with a value of 17.65 $MJ \cdot kg^{-1}$. That is, it was the one that released the most significant amount of heat, followed by CPH-D with a value

of 17.21 MJ·kg^{-1}. Finally, the lowest value of 15.43 MJ·kg^{-1} was obtained in CPH. Syamsiro et al. (2011) reported the value of 17.01 MJ·kg^{-1} for CPH. Since the upper heat capacity is influenced by the content of C, H, O, and N, the data compared to literature may vary. Furthermore, depending on the variety and crop conditions of plant material, the physical properties such as calorific value are considerably affected (Betancur-Prisco et al., 2014). The results show a high potential to use the three materials as solid biofuels. The HHV values in CPH-D and CPH-DF stand out because they come from a platform where added value has already been generated, which indicates that it is possible to make comprehensive use of this waste within the framework of biorefinery concepts.

Conclusion

This paper demonstrates tracing the route for energy use from CPH. Anaerobic digestion turns out to be effective for the generation of biogas in the three materials analysed. The depectinised material CPH-D presented the best performance in biogas production, followed by CPH-DF and CPH. To increase biogas production, SSF should be supplemented with minerals such as Mn to obtain a higher degree of delignification and thus achieve better biogas yields with fungal pretreatment. Regarding the higher heating value, satisfactory results were also obtained in the three materials, so the generation of pellets, especially from CPH-D and CPH-DF, can become an alternative fuel for combustion systems.

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