



## Optimization of coconut husk by anaerobic digestion: Chemical pretreatment to reduce the recalcitrant structure

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### Abstract

Coconut husk is generated in huge amount in India and, it may be used as bioenergy sources for areas with limited fuel resources. Despite their potential applicability for energy, coconut husks and shells are often discarded or burned unprocessed, which leads to poor hygiene and environmental degradation. This study was done for proper utilization of coconut husk by anaerobic digestion. Chemical pre-treatment was found one of the best ways to treat lignocellulosic biomass. Chemical pre-treatment by sodium hydroxide was given to coconut husk to reduce the recalcitrant structure of biomass. The batch test was performed to analyze the biodegradability of pre-treated coconut husk. Results of chemically pre-treated coconut husk were found better as compare to untreated coconut husk. The highest cumulative methane yield was obtained in coconut husk pre-treated with sodium hydroxide (TCH)  $842.25 \pm 29.75$  mL followed by untreated coconut husk (UCH)  $548.60 \pm 6.0$  mL and control  $381.50 \pm 1.5$  mL. Similarly Volatile fatty acid (VFA) and soluble chemical oxygen demand (sCOD) were found higher in TCH as compared to UCH and control.

**Keywords:** Anaerobic digestion; coconut husk; chemical pre-treatment; biogas production

### 1. Introduction

Kerala, Tamil Nadu, Karnataka, Andhra Pradesh, Andaman and Nicobar, Lakshadweep, and Pondicherry are the major coconut-growing regions in India (Coconut Development Board, 2019). Production of coconut shells has increased as a result of consumption of coconut water and green coconut fruit pulp which is tend to become garbage in some areas and have detrimental social, economic, and environmental effects (Ayrilmis et al., 2011; Nunes et al., 2020). According to Ayrilmis et al. (2011), the coconut shell makes up about 85% of the weight of the fruit and it is made up of 33.30% lignin, 30.58% cellulose, 26.70% hemicellulose, 8.86% water, and 0.56% ash. Unripe green coconuts (*Cocos nucifera*), are growing for their water content, also produce a significant number of unripe green coconut husks, a fibrous byproduct. These husks could make up as much as 80% of the mass of the coconut. According to estimates, the world's coconut business produced up to 408,216,000 tonnes of husk in 2013, mainly in tropical regions where coconuts are grown (FAO, 2009; Freire et al., 2017). The countries of Indonesia, the Philippines, India, Brazil, Sri Lanka, Thailand, Vietnam, and Malaysia generate the majority of the world's commercially manufactured coir fibres despite the fact that coconut palms are found across the tropical regions of the world (Arancon, 2007). Around 12.05 million acres of land worldwide are used to grow coconut palms, and in recent years, about 61 million nuts per year have been produced overall (Arancon, 2007). The production of green coconut waste, which accounts for around 85% of the weight of coconuts consumed globally and the industrialisation of the manufacturing of coconut water, has also been grown. Coir fibres are made from the coconut's outer shell. The high lignin content of coir fibres (approximately 40%) makes it one of the toughest natural fibres (Van Dam et al., 2004). The coconut processing industries are still primarily focused on the processing of coconut meat, but little attention is paid to the

byproducts such as coconut water, coconut coir, and coconut shell, which are only produced on modest proportions (Van Dam et al., 2004). The cellulose included in coconut coir fiber can be utilised as a raw material for fermentation-based production of alternative energy sources including bioethanol and biohydrogen. An effective and successful pretreatment is essential to degrade lignin, and extract cellulose. In tropical and rainforest settings, coconut is a perennial fruit that thrives on sandy soils (Ebrahimi et al., 2017). It is most commonly found on islands and along the coasts, where it benefits from both water and sunlight exposure. Each year, Asia, Latin America, and Africa produce several million tonnes of coconuts globally. There were 250–300 million tonnes of coconuts produced worldwide as of the year 2018 (Obeng et al., 2020). There are numerous goods that may be made from the coconut plant and every part of it is beneficial. Fresh coconut fruit is valued for its juice, food, and animal feed. The husks are utilized as a source of raw materials and for wall hangings; the fibres are used, among other things, for clothes and bags (Muharja et al., 2018). The shell typically decomposes slowly and frequently causes problems. For example, in Ghana, where 73% of households rely on firewood for cooking and water heating, coconut husks with the shells still on them as well as other biomaterials like straw, rice husks, corn stalks, sawdust, cereal husks, sugarcane bagasse, and nutshells are potential bioresources that could be used as domestic fuel (Obeng et al., 2020). Anaerobic digestion is one of the ways to optimize the organic waste. Coconut husk is a lignocellulosic biomass which is optimize by giving proper pretreatment. Physical, chemical, biological, or a combination of these methods can be used for pretreatment. Chemical pretreatment is regarded as one of the most well-liked techniques for enhancing lignocellulosic biomass's biodegradability among these (Bensah and Mensah, 2013). The process of chemically altering the physical and chemical

characteristics of lignocellulosic biomass involves the use of chemicals like acids, bases, and ionic liquids (Kanaujia and Singh, 2022). The present study aims to optimize the coconut husk by giving chemical pretreatment with sodium hydroxide. The batch test for anaerobic digestion was performed to look over the biodegradability of feedstock after pretreatment.

## 2. Materials and methods

### 2.1. Collection and analytical

Green coconut shell was collected from market and fresh cow dung was collected from Rajnikhand (near BBAU campus, Lucknow). The coconut husk was cut into an average size of 1 cm and crushed properly. Cow dung and coconut husk were characterized, as listed in Table 1.

**Table.1 Initial Characterization of coconut husk and cow dung**

Parameters	Coconut husk	Cow dung
Moisture content (%)	11.5	81.23
Volatile solid (% of TS)	90.7	56.6
Ash content (% of TS)	2.6	24.5
Fixed Carbon (% of TS)	6.7	17.8

### 2.2. Pretreatment of coconut husk

A 200g of coconut husk was placed in Soxhlet thimble with 1% of NaOH solution. The mixture was heated and condensed at 80° C for 5 hour and condenses. The pretreated coconut husk was named as TCH, the untreated coconut husk was named as UCH and the control where only cow dung were fed.

### 2.3. Batch study

A batch study was carried out for 35 days at mesophilic temperature to examine the biodegradability of untreated and pretreated coconut husk. For the production of biogas, 1000 mL of glass bottles (Borosil, India) served as the bioreactors. In the bottle, 25g of pretreated coconut husk and 50g of cow dung were added, and distilled water was used to keep the mixture's volume at 700 mL. In another reactor 25g of untreated coconut husk was added along with 50g of cow dung. The coconut husk was added as per food to microbe (F/M) ratio 2 which is found best F/M ratio in most of the anaerobic digestion of lignocellulosic biomass (Saha et al., 2018; Veluchamy and Kalamdhad, 2017). One control was also placed in which 50 of cow dung was added only. All experiments were conducted in triplicate. The liquid displacement method was used for the production of biogas, 1000 mL aspirator bottles were filled with distilled water and these bottles were connected via pipe to the bioreactor. The displaced liquid was then collected in a final collection bucket. A daily biogas yield was calculated by the displaced liquid. The anaerobic environment inside the bottles was maintained by assuring air tight rubber cork cap on the mouth of bottle. Micronutrients such as magnesium ( $MgSO_4$ - 400 mg/L), calcium ( $CaCl_2$ -50 mg/L), iron ( $FeCl_3$ -40 mg/L), cobalt ( $CoCl_2$ -10 mg/L), zinc ( $ZnCl_2$ - 0.5 mg/L), and nickel ( $NiCl_2$ -0.5 mg/L) were also added to the reactor, along with feedstock and inoculum, to ensure proper microbial growth.

### 2.4. Analytical methods

According to APHA (2005), many parameters including moisture content (MC), total solids (TS), volatile solids (VS),

pH and soluble chemical oxygen demand (sCOD) were investigated. To analyse the pH a 10 g of substrate and 100 mL of deionized water were added in a conical flask and shaken horizontally for two hours at 150 rpm. The pH titration method developed by DiLallo and Albertson (1961) was used to evaluate volatile fatty acids (VFA).

## 3. Result and discussion

### 3.1. Effect of pH

The pH value of the reactor during the anaerobic digestion process was significantly influenced by the pretreatment of coconut husk was observed in Fig 1(a). The pH value in the range of 6.5 to 7.5 was observed during the anaerobic digestion (AD) process. There was decrease in pH observed within 14 day of AD. However, a further decrease in pH was not observed might be due to the stabilization of anaerobic microbial community. Veluchamy et al. (2018) also reported that after certain duration decrease in pH was not observed. Kanaujia et al. (2023) depicts the sudden decrease in pH was seen after 7<sup>th</sup> days afterwards it was increased.

### 3.2. Effect of volatile solids (VS)

The VS degradation pattern was shown in Fig. 1(b). A decrease in VS can be linked to the production of biogas and indicates that the biomass was lost in the AD system. More biogas was produced by the controlled AD process when VS degradation was higher. Maximum VS reduction was found in control that is 64.5% followed by TCH 70.5 % and UCH 78 %. The highest VS reduction was found in control which is due to absence of any recalcitrant substrate. The second highest VS reduction was found in TCH which is partially degraded due to pretreatment followed by UCH in which lowest VS reduction was found due to presence of more recalcitrant structure.

### 3.3. Effect of soluble chemical oxygen demand (sCOD)

The sCOD measures how much of the substrate has been hydrolyzed and solubilized by the microorganisms to produce biogas. The variation in sCOD of anaerobic digestion of TCH, UCH and control is shown in Fig.1(c). In initial day's sCOD of TCH, UCH and Control was  $1533 \pm 33$  mg/L,  $1038 \pm 8$  mg/L and  $968 \pm 12$  mg/L respectively. The process of hydrolysis transforms cellulose from the lignocellulosic biomass into fermentable reducing sugar, which enhances sCOD. The concentration of sCOD in all the reactors was increased initially then started to fall in the later stage. The highest sCOD was found in TCH-  $4387.5 \pm 42.5$  mg/L followed by in UCH-  $3380 \pm 50$  mg/L and in control-  $3125 \pm 25$  mg/L on 14<sup>th</sup> day.

### 3.4. Effects of volatile fatty acid (VFA)

Acidogenesis, acetogenesis, hydrolysis, and methanogenesis are the primary process involved in AD of lignocellulosic biomass. Accumulated VFA concentrations can be used to assess the efficacy of the hydrolysis process and acidogenesis, respectively. The formation of the VFA started when complex molecules began to decompose. The Fig. 1(d) depicts the variation of VFA concentration during digestion period. VFA content was gradually dropped increasing constantly after 14 days of AD process. The conversion of sugar in to acid, and the subsequent production of carbon dioxide and methane, caused an increase in the VFA content. The highest amount of VFA was found in TCH ( $1175 \pm 75$  mg/L) followed by in UCH ( $759.5 \pm 5.5$  mg/L) and Control ( $606.5 \pm 17.5$  mg/L).

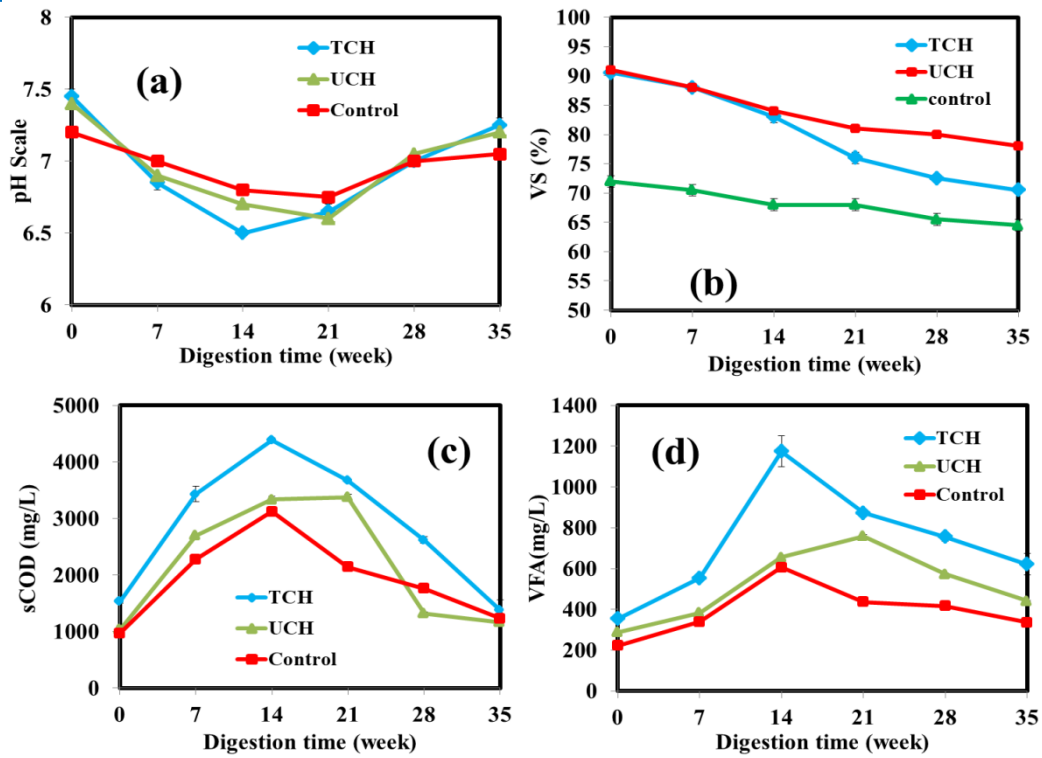


Fig. 1 Variation in analytical parameters before and after pretreatment (a) pHscale, (b) VS content, (c) sCOD and (d) VFA

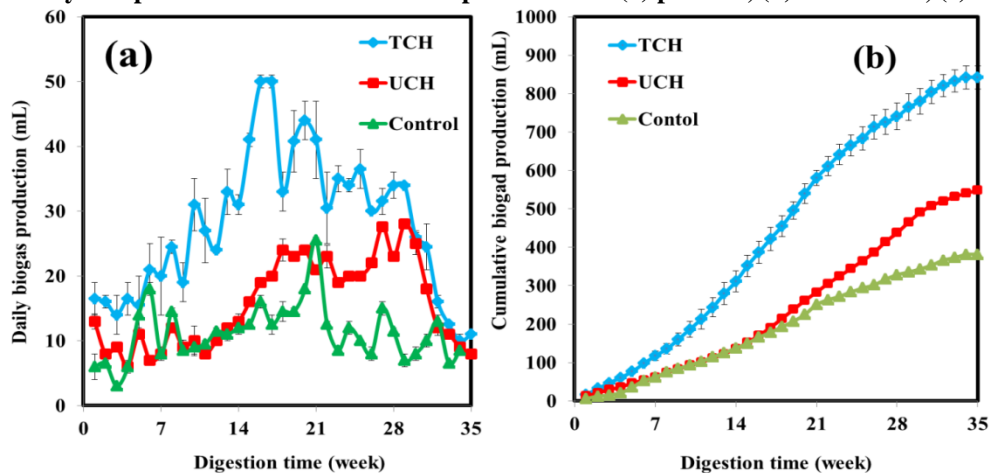


Fig. 2 (a) Daily methane yield and (b) cumulative methane production

### 3.5. Daily and cumulative methane yields

Methane production in this study was measured using the daily methane production rate. Producing methane initially required a lot of time because the substrate was lignocellulose (a plant based biomass). Due to the chemical pretreatment of coconut husk with sodium hydroxide, the maximum substrate utilization rate was seen in TCH in the first 14 days of digestion. Fig.2 shows that the maximum cumulative methane production around  $842.25 \pm 29.75$  mL was found in TCH followed by in UCH ( $541 \pm 6.1$  mL) and in control about  $381.5 \pm 1.5$  mL. Silverstein et al. (2007) proposed that sodium hydroxide was very effective in delignification and reduction of recalcitrant structure. The pretreatment of coconut husk with sodium hydroxide enhance the methane production by reducing the rigid and complex structure of coconut husk.

### 4. Conclusion

The primary agricultural product of the coconut palm is coconut oil; the fibre and husk that are produced are often disposed of as garbage. Utilization and management of coconut

husk is vital therefore AD is one of the alternative. Coconut husk is rigid and recalcitrant in nature, due to presence of lignin, which is required proper pretreatment for full utilization. It is depicts from study the chemical pretreatment of coconut husk was successfully reduced its recalcitrant structure. In this study, it was observed that pretreatment with sodium hydroxide improve the methane production by 2.20 fold as compared to control. The highest cumulative methane was found to be  $842.25 \pm 29.75$  mL in TCH followed by  $541 \pm 6.1$  mL in untreated coconut husk and  $381.50 \pm 1.5$  mL in control. Chemical pretreatment of coconut husk with sodium hydroxide partially dissolved the lignin and hemicellulose which provide microbial access to the cellulose resulting enhance the biogas production.

### Author contribution

**Nikita Kanaujia:** Conceptualization, Writing – original draft.

**Jiwan Singh:** Supervision, review & editing.

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#### Declaration of Competing Interest

The author declares that they have no known financial or interpersonal conflicts that would have appeared to have an impact on the work described in this publication.

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