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Transforming Oil Palm Waste into Energy: A Two-Stage Chemical Approach for Superior Biogas Yield

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ABSTRACT

Oil Palm Empty Fruit Bunches (OPEFB) are significant solid residues from oil palm mills that need effective and efficient disposal. Liquid residues in Palm Oil Mill Effluent (POME) are commonly processed via anaerobic digestion to produce biogas, a renewable energy source. The high organic content of OPEFB makes it a potential raw material for increasing biogas production, but the lignin in OPEFB can inhibit microorganisms that convert COD into biogas. Therefore, an initial treatment to remove lignin and improve the quality of OPEFB is essential. This study explores a chemical process to remove lignin from OPEFB, followed by a physical process to separate fibers of specific sizes. The treated OPEFB is then mixed with treated POME for slurry and enrichment. The goal is to determine the optimum concentration of NaOH that maximizes lignin decomposition in a two-stage chemical pretreatment using NaOH and NH_4OH. The delignification method involves soaking OPEFB in NaOH solutions at concentrations of 3%, 5%, and 7%, followed by treatment with a 15% NH_4OH solution. Lignin decomposition is then analyzed, and biogas production is tested after pretreatment. The study found that the highest lignin reduction occurred (36.12%) with a 7% NaOH and 15% NH_4OH treatment, resulting in a COD increase to 50,000 ppm and approximately 17% methane production.

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INTRODUCTION

The palm oil sector is moving toward sustainability by making biogas capture mandatory for all palm oil mills to reduce greenhouse gas emissions [1]. This industry generates both solid and liquid waste. One significant by-product is the Oil Palm Empty Fruit Bunch (OPEFB), produced mainly after oil extraction. About 0.96 tons of OPEFB are generated for every ton of palm oil. Traditionally, OPEFB has been used as boiler fuel, but this is limited due to unwanted smoke emissions and ash buildup [2]. Despite OPEFB being readily available, only a small portion is recycled as waste, while most are left on plantations to decompose [3][4]. OPEFB comprises approximately 40–43% cellulose, with glucose units linked by β-1,4-glycosidic bonds [5].

The high organic content of OPEFB makes it a potential raw material for biogas production. To enhance its suitability, an initial treatment is needed to remove the lignin, which can hinder microorganisms from converting Chemical Oxygen Demand (COD) into biogas. Biogas primarily consists of 25–45% carbon dioxide (CO_2) and 50–75% methane (CH_4). Without proper management, these gases can contribute to increased greenhouse gas (GHG) emissions [6].

OPEFB can be used as a raw material for biogas production. This material requires an initial delignification process involving breaking down lignin from complex lignocellulose materials to produce purer cellulose. Lignin forms covalent bonds with some hemicellulose components, making it difficult to degrade. This complex lignocellulose structure prevents microbes or chemicals from breaking down cellulose, thereby maintaining its integrity [7]. In the delignification of OPEFB using CHCl, the best results were achieved with a 90-minute treatment and 15% CHCl relative to the weight of OPEFB, yielding 80.66% - 89.62% cellulose, 40.06% - 44.51% hemicellulose, and 79.88% - 81.9% lignin. This treatment improved the degree of delignification by 61.4% compared to delignification without CHCl [8]. The OPEFB pretreatment combines a chemical process to remove lignin with a physical process to separate fibers of specific sizes, followed by slurry and enrichment using treated Palm Oil Mill Effluent (POME) and activated sludge. This approach aims to produce high-quality slurry for a Gas Mixing System Reactor (GMSR) to achieve stable and increased biogas production with Zero Waste High Methane (ZWHM). The enrichment process is expected to synergize and boost biogas production, making the GMSR an ideal solution for maintaining stability and sustainably enhancing biogas output.

Alkaline solutions, such as NaOH, are used to separate lignin from cellulose fibers [9]. This pretreatment can increase cellulose content and effectively remove lignin. However, the required NaOH concentration is relatively high (12–15%), leading to significant waste or by-products from the hydrolysis process [10]. To address this, a second stage involving the addition of NH_4OH is implemented. Adding NH_4OH at a concentration of 15% is reported to be effective for delignification in various biomass feedstocks [11].

This study aims to determine the optimum concentration of NaOH that achieves the highest lignin reduction through a two-stage process. This study will allow the treated OPEFB to be used as a raw material for increasing biogas production from POME. Additionally, the study aims to measure the increase in COD value after the two-stage chemical pretreatment and to assess the methane gas content produced during the biogas production process.

MATERIALS AND METHODS

Oil palm empty fruit bunch (OPEFB) samples were obtained from Sei Pagar Palm Plantation in Riau (SPA) and Cikasungka Palm Plantation in Jawa Barat (CKS), Palm Oil Mill Effluent (POME) samples were obtained from Cisasungka, Jawa Barat Palm Plantation, Sodium Hydroxide from Merck-Sygma Aldrich, and Ammonium Hydroxide from Sigma Aldrich.

OPEFB collection and preparation

OPEFB fiber was washed several times with tape water and sun-dried for a few days until the moisture content was minimal. This OPEFB was cut into 2-5 cm in length and then crushed into 0.5 mm size using a cutting machine.

Delignification Process

Oil Palm empty palm oil bunch powder 50 g is put into a glass beaker and added with NaOH with various concentrations of 3, 5, and 7%. The ratio of OPEFB and NaOH is 1:5. Samples were soaked for 90 minutes and then filtered and taken solids only. After that, samples are washed using aquadest until the pH is neutral. The samples that have been washed are dried using an oven for 10 hours at a temperature of 105°C.

OPEFB, which underwent the first delignification process with NaOH, was then dissolved into a 15% NH_4OH solution and heated for 60 minutes at 100°C. Samples are washed with aquadest to remove the NH_4OH solution. It is dried in an oven and then analyzed for lignin and cellulose content.

Analysis of Sample and Product

OPEFB was analyzed in proximate content using gravimetry methode, lignin, and hemicellulose. POME was analyzed in COD using UV-Vis spectrometry, and PH was analyzed using a pH meter, Metre Toledo. Biogas production was analyzed using gas chromatography TCD-Bruker.

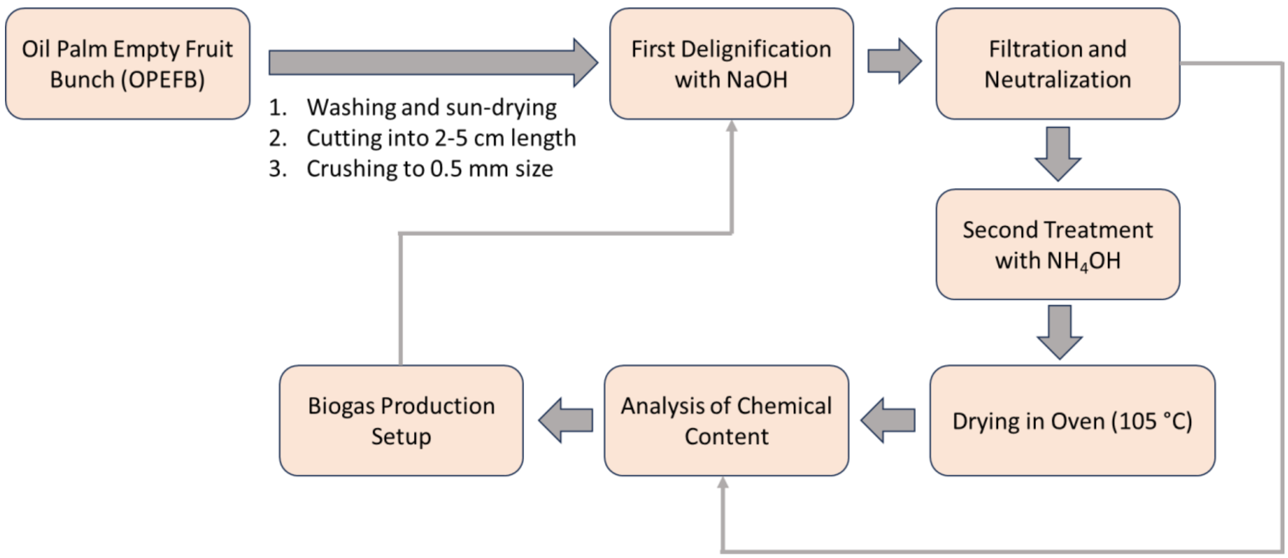

Figure 1. Schematic of the delignification process of OPEFB until it produces biogas

Biogas Production Set up

The anaerobic sludge used as the inoculum for the anaerobic digesters was concentrated by removing 30% of the water using a centrifuge. Once thickened, the sludge was transferred into 250 ml conical flasks and connected to the biogas collection system for degassing. This result was done using a stopper and a tube fitted with a check valve. The degassing process was carried out for seven days or until no biogas was produced.

The overall delignification process for producing biogas from OPEFB is illustrated in **Figure 1**. Biogas production involved mixing POME and sludge in a ratio of 3:7 by volume, with 70 ml of POME and 30 ml of sludge. OPEFB, pretreated with various NaOH concentrations, was added to the reactor in a ratio of 1:9. The operation was conducted under mesophilic conditions at 34-37°C and a pH of 7. The produced gas was analyzed using a Gas Chromatograph TCD.

RESULT AND DISCUSSION

Oil Palm Empty Fruit Bunches (OPEFB) are one of the most promising lignocellulosic biomass sources [12]. Lignocellulosic biomass consists of lignin, cellulose, and hemicellulose. Lignin hinders the enzymatic breakdown of hemicellulose and cellulose because of its complex, tightly bound, irregular, and random structure, primarily aromatic compounds [13,14]. **Table 1** shows the lignin and cellulose content of OPEFB from two palm plantations, Sei Pagar (SPA) and Cikasungka (CKS). **Table 1** shows the lignin and cellulose content of OPEFB from both the Cikasungka and Sei Pagar palm oil mills, which are similar. OPEFB from Cikasungka has an initial lignin content of 11.59% and a cellulose content of 88.41%. Meanwhile, OPEFB from Sei Pagar has an initial lignin content of 10.99% and a cellulose content of 89.01%.

Table 1. Lignin and Cellulose Content of OPEFB

Samples	Content (%)		
	Lignin	Cellulose	ADF
Cikasungka	11.59	87.11	45.43
Sei Pagar	10.99	89.01	48.89

Aliphatic chains connect the aromatic groups in lignin with 2-3 carbons. Due to the complexity of lignin, breaking down lignocellulosic components is challenging. Lignin has a more crystalline structure than cellulose and hemicellulose, making it difficult to degrade. Therefore, lignocellulose must be broken down to disrupt its crystalline structure so cellulose and hemicellulose can be degraded by cellulase [15]. Lignocellulosic biomass is a complex material, with 50–80% of its dry weight consisting of cellulose and hemicellulose, typically about 45% and 25%, respectively. The polysaccharides are bound by approximately 30% lignin on a dry basis, partially covalently associated with hemicellulose. This association prevents hydrolytic and acidic enzymes from accessing certain regions of holo-cellulose [16].

In addition to analyzing lignin and cellulose content, the water content, volatile matter, ash, and bound carbon in OPEFB were also investigated. The results of the water, volatile matter, and ash content analysis are shown in **Table 2**.

Table 2. Moisture and ash content of OPEFB

Sample	Moisture (%)	Ash (%)
Cikasungka	8.47	7.02
Sei Pagar	12.19	7.18

Figure 2. (a) raw OPEFB, (b) OPEFB after physic treatment, and (c) Chemical pretreated OPEFB

Unfortunately, no significant difference in ash content is observed between OPEFB from Cikasungka and Sei Pagar, with values of 7.02% for Cikasungka and 7.18% for Sei Pagar. However, the moisture content of OPEFB from Cikasungka is higher than Sei Pagar's. The moisture content in OPEFB is still high, necessitating drying before further processing.

OPEFB contains 82.4% hemicellulose, 62.9% cellulose, and 28.0% hemicellulose [17]. The initial processing of OPEFB into a slurry is expected to increase the COD value, making it suitable for anaerobic digestion to produce more stable and higher amounts of biogas. This process helps decompose solid waste as a raw material for biogas production.

For the pretreatment process, OPEFB was treated using alkaline solutions such as NaOH at concentrations of 3%, 5%, and 7%, followed by two steps of delignification using a 15% NH_4OH solution. Pretreatment with alkaline substances is expected to alter the lignocellulose composition of OPEFB. This result allows for a more comprehensive analysis of the factors that enhance bioconversion. The results of pretreating OPEFB with low concentrations of NH_4OH have led to chemical changes, as shown in **Figure 2**.

Table 3. Percent Lignin remover with pretreatment OPEFB with NaOH and NH_4OH

Samples	Stage 1 (NaOH)	Stage 2 (NaOH+NH_4OH 15%)
Sei Pagar 3%	22.11	33.76
Sei Pagar 5%	32.85	29.57
Sei Pagar 7%	27.66	36.12
Cisasungka 3%	15.15	23.52
Cisasungka 5%	23.00	28.27
Cisasungka 7%	21.88	27.84

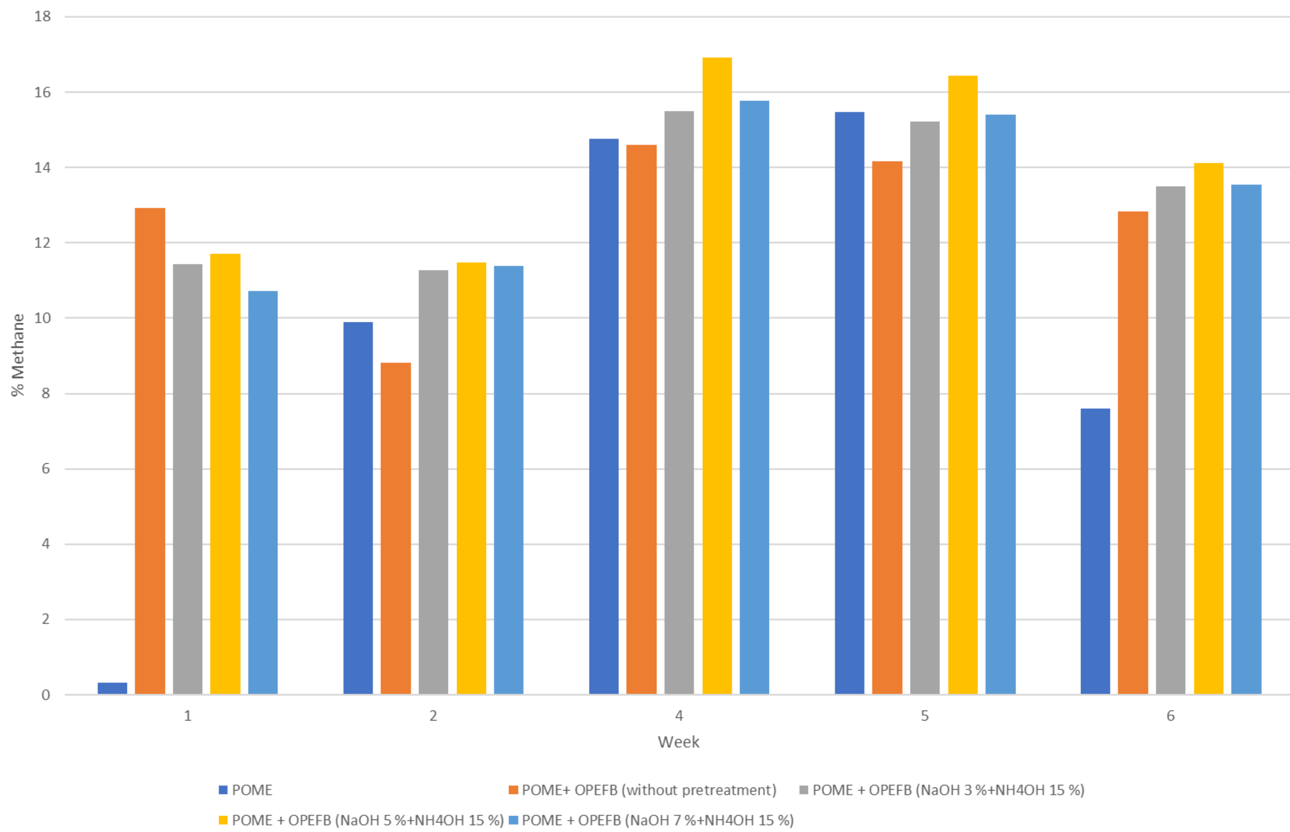

Figure 3. Percent Methane per Week with various chemical pretreatment

Chemical pretreatment with NaOH in the first stage and NH_4OH in the second stage effectively decomposes lignin. The highest lignin degradation was achieved using 7% NaOH and 15% NH4OH, resulting in 36.12% lignin decomposition in OPEFB from the Sei Pagar palm plantation (**Table 3**). This result is because the alkaline hydrolysis process disrupts the lignin structure [18]. In the delignification process, higher NaOH concentrations facilitate lignin degradation due to lignin's low softening and melting points [19, 20].

Table 4. COD and PH analysis after chemical pretreatment

Samples	Analyse	
	COD (ppm)	pH
POME	69,810	4.06
POME+ OPEFB (without pretreatment)	78,890	4.08
POME + OPEFB (NaOH 3 %+NH_4OH 15 %)	88,980	4.05
POME + OPEFB (NaOH 5 %+NH_4OH 15 %)	116,900	4.08
POME + OPEFB (NaOH 7 %+NH_4OH 15 %)	83,260	4.07

This research uses palm oil mill effluent (POME) as the raw material with a high chemical oxygen demand (COD) content. Adding chemically pretreated OPEFB increases the COD value, as shown in **Table 4**. The COD value rises from 15,000 ppm to 50,000 ppm following the two-stage chemical pretreatment of OPEFB. However, the addition of OPEFB does not significantly impact pH levels.

Co-digestion technology is increasingly used to process various organic wastes, both liquid and solid, simultaneously [21]. Unlike traditional mono-digestion, co-digestion enhances biogas production by efficiently managing two or more waste types. It reduces the equipment needed for waste streams and simplifies handling mixed waste while increasing the digester's organic loading rate (OLR) and biogas output [22]. Co-digestion also provides advantages like toxicity dilution, nutritional balancing, and beneficial synergistic effects, leading to higher methane yields and overall efficiency improvements in digestion [23]. **Figure 3** illustrates methane production from POME mixed with chemically pretreated OPEFB. **Figure 3** shows that adding OPEFB to POME increases methane gas production after undergoing a two-stage chemical process. The highest methane gas production was observed in the fourth week. Specifically, the most excellent methane production occurred in the mixture of POME and OPEFB after chemical pretreatment with 5% NaOH and 15% NH_4OH. This increase is due to the higher COD value and the carbon and nitrogen content in the co-digestion process [24]. Carbon is the primary food source for microbial growth, making POME's carbon and nitrogen levels crucial for digestion [25]. Nitrogen aids in the creation of enzymes. However, the low concentration of these nutrients in POME limits the amount of biogas produced.

On the other hand, OPEFB has a relatively high organic content, which means it can be used as a carbon source during co-digestion and help balance POME's low nitrogen levels [26]. However, OPEFB's resistance to bacterial enzymatic activity makes biodegrading challenging [27]. Therefore, pretreatment of OPEFB is necessary to improve its biodegradability and increase methane production during digestion.

CONCLUSION

Oil Palm Empty Fruit Bunch (OPEFB) pretreatment using NaOH and NH_4OH can degrade the lignin and cellulose in OPEFB. The highest lignin degradation was observed with pretreatment using 7% NaOH and 15% ammonia, resulting in a lignin content reduction of 36.12% in OPEFB from Sei Pagar.

Adding chemically pretreated OPEFB also increases the COD value of the raw materials for biogas production, with an average increase from 15,000 to 50,000 ppm. This result, in turn, boosts methane gas production in the co-digestion process.

Based on the results of this study, future research should focus on kinetic studies to understand the reaction mechanisms and rate-determining steps in the degradation process. Additionally, exploring more environmentally friendly and less hazardous chemicals for OPEFB pretreatment and analyzing the costs, benefits, and environmental impacts of applying this research on an industrial scale is essential.

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