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Optimization for biohydrogen purification process by chemical absorption techniques



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Abstract

Palm oil decanter cake and crude glycerol, which are characterized by their highly biodegradable organic content and nutrient-rich composition, are attractive ingredients for biohydrogen production. In this experiment, we investigated (1) how to produce hydrogen more effectively by co-fermenting palm oil decanter cake and crude glycerol and (2) how to improve the quality of the hydrogen gas produced via chemical absorption technology. This study was divided into two parts. In the first part, the co-fermentation was conducted with a fixed decanter cake concentration of 1% total solids (TS) w/v and variable crude glycerol concentrations (0.25–2.0% w/v). The results showed that maximum biohydrogen production was achieved with 2.0% w/v crude glycerol, which had a hydrogen yield of 131 L kg⁻¹ TS_{added} and a hydrogen productivity of 1310 mL L⁻¹ d⁻¹. In the second part of the experiment, biohydrogen purification was conducted using the chemical absorption technique by varying four different alkaline solutions: mono ethanolamine (MEA), ammonia (NH₃), sodium hydroxide (NaOH), and potassium hydroxide (KOH). The highest hydrogen purity of 98.9% v/v was reached with the MEA solution at a 5 M concentration and a 280 mL min⁻¹ feed mixed gas flow rate for an absorption time of 5 min. However, to achieve sustainable waste management in palm oil mill plants, the feasibility of integrating the biohydrogen production process with palm oil mill effluent from the biogas plants and applying a hydrogen gas quality improvement system need to be investigated further.

Keywords Absorption techniques, Biohydrogen production, Biohydrogen purification, Co-fermentation, Crude glycerol, Palm oil decanter cake

1 Introduction

Energy is an important resource in developing countries. In 2022, more than 82% of the energy used globally came from the burning of fossil fuels [1] in the form of coal, crude oil, and natural gas. According to the annual report of the Energy Policy and Planning Office in 2022, up to 78% of Thailand's energy stemmed from fossil fuels [2]. Due to the trend of increasing energy usage in households, industry, and transportation,

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¹ Faculty of Public Health, Mahidol University, Bangkok 10400, Thailand ² Center of Excellence on Environmental Health and Toxicology (EHT), OPS, MHESI, Bangkok 10400, Thailand coupled with the limited availability of fossil fuel resources, energy shortages will occur in the future. In addition, the combustion of fossil fuels, which are mainly composed of hydrocarbon compounds, directly causes the emission of pollutant gases such as carbon dioxide (CO_2), sulfur oxide (SO_x), and nitrogen oxide (NO_x) into the atmosphere [3]. Such combustion is also related to the greenhouse effect and abnormal seasons, such as global warming [3-5]. For this reason, many researchers have attempted to study the feasibility of new alternative energy sources in the form of clean energy, which does not adversely affect the environment and can replace the use of energy from fossil fuel combustion. Hydrogen energy is considered an alternative energy because hydrogen gas is non-toxic and can be utilized to provide heat, electricity, and



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transportation and thus increase the availability of lowcost, clean energy [6, 7]. Furthermore, the combustion of hydrogen does not release any pollutants into the atmosphere and generates heat energy up to 242 kJ mol⁻¹, as shown in the following reaction (Eq. (1)) [8]. The combustion of pure hydrogen gas and oxygen gas results in only steam (H₂O) and heat.

$$H_2 + \frac{1}{2}O_2 \to H_20 \; (\triangle H_{Too, H20}^{f, gas} = 242 \; kj \; mol^{-1}) \; (1)$$

In biohydrogen production, the purification process is considered a key issue as high-quality hydrogen can be utilized for power generation by fuel cell applications. The enriched hydrogen within the gas blend formed during fermentation can be used to obtain fuel with an upgraded power value. This can be achieved by removing the major byproduct, specifically, CO₂. Many technologies have been applied in H₂ purification, such as pressure swing adsorption, absorption, and membrane technology [9, 10]. The process of biogas purification through chemical absorption may offer a better alternative. Because of the chemical absorption, this process can be operated at low pressure with a product stream of high purity. The factors influencing the absorption of substances are (1) pH of the solvent, (2) concentrations, and (3) temperatures of both the gas and solvent. Solvents are considered economically attractive absorbents since they have low energy requirements and no environmental impact and are easily renewable and cost-effective [10, 11]. According to ISO hydrogen fuel specifications, biohydrogen must have a minimum hydrogen purity of 99.99% to be used as a fuel source [12, 13]. However, a hydrogen supply gas with a purity of at least 98% may be necessary for hydrogenation processes [14].

In this study, we focused on enhancing the efficiency of biohydrogen production via the co-digestion of crude glycerol from the biodiesel production process with palm oil decanter cake and optimizing the chemical absorption process to improve the purity of the produced hydrogen. This process could be integrated with those at palm oil mill plants to ensure the sustainable management of waste from these plants in the future.

2 Materials and methods

2.1 Raw materials

Palm oil decanter cake, a feedstock for biohydrogen production, was collected from a palm oil mill plant in the Suksomboon palm oil industry in Chonburi, Thailand. The sample was stored at 4 °C before use to reduce microbial activity and reduce the physical and chemical changes.

Crude glycerol with 78.4% w/v glycerol purity was also obtained from the Suksomboon palm oil industry in Chonburi, Thailand.

Anaerobic sludge was obtained from biogas plants in the Suksomboon palm oil industry in Chonburi, Thailand. The sample was heated at 100 °C for 30 min before use. This pretreatment of anaerobic sludge is undertaken to enrich the hydrogen producers and often relies on the spore-forming characteristics of hydrogen producers such as *Clostridium*, which are ubiquitous in anaerobic sludge and sediments. These organisms have a better chance of survival under drastic anaerobic sludge pretreatment conditions than non-spore-forming bacteria, such as methanogens, as the spores can germinate again under favorable conditions. Heat treatment of mixed cultures to enrich hydrogen producers is a simple, inexpensive, and effective method [15].

The characteristics of the raw materials are shown in Table 1.

2.2 Biohydrogen production process

The experimental biohydrogen production process was conducted in a 20 L semi-continuous stirred tank reactor (Fig. 1). The reactor had a working volume of 16 L and was operated for a hydraulic retention time (HRT) of 2 d. The process started with the co-fermentation of 1% w/v total solids (TS) of palm oil decanter cake and 0.25% w/v crude glycerol with 4 L of anaerobic sludge. We subsequently adjusted the volume to 16 L using tap water. A new mixture of substrate (8 L) was fed into the reactor daily, and a corresponding volume of digestate was removed to keep the operating volume at 16 L. The concentration of glycerol was increased slightly by 0.25,

 Table 1
 Characteristics of palm oil decanter cake, crude glycerol, and anaerobic sludge

Parameter	Decanter cake	Crude glycerol	Anaerobic sludge
Total solids (TS)	27.4% w/v	n.d.	31,700 mg L ⁻¹
Total volatile solids	n.d.	n.d.	14,900 mg L ⁻¹
Total COD	937,000 mg kg ⁻¹	1,080,000 mg L ⁻¹	18,200 mg L ⁻¹
Soluble COD	n.d.	n.d.	5,300 mg L^{-1}
рН	n.d.	6.53	9.3
Total carbon ^a	n.d.	n.d.	47.4% w/w
Total nitrogen ^a	n.d.	n.d.	4.3% w/w
Cellulose ^a	n.d.	n.d.	50.1% w/w
Hemicellulose ^a	n.d.	n.d.	30.7% w/w
Lignin ^a	n.d.	n.d.	10.4% w/w

n.d. not determined

^a Additional information from previous co-authors [16]



Fig. 1 Schematic diagram of 20 L reactor

0.50, 1.00, and 2.00% w/v crude glycerol, respectively, and the decanter cake was fixed at 1% TS w/v throughout the fermentation process. The pH of the substrate was adjusted to 5.5 with 85% phosphoric acid (H_3PO_4) as a pretreatment before use. H_3PO_4 is known to be a weak acid and therefore does not affect systemic microorganisms. It can break down lignocellulose biomass and improve cellulose digestibility. Because H_3PO_4 is environmentally friendly, the solid residue can be used as fertilizer. In addition, the lack of oxidizing power of H_3PO_4 makes it more beneficial in the treatment of lignocellulose biomass [17, 18]. The reactor was operated under thermophilic conditions (55 °C) with agitation at 100 rpm.

2.3 Chemical absorption process

The absorption process was conducted using four types of alkaline solutions, namely, potassium hydroxide (KOH), sodium hydroxide (NaOH), mono ethanolamine (MEA), and ammonia (NH₃). The absorption process was set up in a 500 mL Duran bottle with a working volume of 500 mL. The absorption process was started by fixing the percentage of gas composition for H_2 :CO₂ at 60:40, which was fed into the reactor with an alkaline solution of a different molarity. Then, to determine its final composition, the pure hydrogen that spilled over at the exit point was gathered into a gas syringe. For each experiment, the data were averaged from two replicates for reproducibility.

The parameters involved in the absorption process are listed in Table 2.

Table 2 Parameters for absorption technology adapted fromRohani et al. [19]

Parameter	Parameter range
Inlet gas flow rate (mL min ⁻¹)	280
Inlet gas composition ($H_2:CO_2$) (%, v/v)	60:40
Types of alkaline solutions	KOH, NaOH, NH _{3,} and MEA
Alkaline solution concentration (M)	1, 2 and 5
Alkaline solution temperature (°C)	Room temperature (20–25)
Data recorded times (min)	5, 15, 30, 60 and 90
Size of the reactor (mm)	Diameter 86 and height 181
Alkaline solution volume (mL)	500

3 Results and discussion

3.1 Biohydrogen production from co-fermentation of palm oil decanter cake with crude glycerol

We investigated the co-fermentation of 1% w/v TS palm oil decanter cake with different concentrations of crude glycerol for hydrogen production using anaerobic sludge under a controlled thermophilic condition of 55 °C and 100 rpm in a semi-continuous stirred tank reactor. The crude glycerol organic load strongly affected the overall fermentation process. Biohydrogen production with 2.0% w/v crude glycerol achieved the maximum hydrogen production and hydrogen content detected in the reactor at 17,900 mL and 53.1% v/v, respectively (Fig. 2). Methane was not detected during the fermentation process, which indicated that there was no methanogenic activity in the reactor.



Fig. 2 Parameter changes under the co-fermentation of palm oil decanter cake with crude glycerol (a) Hydrogen production, and hydrogen content; (b) pHs; (c) butyric acid and acetic acid; (d) ethanol, and propionic acid

The biohydrogen production trend measured by the percentage of hydrogen gas and the volume of hydrogen produced increased with the increasing crude glycerol concentration. The crude glycerol was added in increasing volumes as a co-substrate to help decompose organic matter. Although the pH in the fermentation system was maintained, we found that the increase in crude glycerol decreased the pH in the semi-continuous hydrogen fermentation system (Fig. 2b). Because nutrients with a simple molecular structure were continuously fed into the system, it allowed the microorganisms in the fermentation tank to function unimpeded. In addition, the crude glycerol contained a high chemical oxygen demand (COD) and a pH of 6.53. It was not only utilized as a cosubstrate for the hydrogen production, but also as an alternative pH-adjusting substance to keep the pH value within the reactor up, thereby resulting in semi-continuous hydrogen production.

Butyric acid and acetic acid were the primary volatile fatty acids detected in the liquid samples throughout the fermentation process (Fig. 2c). It could be concluded that this hydrogen fermentation process occurred according to the butyric acid-producing pathway, with butyric acid and acetic acid as the main products accumulated in the liquid sample. The results showed that during the first fermentation phase (days 1-41), not only were these the predominant acids produced within the reactor, but propionic acid was also highly accumulated compared to the other phases (Fig. 2d). Theoretically, 2 mol H_2 would be consumed from the propionic acid producing pathway. In Fig. 2d, propionic acid remained high from day 7-37, and the trend until day 63 was consistent with the $\rm H_2$ content in Fig. 2b. Many studies have shown were reported that the H₂ is consumed by propionic acid producing and homoacetogenesis pathways when using organic waste in the dark fermentation. If high levels of propionic acid were to have been found, the yield of hydrogen would have been reduced. Bacteria with propionic acid fermentation pathways may also have been evident. This describes propionic acid-type fermentation in which this group of bacteria uses a mixture of hydrogen gas to produce propionic acid, as in Eq. (2) [20], which results in a decrease in the yield of hydrogen.

$$C_6H_{12}O_6 + 2H_2 \rightarrow 2CH_3CH_2COOH + 2H_2O$$
(2)

When comparing the third and fourth phase fermentation, the percentage of hydrogen was similar, but the volume of hydrogen was different because of the increasing number of acidogenic bacteria in the fourth phase. Increasing the acetic acid pathway in the fermentation process resulted in an increased amount of hydrogen gas. Theoretically, if acetic acid were the only constituent in the fermentation process, hydrogen gas would be obtained as a product at 4 mol mol^{-1} glucose as a substrate, as shown in Eq. (3) [15, 21].

$$C_6H_{12}O_6 + 2H_2O \rightarrow 2CH_3CH_2COOH + 2CO_2 + 4H_2$$
(3)

Conversely, if butyric acid were the only component in the fermentation process, hydrogen gas would be obtained as a product at 2 mol mol⁻¹ glucose as a substrate, as shown in Eq. (4) [15, 21].

$$C_6H_{12}O_6 \rightarrow CH_3CH_2CH_2COOH + 2CO_2 + 2H_2$$
 (4)

In practice, the fermentation pathways occur in parallel with both the acetic and butyric acid pathways. This is especially true in fermentation processes with mixed cultures, such as in this experiment. As a result, both acetic and butyric acid were often found in the fermentation process, and the yield of hydrogen was consistently lower than theoretical value [11].

In the fourth phase of fermentation, butyric and acetic acid were primarily generated in the reactor. Ethanol was also accumulated at a high level compared to in the other fermentation phases (Fig. 2d). The high accumulation of undissociated acid above 19 mM proved to be a factor that inhibited acidogenesis and was a threshold concentration for a decrease in hydrogen yield and the onset of solventogenesis [16, 22]. Ethanol is an indicator of a solventogenesis reaction in a reactor. This reaction can occur when there is a proton imbalance and during detoxification into the bacterial cell. Within the acidity condition, the non-dissociated acid form may infiltrate through the cells, and protons will be discharged into the cells, which leads to harmful conditions. Subsequently, solventogenesis will be generated to maintain a neutral condition for a normal metabolic process.

With respect to waste reduction under co-fermentation using 1% w/v TS palm oil decanter cake with different concentrations of crude glycerol, our results showed that maximum soluble COD removal was achieved with 0.25% w/v crude glycerol waste and TS removal with 0.50% w/v crude glycerol waste (Tables 3 and 4). They further revealed the low removal efficiency of both soluble COD and TS because the hydrogen production process was a part of the anaerobic fermentation process. Organic matter is degraded from large organic molecules to smaller molecules resulting in the accumulation of various volatile organic compounds.

In terms of the hydrogen gas yield and productivity, the maximum hydrogen gas yield and productivity were observed at 2% w/v crude glycerol waste (Table 5). When

Table 3 Soluble chemical oxygen demand removal in the biogas production from co-fermentation of palm oil decanter cake with crude glycerol

Crude glycerol (% w/v)	SCOD influent (mg L^{-1})	SCOD effluent (mg L^{-1})	SCOD removal (%)
0.25	7,100±700	6,400±700	9.2±3.3
0.50	10,100±1,100	$9,000 \pm 600$	8.5 ± 3.9
1.00	17,300±1,900	15,300±1,200	8.6 ± 3.0
2.00	33,400±1,500	30,600±2,100	7.4±2.4

Table 4 Total solids removal in the biogas production from co-fermentation of palm oil decanter cake with crude glycerol

Crude glycerol (% w/v)	TS influent (mg L ⁻¹)	TS effluent (mg L ⁻¹)	TS removal (%)
0.25	23,200±1,500	21,400±1,500	8.1±2.5
0.50	23,300±2,100	19,800±1,300	13.7 ± 3.5
1.00	31,500±2,800	26,900±2,300	12.9 ± 5.2
2.00	$41,500 \pm 2,100$	$36,000 \pm 2,400$	13.2 ± 5.0

 Table 5
 Summary of hydrogen gas yield and productivity comparison

Crude glycerol (% w/v)	Yield (L kg ⁻¹ TS _{added})	Productivity (L L ⁻¹ d ⁻¹)
0.25	18.5±2.7	0.2±0.03
0.50	30.4±4.6	0.3 ± 0.05
1.00	54.2±4.6	0.5 ± 0.05
2.00	131±11	1.3 ± 0.10

comparing the hydrogen yields obtained in the present study with other studies, the hydrogen yields from the co-fermentation of decanter cake and crude glycerol were higher than those from other raw materials such as rice straw, potato waste, and raw wet steam-exploded cornstalk (Table 6). We noted that the type of substrate used for co-fermentation and different operating conditions affected the rate of hydrogen production. Due to the varying chemical compositions of the substrates, the difficulties with hydrolysis and the substrate transformation to hydrogen gas were different.

3.2 Biohydrogen purification using the chemical absorption technology

In this experiment, the effects of the concentrations of the alkaline solutions (NaOH, KOH, NH₃, MEA) at 1, 2, and 5 M on the hydrogen purification process were performed at the highest feed gas flow rate (280 mL min⁻¹) [19]. Our results showed that the efficiency of the CO_2 absorption increased with increasing concentrations of the alkaline solutions (Fig. 3a and d). The highest hydrogen purity was found when using 5 M of the MEA solution, which was able to achieve a maximum hydrogen gas of 99% v/v (Fig. 3a). However, the highest purity of 71% v/v after 90 min of absorption was obtained when comparing the same condition of 2 M of an alkaline solution. Due to the high stability of NH_3 , the efficiency of CO_2 absorption is low [28]. The gaseous CO_2 absorption with aqueous ammonia is shown as Eq. (5). The use of NH_3 as an absorbent in the removal of CO_2 from biogas plants is therefore not yet popular, and its efficacy needs to be studied further [28].

$$CO_2 + NH_3 + H_2O \rightarrow NH_4HCO_3$$
 (5)

When increasing the concentration to 5 M of MEA after 90 min of absorption, the result showed a higher

Feedstocks	Operation mode	рН	Temp. (°C)	Yield (L kg ⁻¹ TS _{added})	References
Rice straw	Batch	6.5	55	24.8	[23]
Raw wet steam-exploded cornstalk	Batch	6.5		12.0	[24]
Corn stover	Batch	7.2	37	51.9	[25]
Garden wastes	Batch	7.0	32	97.0	[26]
Potato waste	CSTR	5.5	35	30.0	[27]
DC and GLC	Semi-continuous	7.0	55	23.0	[16]
DC and GLC	Semi-continuous	7.0	55	43.3	[17]
DC and GLC	Semi-continuous	5.5	55	131	This study

DC Decanter cake

GLC Crude glycerol



Fig. 3 H₂ content via absorption technique with different alkaline solutions (a) MEA, (b) NH₃, (c) KOH, and (d) NaOH

percentage of hydrogen than the other alkaline solutions because 1 mol of MEA would react in equilibrium with 1 mol CO₂, as demonstrated in Eq. (6) [11, 28, 29]. Meanwhile, the use of more NaOH and KOH at 2 mol would react in equilibrium with 1 mol CO₂, as shown in Eqs. (7)–(8) [11, 30]. Moreover, a previous study revealed that a higher absorbent concentration may release more free ionic species, which would provide a more noteworthy CO₂ response within the absorption process. However, the trend of reduced gas purity showed the presence of a CO₂ saturation stage during absorption [31].

$$RNH_2 + CO_2 + H_20 \rightarrow RNH_3^+ + HCO_3^- \tag{6}$$

$$2NaOH + CO_2 \rightarrow Na_2CO_3 + H_2O$$
(7)

$$2\text{KOH} + \text{CO}_2 \rightarrow \text{K}_2\text{CO}_3 + \text{H}_2\text{O} \tag{8}$$

In theory, MEA is less effective in absorbing CO_2 than NaOH. It has been shown that 1.39 t of MEA and 0.9 t of NaOH are theoretically needed to produce 1 t of CO_2 , respectively [29, 32]. The main drawbacks of this approach have been reported in a previous study and include the substantial solvent energy needed to regenerate the chemical solution, the initial cost of the amine solvents, and the solvents that are lost through evaporation. Alkali salt is therefore attractive because it is less expensive and more accessible [29].

When considering the CO_2 removal efficiency in this work compared with that in previous studies (Table 7), it was evident that the type of alkaline solution used for absorption and the various operating circumstances

Table 7 Compare the CO2 removal efficiency from this researchcompared with previous research

Initial concentration CO ₂ (%, v/v)	Type of alkaline solution	Concentration of solution (M)	Removal efficiency (%)	References
46	NaOH	3.0	66	[30]
40	MEA	1.6	75	[38]
45	КОН	9.5	93	[39]
47	NaOH	0.1	93	[40]
40	NaOH	2.9	85	[41]
46	NH ₃	11.8	65	[42]
40	MEA	5.0	97	This study
40	NH ₃	5.0	85	This study
40	КОН	5.0	94	This study
40	NaOH	5.0	73	This study

had an impact on the hydrogen percentage rate. The absorbents must have a high quenching capacity for CO₂, be moisture-tolerant, and exhibit a strong preference for CO₂ over other gases. The absorption process can be more effective if the gaseous pollutants (CO_2) are more soluble in an absorbent than in biogas [33]. The concentration of the sorbent is crucial in chemical absorption. As the viscosity of the solution and the produced biogas intake rate are increased, the diffusion coefficient and absorption rate will decline. The amount of CO₂ in the gas stream and the surface of the sorbent have an impact on the rate of absorption [29, 34]. The pH of the solvent has a major impact on both the rate of absorption and the overall absorption process, with the rate of absorption decreasing with increasing acid or base in the process. While amine and NH_3 perform best in the pH range of 11.5–12, the optimal pH values for KOH and NaOH are 13 and 12.9, respectively [29, 35, 36]. Regeneration, requires a very small amount of energy. The heat of absorption varies between -25 and -50 kJ mol⁻¹ for physical absorption and between -60 and -90 kJ mol⁻¹ for chemical absorption. The ability to use regenerate energy in a reasonable way is a benefit of the absorber [29, 37]. In the process of chemical absorption, the choice of the proper solvent is crucial. To increase the efficiency and effectiveness of absorption, several more aspects must be considered in addition to those listed above. These include accessibility, affordability, volatility, gas solubility, innocuous gualities, and toxicity to both humans and the environment [29].

4 Conclusions

In this experiment, we aimed to enhance the efficiency of hydrogen production through co-fermentation using decanter cake and crude glycerol. The experiment was lab-scale and focused on the inclusion of the appropriate crude glycerol concentration for the hydrogen production process. We studied methods to improve the quality of hydrogen gas after the production process via absorption technology. Maximum biohydrogen production was observed at 2.0% w/v crude glycerol with a hydrogen yield of 131 L $\rm kg^{-1}~TS_{\rm added}$ and a hydrogen production rate of 1.3 L L⁻¹ d⁻¹. However, to achieve sustainable waste management at palm oil mill plants, the feasibility of using wastewater from biogas plants for dilution instead of tap water should be studied by controlling the concentration of organic matter before use. We performed biohydrogen purification using the absorption technique with four different solvents. The highest hydrogen purity (99% v/v) was found when using an MEA solution at a concentration of 5 M and a 280

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Authors' contributions

Wichayaporn Chusut analyzed the data, discussed the results, and writingoriginal draft preparation. Suwimon Kanchanasuta: project administration, acquisition of funding, provided conceptualization, discussed the results and final proofed the manuscript. Duangrat Inthorn has read and contributed to the final version of the manuscript; All authors have read and agreed to the published version of the manuscript.

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Declarations

Competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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