Quality improvement of syngas from gasification process of palm kernel using NiO/CaO catalysts on ceramic supporter in coupled with biochar absorbent from agricultural residues

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Abstract. In this study, palm kernel shells were utilized in the gasification process to produce syngas. In addition, biochar prepared from pyrolysis of the left-over mangosteen and durian peels were used in the gasification process to enhance the tar removal efficiency. The expected outcomes of this study could result in not only the generation of renewable energy from but also the waste utilization of agricultural residues. The effect of catalysts and biochar on the syngas quality improvement were particularly studied. Palm kernel shell was used as raw material. The compositions and heating values of the biomass were characterized by the proximate, ultimate, and bomb analyses, respectively. Syngas was produced from a downdraft gasifier connected with catalytic and adsorption units in sequence. The gasification process was operated with 1 kg of palm shell per batch at a fixed air flow rate of 25 L/min. The NiO/CaO (10 %wt) catalysts on ceramic supporter with various NiO contents of 2%, 4%, and 8% were synthesized by co-impregnation. Biochars were synthesized by pyrolysis process of durian and mangosteen peels at around 300 - 500 °C for 2 hrs. Morphology and compositions of the synthesized catalysts and biochar were analyzed. The results of SEM analysis showed that the NiO and CaO were deposited and well dispersed on the porous ceramic ring supporters. The presence of active NiO on the catalyst was also confirmed in FTIR result with (wavelength of 692 cm⁻¹). Biochar from durian and mangosteen peels, with BET surface area of 0.9219 - 0.9989 m²/g and adsorption pore size of 11.193 - 11.912 nm, were obtained from a pyrolysis process at 400 - 700 °C. The syngas samples were collected from the gasification unit every 15 min to 60 min. The gas chromatography was used to analyze the syngas compositions. The GC results indicated that increasing NiO contents in the catalysts tended to result in increasing CH₄ composition of the syngas for both systems with and without biochar. With tar filtration unit containing biochar, the ability of tar removal was significantly increased by 2 to 9 times comparing to the system without biochar.

1. Introduction
Thailand is one of important countries in term of food resources. Thailand has abundant areas of agriculture, including, rice, corn, sugar crane, and oil palm, etc. In southern Thailand, oil palm is one of major economic agriculture and the industry itself is continuously growing. This leading to a massive
amount of biomass residues left from agricultural activities and palm oil refinery. In 2018, the estimated plantation area of oil palm agriculture in Thailand was about 2 million acres, yielding 15.39 million tons of oil palm crops for palm oil production [1]. Biomass residues of palm oil refinery is approximately 78-82% composing of empty fruit bunch, fiber, and kernel shell with the heating values of 16.32, 17.25, and 18.53 MJ, respectively. These biomass residues can be used as feedstocks in thermo-chemical process to produce biofuel or syngas. Syngas, primarily composed of hydrogen, methane, carbon monoxide, carbon dioxide, light hydrocarbons, can be produced by gasification process of biomass; however, heavy and light tar is also generated. Tar in syngas leads to less efficiency and gas engine damage because of its high viscosity. There are several of approaches to remove tar from the produced syngas for example separation processes. One practical approach is to decrease the tar content by thermal cracking reactions with the aid of steam or catalyst to transform tar into high heating value gaseous components [2].

Lately, there are several research studies concerning an improvement of quality and quantity of syngas using nickel oxide or base oxide in downdraft gasifier [3] Nickel oxide is mostly used to enhance tar cracking and hydrocarbon removal in syngas at temperature higher than 780 °C [4] However, nickel oxide is relatively pricey, therefore, other oxide catalysts such as calcium oxide is used together with nickel oxide to save the cost. Phanpruk and Khamwichit [5] found that yields of syngas produced from gasification process of kernel shell using downdraft gasifier were relatively comparable for nickel oxide and calcium oxide (74.25 and 67.63%, respectively).

The purpose of this research was to study the effects of NiO/CaO-based catalysts and biochar obtained from agricultural biomass residues on the syngas’s quality improvement from the gasification process of palm kernels. Three different dosage of NiO/CaO (2%, 4%, 8% NiO/10% CaO) doping on ceramic rings were synthesized by co-impregnation sol-gel process. Biochar synthesized from durian and mangosteen peels was utilized to adsorb liquid and gaseous tar.

2. Materials and methods

2.1. Catalyst preparation
Nickel nitrate hexahydrate solution (Ni(NO₃)₂•6H₂O) and Calcium nitrate solution (Ca(NO₃)₂•4H₂O) were used as precursors to prepare 2%, 4%, 8% NiO/10% CaO of the gel solution. Ceramic rings were used as substrate due to their high surface area [2]. The ceramic rings were soaked in precursor solutions in the hot basin at 70 °C until the gel solution was completely dried. Then they were heated in the oven at 110 °C for 24 h before calcining 950 °C for 6 h. Finally, the calcined ceramic rings were left at room temperature in the desiccator.

2.2. Biochar preparation
Durian and Mangosteen peels were sun-dried and chopped to be approximately 2 cm in diameter before putting into 18 L of bucket. The bucket filled with the dried and chopped biomass was placed along with woodchips and palm fiber (used as fuel for synthesis of biochar) into 100 L Stainless Steel tank. Pyrolysis process of biochar was conducted at 400-600 °C for 2 h.

2.3. Downdraft gasification process
Palm kernel shell (PKS) was dried in the oven at 80 °C for 24 h prior to be placed in the gasifier with the amount of 1 kg. Air was pumped at a constant rate of 25 L/min to gasifier to maintain PKS/air ratio of 1:0.6. Process is comprised of gasifier, catalytic reactor, tar condenser, and tar filter. The synthesized ceramic ring doped with catalyst was filled in the catalytic reactor with the ratio of PKS/catalyst of 1:0.25. Cracked tar from catalytic reactor was then condensed in the tar condenser. Syngas was passed to tar filter unit, packed with 1 kg of biochar (the amount ratio of palm kernels to biochar of 1:1), for the adsorption of gaseous tar. Figure 1 illustrates the gasification process diagram used in this study.
2.4. Characterization of biomass, biochar and catalytic ceramic ring

2.4.1. Composition and heating value

Compositions of biomass (kernel shell) and synthesized biochar from durian and mangosteen peels in this study were analysed according to ASTMD3172-3175 standard. Thermo Quest model Flash EA 1112 was used to analyse elemental composition of both biomass and biochar. While heating value of kernel shell was analysed using Bomb calorimeter model AC 500.

2.4.2. Morphology

Morphological properties of biomass, biochar, and catalyst were examined by Scanning Electron Microscope (SEM, Model Merlin compact, Zeiss). The specimens were mounted on aluminum stubs, sputter-coated with gold and examined in a Merlin compact scanning electron microscope. Samples were analyzed at 1,000x and 10,000x magnifications.

2.4.3. Specific surface area of the synthesized biochar and catalyst

Specific surface areas and pore diameters of the synthesized biochar were analyzed using the Brunauer–Emmett–Teller (BET) technique (Surface Area and Porosity Analyzer (BET), ASAP2460, Micromeritics, USA).

2.4.4. Chemical property characterization of catalyst

Functional groups of chemical structures of doped ceramic ring were analyzed by ATR-FTIR Spectrometer (Model Tensor 27, Bruker) using ATR-FTIR technique.

2.5 Syngas analysis

Syngas produced from the gasification process of kernel shell was collected at a sampling point of tar filter unit every 15 min from the beginning of process to 60 min. Gas samples were collected into Tedlar bags then were analysed by Gas Chromatography model 7890.
3. Results and discussions

3.1. Biomass characterization
The results of proximate and ultimate analysis of the palm kernels used in this study are shown in Table 1. Volatile matter was found to be approximately 71.6%, whereas the fixed carbon and moisture contents were 15.1% and 9.9%, respectively. Since the kernels contained high amount of volatile matter, the material can be potentially combusted in the gasification process to generate energy. The CHN-O elemental analysis indicated that the combustible kernels consisted of carbon, hydrogen, and oxygen which were suitable to be used as biomass raw material for the purposed gasification. As shown in Table 2, high heating value (HHV) and low heating value (LHV) of the shell obtained from Bomb calorimeter measurement were roughly 4,830 and 4,225 kcal/kg, respectively. In general, the results were in good agreement with past related research studies reported elsewhere in which the biomass materials were successfully utilized to produce syngas.

Table 1. Proximate and Ultimate analysis results for the palm kernels.

| Moisture content | Ash (% wt dry basis) | Volatile matter | Fixed carbon | C (% wt dry basis) | H (% wt dry basis) | O (% wt dry basis) | N (% wt dry basis) | Ref.          |
|------------------|----------------------|-----------------|--------------|-------------------|-------------------|-------------------|-------------------|--------------|
| 9.94             | 3.41                 | 71.57           | 15.08        | 52.30             | 52.30             | 39.17             | 0.42              | This study    |
| 12.99            | 2.73                 | 70.96           | 13.31        | 46.61             | 5.55              | 35.85             | 0.20              | [6]          |
| 11.00            | 2.10                 | 67.2            | 19.7         | 49.74             | 5.32              | 44.86             | 0.08              | [7]          |
| 10.23            | 3.24                 | 85.11           | 1.42         | 47.88             | 5.15              | 42.69             | 0.94              | [8]          |
| 6.33             | 11.75                | 62.82           | 19.10        | 44.29             | 9.01              | 43.13             | 2.37              | [9]          |
| 9.70             | 4.48                 | 80.81           | 13.81        | 48.61             | 5.70              | 44.70             | 1.01              | [10]         |

Table 2. Heating values of palm kernels.

| HHV (kcal/kg) | LHV (kcal/kg) | Ref.          |
|--------------|--------------|--------------|
| 4,830.70     | 4,225.39     | This study   |
| 4,512.13     | 3,996.46     | [6]          |
| 5,469.57     | 5,812.96     | [11]         |
| 4,497.98     | N/A          | [10]         |
| 3,857.46     | N/A          | [12]         |

3.2. Catalyst characterization
Morphology of the prepared catalysts containing NiO and CaO on ceramic supporter was analyzed from the SEM results. Figure 2 illustrates the SEM pictures of the (a) NiO (2 wt%)/CaO (10 wt%)/Ceramic ring, (b) NiO (4 wt%)/CaO (10 wt%)/Ceramic ring, and (c) NiO (8 wt%)/CaO (10 wt%)/Ceramic ring catalysts at a magnification around 10,000x magnifications. The micrographs exhibit rough surface with uniform distribution of the NiO/CaO catalyst crystals on the porous supporter. At fixed 10 wt% CaO, the size of the disperse agglomerates tended to increase with increasing co-impregnated NiO contents. The porosity of the catalysts was observed to be decreased as the NiO increased from 2 wt% to 8 wt%.
NiO and CaO contents were evaluated from element analysis of the EDX results. Figures 3 shows the EDX results of the prepared NiO (x%)/CaO (10%)/Ceramic catalysts with varied NiO contents of 2 wt%, 4 wt%, and 8 wt%. The EDX analysis suggested that active NiO and CaO were successfully co-impregnated. The catalysts contained Ni and Ca contents relatively close to the prepared solution concentrations. The increase in the concentration of Ni(NO$_3$)$_2$.6H$_2$O in the solution generally increased the Ni content in the catalyst. The Ni compositions were found to be 2.86% for the NiO(2%)/CaO(10%), 6.85% for the NiO(4%)/CaO(10%), and 10.52% for the NiO(2%)/CaO(10%), respectively. The presence of NiO and CaO deposited on the ceramic surface as seen from the EDX results was the indicator of effective impregnation of the active compounds. This could be attributed to the interaction between NiO/CaO and the silicate oxide (SiO$_2$) component of the ceramic supporter, as suggested in Wang’s study [13]. In addition, the presence of active NiO on the catalyst was also confirmed in FTIR result where the peak at the wavelength of 692 cm$^{-1}$ associated with Ni-O stretching vibration band was observed [14], as shown in Figure 4. The specific surface area of the prepared NiO/CaO catalysts obtained from BET analysis was found to be 6.192 m$^2$/g with an average pore size of 14.48 nm.

Figure 2. SEM results at 15 kV (1,000x magnifications) of (a) Ceramic ring, (b) 2%NiO/10%CaO/Ceramic ring, (c) 4%NiO/10%CaO/Ceramic ring and (d) 8%NiO/10%CaO/Ceramic ring.

Figure 3. EDX results at 15 kV (1,000x magnifications) of (a) Ceramic ring, (b) 2%NiO/10%CaO/Ceramic ring, (c) 4%NiO/10%CaO/Ceramic ring and (d) 8%NiO/10%CaO/Ceramic ring.
3.3. Biochar characterization

Biochar synthesized from durian and mangosteen peel residues was used as the physical absorbent for tar removal in this study at a fixed ratio of 50:50 by weight of durian and mangosteen. Figure 5 illustrates SEM results of biochar at 10 kV with a magnification of 500 and 10k times. The SEM pictures of the sectional fracture surface of both from durian and mangosteen show porous structure with various pore size across. It seemed that the pore size distribution of the biochar made from durian peels appeared to be more uniform than those of obtained from mangosteen. Biochar from mangosteen possessed porous structure with broader pore size distribution across the surface. The SEM results were in good with BET surface analysis.

**Figure 4.** FTIR analysis of Ceramic ring, 2%NiO/10%CaO/Ceramic ring, 4%NiO/10%CaO/Ceramic ring and 8%NiO/10%CaO/Ceramic ring.

**Figure 5.** SEM results at 10 kV of
(a) durian-peel biochar (500x magnification),
(b) durian-peel biochar (1,000x magnification),
(c) mangosteen-peel biochar (500x magnification) and
(d) mangosteen-peel biochar (1,000x magnification)
The EDS analysis indicated the main elemental compositions of the prepared biochar, as shown in Figure 6. The biochar obtained from durian peels consisted of 75.1% C, 12.4% O, and 11.3% K, respectively. The elemental composition results were typical for biochar which could be used as adsorbent to physically adsorb tar compounds from syngas in the gasification process [15].

![Figure 6. EDX results at 10 kV (500x magnification) of (a) durian-peel biochar and (b) mangosteen-peel biochar.](image)

Table 3 shows the BET analysis results of the synthesized biochar. The specific surface areas (SSF) were found to be 0.9989 m²/g for the durian-peel biochar, and 0.9219 m²/g for the mangosteen-peel biochar. In addition, the average pore diameter of the durian-peel biochar (11.193 nm) was a bit relatively smaller than that of the mangosteen-peel biochar (11.912 nm). It was believed that the mixture of 50:50 ratio between the materials would be capable to adsorb condensed volatile tar residues with wide spectrum of molecular sizes. The gasification performance study will be further discussed.

| Biochar Type     | BET Surface Area (m²/g) | Adsorption Average Pore Diameter (nm) | Ref.       |
|------------------|-------------------------|--------------------------------------|------------|
| Durian-peels     | 0.9989                  | 11.193                               | This study |
| Mango-peels      | 0.9219                  | 11.912                               |            |
| Palm oil char    | 1                       | N/A                                  | [16]       |
| Peanut hull char | 1.54                    |                                      |            |

3.4. Gasification performance study

Compositions of syngas for each experiment were illustrated in Table 4. The gas chromatography (GC) results of the syngas samples obtained from the gasification process at 15, 25, 35, 45, and 60 min were used to analyze the compositions of syngas produced from the process. In this study, CH₄ and CO₂ were detected by GC analysis. From the table, the presence of the catalysts affected the syngas compositions. Increasing NiO contents in the catalysts tended to result in increasing CH₄ composition of the syngas. The analysis of GC results for the experiment without biochar suggested that the system with the catalyst with 8% NiO gave the highest maximum percent composition of CH₄ in syngas (4.61%). Followed by maximum compositions of CH₄ at 3.01% for 4% NiO, 2.44% for 2% NiO. Maximum compositions of CH₄ for no-catalyst and 2% NiO were insignificantly different. It was believed that the catalysts would facilitate the further chemical cracking reactions of the volatile tar compounds. The results were in good agreement with [6] study in which NiO was demonstrated to effectively improve the CH₄ yields for the gasification process of palm residues. For the experiment with the use of biochar, the maximum composition of CH₄ for 8% NiO was the highest (3.27%), followed by those of 2% NiO and 4% NiO (2.96%, 1.14%). From the observation of experiment, the best flames occurred during 25-35 mins of the processing time, in which the color of the flames were light blues, indicating an existence of high syngas and low tar. From the results, the %CH₄ values obtained from the system with biochar filter were slightly lower than those of the system without the filter unit. It is to mention that the back pressure built-up in the system having the packed-bed biochar filter unit installed was relatively high, resulting in decreasing
flowrate of the syngas product that flew past the filter. During the process, the rest of the produced gas was released at the bypass outlet. The difference in flow characteristics particularly the pressure drop across the filter unit could affect the physical adsorption of other components of the produced gas besides tar.

### Table 4. CH\textsubscript{4} and CO\textsubscript{2} composition of syngas.

| Sample                               | 15 min | 25 min | 35 min | 45 min | 60 min |
|--------------------------------------|--------|--------|--------|--------|--------|
|                                      | \(\text{CH}_4\) (%vol) | \(\text{CO}_2\) (%vol) | \(\text{CH}_4\) (%vol) | \(\text{CO}_2\) (%vol) | \(\text{CH}_4\) (%vol) | \(\text{CO}_2\) (%vol) |
| Without Biochar                      |        |        |        |        |        |        |
| No Catalyst (ceramic supports)       | 0.09   | 2.29   | 0.10   | 3.32   | 0.15   | 4.30   | 0.19   | 5.36   | 0.13   | 4.85   |
| 2%NiO/10%CaO                        | 0.35   | 3.20   | 1.37   | 19.44  | 2.44   | 33.40  | 2.75   | 41.56  | 0.64   | 24.58  |
| 4%NiO/10%CaO                        | 0.73   | 33.55  | 3.01   | 50.17  | 2.41   | 61.21  | 1.37   | 57.86  | 0.44   | 50.00  |
| 8%NiO/10%CaO                        | 0.63   | 13.85  | 1.42   | 32.95  | 4.61   | 59.99  | 1.74   | 50.04  | 0.52   | 44.35  |
| With Biochar                         |        |        |        |        |        |        |        |        |        |        |
| No Catalyst (ceramic supports)       | 2.30   | 28.48  | 1.41   | 27.94  | 1.55   | 32.13  | 0.89   | 30.77  | 0.41   | 23.94  |
| 2%NiO/10%CaO/Ceramic ring            | 2.96   | 50.20  | 3.70   | 66.40  | 1.46   | 48.42  | 0.75   | 42.54  | 0.68   | 33.84  |
| 4%NiO/10%CaO/Ceramic ring            | 1.27   | 21.07  | 1.64   | 29.69  | 1.14   | 27.64  | 0.66   | 29.68  | 0.77   | 24.37  |
| 8%NiO/10%CaO/Ceramic ring            | 2.84   | 43.65  | 3.27   | 39.48  | 2.37   | 30.91  | 2.43   | 37.47  | 1.40   | 32.43  |

#### 3.5. Adsorption of tar

To enhance tar removal efficiency, a filter packed with 1 kg of 50:50 (durian/mangosteen) biochar was connected to the system to trap volatile tar compounds prior the gas exit. Table 5 shows the amount of condensed tar in the filter unit after 60 min of the gasification process of 1 kg kernels. Without biochar filter, little amount of the tar approximately 0.2 kg was condensed and coated onto the internal surface of the filter unit. However, the amount of tar trapped in the filter having biochar was observed to be increased from 2 to 9 times for 0%, 2%, 4%, and 8% NiO content in the catalysts. The results were in good agreement with the study by Kuchonthara’s study [2], in which the NiO/Al\textsubscript{2}O\textsubscript{3} catalysts were used to improve the catalytic reforming performance of the gasification process of rice husks that resulted in a significant amount of tar reduction. When compared to the system with filter units filled with rice husks in Phanpruk’s study [6], it was found that the tar removal efficiency of the biochar filter was significantly improved by almost 100 percent. This may result from the superior adsorption performance due to binodal pore size distribution of the mixed biochar from both durian and mangosteen peels capable of broader adsorption range of aromatic hydrocarbon compounds containing in tar. Similar observation was also reported elsewhere [17].

### Table 5. Amount of tar remaining in Tar Filter Unit.

| Sample                               | Adsorbed tar (kg) per 1 kg of PKS |
|--------------------------------------|----------------------------------|
|                                      | Without Biochar | With Biochar |
| No Catalyst (ceramic supports)       | 0.02              | 0.04          |
| 2%NiO/10%CaO/Ceramic ring            | 0.02              | 0.03          |
| 4%NiO/10%CaO/Ceramic ring            | 0.02              | 0.12          |
| 8%NiO/10%CaO/Ceramic ring            | 0.02              | 0.18          |

#### 4. Conclusion

With the conjugation of using both co-impregnation of NiO/CaO and biochar, quality of syngas was distinctly improved in term of the amount of adsorbed tar from 2 to 9 times. Increasing in NiO contents tended to increase maximum \(\text{CH}_4\) composition of syngas for both with and without the use of biochar.
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