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H₂-Rich and Tar-Free Downstream Gasification Reaction of EFB by Using the Malaysian Dolomite as a Secondary Catalyst

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Abstract: In this study, Malaysian dolomites as secondary catalysts are placed at the downstream of the fluidized-bed gasifier. Three types of Malaysian dolomites with different elemental ratios of CaO-MgO content denoted as P1, P2, and P3 are investigated with EFB gasification reaction at different cracking temperatures (700–900 °C). The performance of the catalysts with a variation of catalyst to biomass weight ratio (C/B) (0.05 to 0.30 *w/w*) is evaluated. The findings showed that the total gas yield increased by 20%, hydrogen increased by 66%, along with an almost 99% reduction in tar content with P1 catalyst with the following reaction conditions: gasification temperature of 850 °C, equivalence ratio (ER) of 0.25, and cracking temperature of 900 °C. Malaysia dolomite could be a secondary catalyst to provide a better alternative, tar-free hydrogen-rich gas with the possibility of regeneration and re-use.

Keywords: empty fruit bunch; gasification; dolomite; hydrogen fuel; tar cracking

1. Introduction

As the world's leading energy source, fossil fuels have resulted in economic instability, unacceptably high levels of GHG emissions, and unprecedented anthropogenic climate change [1]. Currently, fossil fuels account for 80% of the energy demand which has led to fossil fuel depletion [2]. This has urged research to explore renewable sources as alternative sources of energy. In this case, hydrogen, a clean [3] and efficient energy form [4] is expected to reduce oil dependency. Hydrogen production not only satisfies the increasing energy demand but is also economically viable and technologically sustainable with similar technology employed without any significant change to replace fossil fuels [5,6]. Besides, since it can be stored as gas or liquid, hydrogen is reasonably safe and adaptable for fueling car engines [7] and fuel for fuel cells, a highly efficient clean power production technology through electrochemical reactions [8].

There are a variety of processes for hydrogen production such as combustion, pyrolysis, liquefaction, and biomass gasification [9]. According to Tian et al. [10], hydrogen production through biomass combustion is not sustainable due to its low heating value (LHV), corrosion, intolerably high moisture content, wide particle size distribution, and low homogeneity [10]. Meanwhile, the liquefaction process has difficulties in determining the operating conditions and very low hydrogen yield, whilst pyrolysis reaction is more

favorable to produce liquid (bio-oil) and solid products (biochar) instead of a gaseous product. Compared to other thermochemical processes route, gasification is a promising route for hydrogen production for organic materials. Many reported works on biomass gasification have been conducted using different operating conditions such as gasifier types (fixed bed, moving bed and fluidized bed), gasification agents (air, oxygen, steam, or their mixtures), and operating parameters (temperature, pressure, equivalent ratio). Furthermore, gasification is known to be highly efficient and clean that is capable of converting organic material into syngas [11].

The gasification of biomass for hydrogen production is promising for a sustainable future since biomass is a cheap and abundant carbon-neutral energy source with good synergy with existing conventional fossil fuel power plants [1]. The overall reaction of biomass gasification is rather complex is expressed in detail in Figure 1.

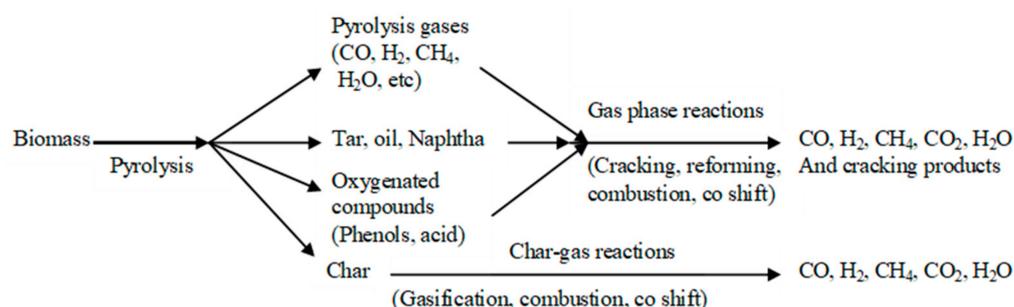


Figure 1. Reaction sequence for gasification of biomass [12].

Malaysia, the second-largest palm oil producer, produce approximately 54.1 Mt of waste from the palm oil industry annually [13–15]. The biomass part of the wastes comprises of empty fruit bunch (EFB), palm kernel shell (PKS), palm oil fronds (POF), trunks, and fiber [16,17]. To utilize these wastes and ensure sustainable energy development, the Malaysian government launched the National Biofuel policy in 2006, which sparks interest in biofuel production from these waste products [7,18]. To date, reports on biomass transformation through gasification are widely available in the literature [10,19–22]. The main drawback of this process with biomass as feedstock is the low H_2/CO ratio. Low conversion efficiency in the downstream products causes a low yield of syngas accompanied by the high amount of tar. Referring to Tian et al. [10], the high amount of tar produced may lead to several operational issues in downstream equipment such as corrosion and blockage in the pipeline due to tar condensation, reduce overall efficiency, and high cost for maintenance on the filtration system. Several works have reported on various methods and parameter optimizations to achieve hydrogen-rich syngas with high syngas to tar yield ratio and low impurity content [23].

At present, the research interest with biomass gasification has narrowed into producing a tar-free product gaseous. Tar is a complex mixture of condensable hydrocarbon (HCs) with 1-5-ring aromatic compounds, other oxygen-containing HCs and complex polyaromatic HCs (PAH) [24]. Tar can be eliminated by non-catalytic (collection and disposal method), catalytic (catalytic tar conversion), or by biomass-coal co-gasification [25]. Employing a catalyst has been found to enhance the gasification process by augmenting the amount of hydrogen, reducing the gasification temperature, and converting liquid tar into gases [5]. Hence, with higher desired product yield, the use of catalyst is economically advantageous. The only issues of catalyst are in the regeneration and recovery of the catalyst due to deactivation because of carbon fouling and ash sintering. Catalysts for use in biomass conversion should be effective in the removal of tar, resistant to activation, easily regenerated, and inexpensive. Additionally, to enhance the economic viability of EFB gasification, it is necessary to minimize tar content in product gas by a suitable low-cost catalytic hot gas cleaning. The purpose of catalyst in present work is both for purification and composition adjustment towards hydrogen-rich syngas.

Dolomite is a low cost and abundantly [26] available tar cracking catalyst and an efficient heat carrier in the gasification process [27]. Dolomites from different sources have different chemical compositions, surface area, and pore size, and hence different impacts on the gasification process, as reported by Quitete and Souza, [28]. From their observation, dolomites were significantly active above 800 °C and able to retain a small amount of H₂S and HCl that enhance tar cracking reaction. Our previous work [25] reported on Malaysian dolomite studied from three different sources as a primary catalyst in the gasification reaction of EFB. The results showed an approximately 78% and 75% significant tar reduction for P1 and P2 which has high content of CaO and MgO, respectively. However, the tar yields recorded in the previous work were still above 10.0 mg/g for P1, P2, and P3 catalysts. Herein, this research aimed to examine the tar cracking efficiency using P1, P2, and P3 as a secondary catalyst for the downstream gasification reaction of EFB. In addition, the effect of several factors such as the ratio of catalyst to biomass (C/B), gasification reaction performance and tar conversion were evaluated in this work.

2. Materials and Methods

2.1. Materials

Dried EFB was used as the feedstock with a diameter of 0.3–0.5 mm. A fixed bed gasifier with silica sand (0.10–0.15 mm) was used as the bed material. Dolomites from three different origins with different elemental composition [29] denoted as P1, P2, and P3 were employed as a catalyst in the gasification reaction in the downstream chamber. The catalysts' main chemical compositions evaluated by XRF and the pore characterization after calcined evaluated by BET are displayed in Table 1. For an in depth evaluation of the effect of preparation and characterization evaluation of these catalysts, refer to our previous study [29]. These catalysts were ground (0.5–1.0 mm diameter), sieved, and reactivated at 900–1000 °C with an absence of oxygen (with N₂ purging) for 4 h prior to the gasification reaction. Solid-phase extraction (SPE) tubes (VARIAN Ltd., Palo Alto, CA, USA) used for the tar sampling and sample preparation were loaded with 500 mg of aminopropylsilane (NH₂) phase (surface area~460–520 m²/g) bonded to silica gel (average particle size of 47–60 µm and average pore diameter of 60–87 Å).

Table 1. Chemical composition and pore characterization of the catalysts (extracted from [29]).

	P1	P2	P3
Chemical composition (%)			
CaO	30.0	32.0	23.0
MgO	21.0	21.0	17.0
SiO ₂	0.07	0.09	15.37
Al ₂ O ₃	0.04	0.08	1.69
Fe ₂ O ₃	0.07	0.12	0.51
Pore characteristics			
BET surface area (m ² /g)	15.25	16.85	6.16
Pore volume (cm ³ /g)	0.32	0.104	0.09
Average pore diameter (Å)	416	233	215

2.2. Downstream Catalytic Gasification Reaction

The secondary catalysis experiments took place in a downstream fixed-bed gasification reactor as shown in Figure 2. First, 30.0 g of the catalyst was placed in the second fixed-bed reactor, and then inserted into the furnace which electrically heated at 10 °C/min to the desired cracking temperature. The effect of the secondary catalyst was investigated at different cracking temperature varied from 700 up to 900 °C while the gasifier temperature was kept at 850 °C. The catalytic system inserted between the fluidized-bed gasifier and cooling system as illustrated in Figure 2. The results obtained were compared with previously reported work [25].

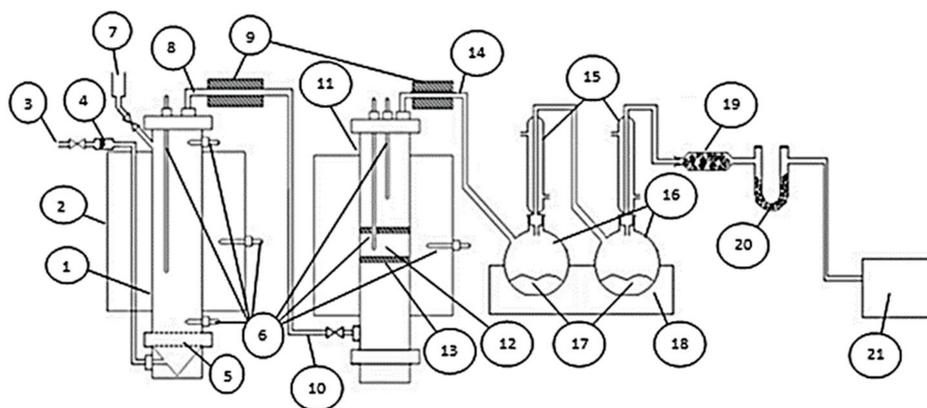


Figure 2. A schematic diagram of the secondary catalytic gasification setup: (1) Primary gasifier; (2) electric furnace; (3) Gas inlet; (4) Gas flow meter; (5) Gas distributor; (6) Thermocouples; (7) Feeder; (8) Gas outlet; (9) Heating tape; (10) Gas inlet; (11) Secondary gasifier (12) Secondary catalyst; (13) Catalyst holder; (14) Gas outlet; (15) Condenser; (16) Neck flask; (17) Dichloromethane; (18) Ice path; (19) Fiber-glass wool filter; (20) Silica gel; (21) Gas bag.

2.3. Catalyst Deactivation-Regeneration

A series of experiments were performed to study the Malaysian dolomite deactivation and aging. The dolomite was activated (calcined) prior to use in the cracking analysis. Then, the output gas from fluidized bed gasifier (at gasification temperature of 850 °C and ER = 0.25) was passed over catalyst bed in downstream fixed bed reactor (at cracking temperature of 900 °C) for sixty minutes. After cracking test finished, the cracking reactor was disconnected from gasifier and catalyst was regenerated by calcining in air (100 mL/min) at 1000 °C for 4 h. Then, the cracking reactor was reconnected again to the gasifier and another cracking test was performed for sixty minutes. This procedure was repeated twice.

2.4. Product Analysis

The gaseous product collected in the gas bag and analyzed with gas chromatography with a thermal conductive detector (GC-TCD). The solid-phase adsorption (SPA) method was used to carry out the sampling and analysis of tar compounds. In this paper, the tar referred to major GC detectable aromatic and phenolic compounds. Detailed methodology for both products (gaseous product and tar) can be referred to the earlier published work [25].

3. Results and Discussion

3.1. Catalytic Activity in Downstream EFB Gasification Reaction

The catalytic activity of Malaysian dolomites was investigated at different catalytic bed temperatures (cracking reaction temperatures). From the result in Figure 3, as the temperature in the catalytic bed increased from 700 to 900 °C, the gas yield increased, whereas the total tar yield decreased significantly. The secondary dolomites (P1, P2, and P3) show a marked effect on gas yield and tar destruction. P1 dolomite revealed a stronger catalytic effect compared to P2 and P3 dolomites. As the temperature increased, the gas yield obtained with the aid from P1 dolomite in the cracking reactor rose by 23.6% (from 1.496 to 1.732 Nm³/kg), whereas, for P2 and P3, the gas yield increased by ~22.4 and 17.6%, respectively. Simultaneously, the total tar yield decreased significantly with increasing of catalytic bed temperature to reach less than 0.08 wt.% at cracking temperature of 900 °C for P1 dolomite and 0.10 and 0.16 wt.% for P2 and P3, respectively. The initial tar produced in the non-catalytic gasification process at a gasification temperature of 850 °C was 4.12%. Bio-oil was undetected due to the thermal and catalytic effect of the secondary reactor. In the meantime, the tar yield was not affected by the secondary catalyst because only the gaseous products from the gasifier were passed through the catalytic bed in the secondary reactor.

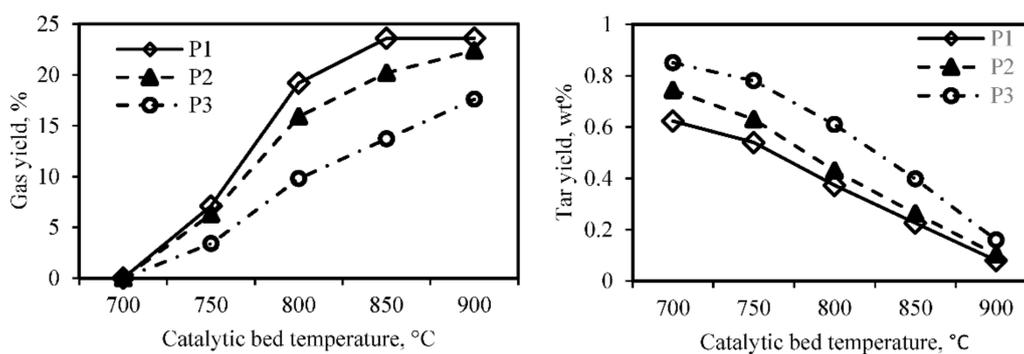


Figure 3. Effect of different catalyst to biomass (C/B) ratio on overall gas product composition.

Meanwhile, varying the catalytic bed temperature shows a marked change in produced gas components. As shown in Figure 4, the contents of H_2 , CO , and CH_4 were increased as a result of the occurrence of tar reforming reaction (Equation (1)) and water-gas shift reaction [7], while the content of CO_2 was decreased due to most of CO component was consumed in the tar conversion reactions, in addition to carbonation reaction, where CaO reacted with CO_2 , to produce $CaCO_3$. Dolomites had a pronounced effect of favoring the production of H_2 and CO at higher cracking temperature whereas the CO_2 content decreased and CH_4 content slightly increased. The highest H_2 content (45.88 vol.%) was obtained with P1 dolomite as a secondary catalyst at cracking temperature of 900 °C, whereas for P2 and P3 the H_2 content was 45.35 and 40.3 vol.%, respectively, at the same catalytic conditions.

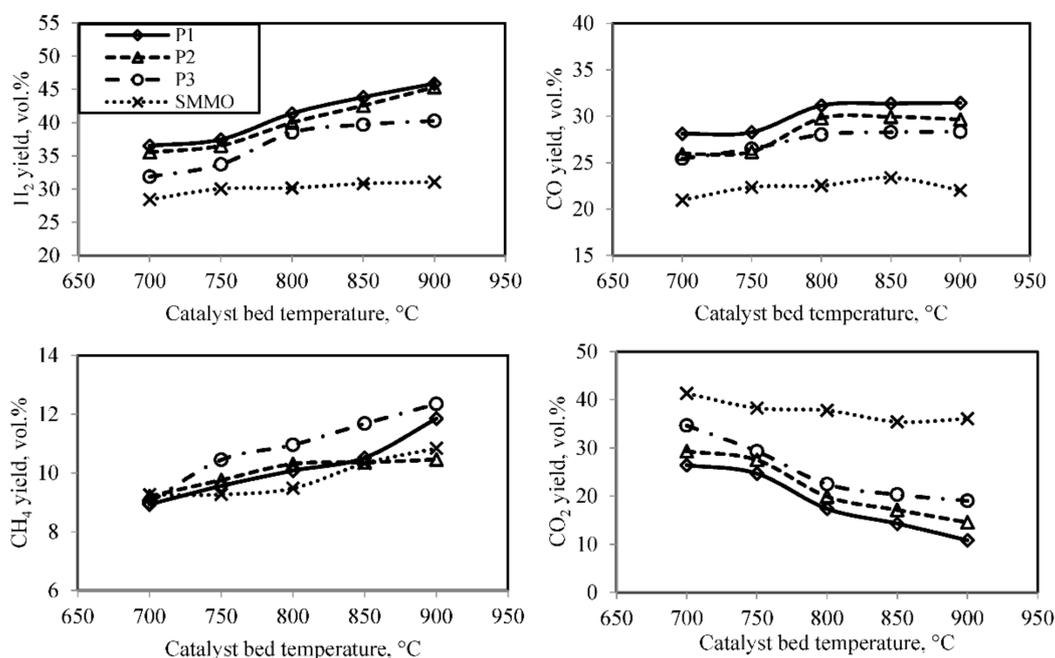


Figure 4. Effect of different catalyst to biomass (C/B) ratio on syngas product composition.

3.2. The Influence of Secondary Dolomite Catalysts on Gasification Performance

The presence of dolomites as a secondary catalyst leads to an increase in the total gas yield, in addition to enhance the evolution of combustible gases (i.e., H_2 , CO , and CH_4) which in turn results in an increase in higher heating value (HHV) and cold gas efficiency of the produced gas, as illustrated in Figure 5.

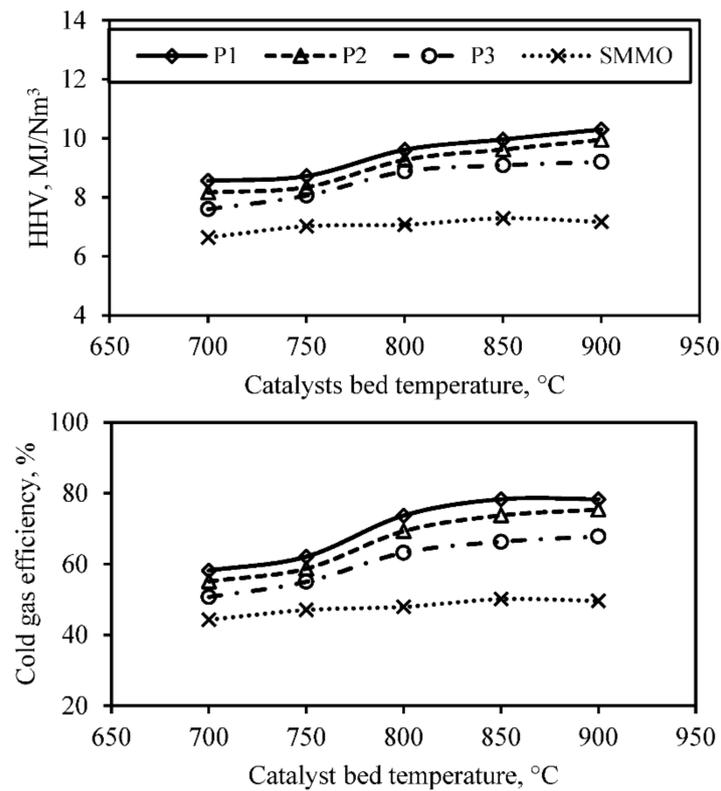


Figure 5. Effect of different catalyst to biomass (C/B) ratio on gasification performance.

In case of P1 dolomite, as the temperature of the catalytic reactor increased from 700 to 900 °C, the HHV and cold gas efficiency of produced gas increased from 8.56 to 10.30 MJ/Nm³ and 58.16–81.03% respectively, compared to 8.20 MJ/Nm³ and 54.47% for the non-catalytic gasification process. Meanwhile, for the P2 and P3 dolomites, the HHV of the produced gas increased to 9.95 and 9.20 MJ/Nm³ respectively, under the same catalytic conditions. As shown in Figure 4, at the same gasification and cracking conditions, P1 dolomite as a secondary catalyst shows higher catalytic activity than P2 and P3 dolomites in terms of the total gas production and selectivity for H₂ production. A comparison on the effect of secondary catalyst with non-catalytic gasification process in terms of product yields and performance is shown in Table 2.

Table 2. Experimental results of empty fruit bunch (EFB) gasification reaction with and without Malaysian Dolomite catalysts.

Catalyst	Without Catalyst [29]	Primary Catalyst [25]		Secondary Catalyst (Present Work)	
		P1	P1	P2	P3
Gasifier Temperature (°C)	850	850	850	850	850
ER	0.25	0.25	0.25	0.25	0.25
EFB Particle size (mm)	0.3–0.5	0.3–0.5	0.3–0.5	0.3–0.5	0.3–0.5
Temperature catalyst bed (°C)	900	900	900	900	900
Gas yield (Nm ³ /kg)	1.46	1.57	1.75	1.71	1.65
Tar yield (mg/g)	41.20	8.80	0.08	0.10	0.16
Gas composition (vol.%)					
H ₂	27.31	36.87	45.88	45.35	40.29
CO	33.08	36.56	31.45	29.66	28.33
CH ₄	13.79	16.62	11.85	10.46	12.35
CO ₂	25.63	9.93	10.82	14.53	19.03
Gas HHV (MJ/Nm ³)	8.20	9.96	10.30	9.95	9.20
Hydrogen yield (g H ₂ /kg EFB)	21.55	31.10	37.70	37.26	32.80

3.3. The Influence of the Secondary Catalyst on Tar Cracking and Conversion Efficiency

Figure 6a shows the total tar content in produced gas derived from EFB gasification at 850 °C was strongly reduced after the catalytic reaction. The calculated tar conversion efficiency (%) for P1, P2, and P3 were ~99%, 97%, and 94%, respectively. The P1 dolomite shows the highest reduction in tar content at cracking temperature of 900 °C, whereas P2 and P3 dolomites show the lower effect on the tar destruction under the same catalytic conditions. This can be explained by the fact that most of the thermally stable tar compounds are converted at a relatively high temperature between 800–900 °C [30]. The tar conversion resulting from EFB gasification over the secondary catalysts at different cracking temperatures is shown in Figure 6b.

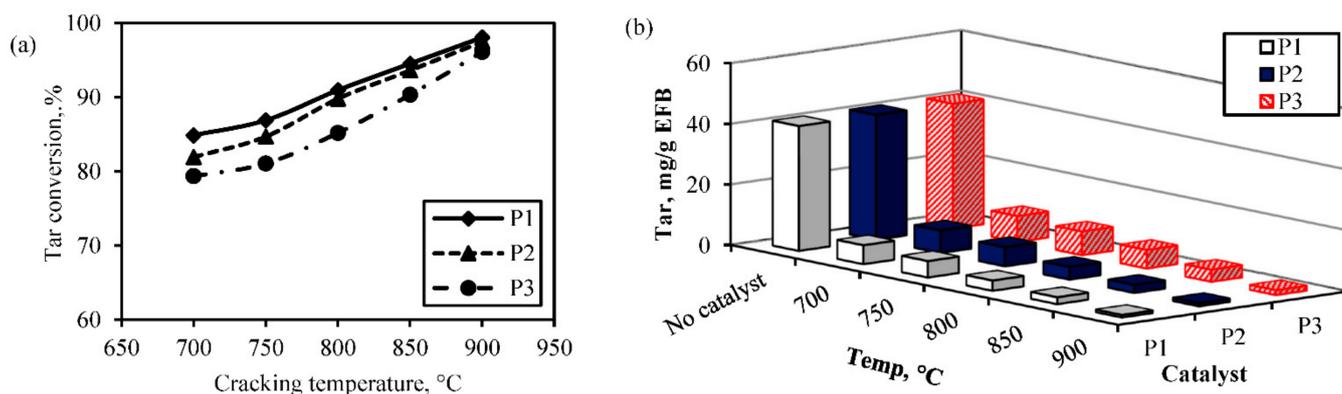


Figure 6. Effect of different catalyst to biomass (C/B) ratio on (a) tar conversion (b) tar yield.

The SPA analysis indicates that all phenolic compounds and most of aromatics (except naphthalene) compounds were not detected when P1 and P2 dolomites were used as a secondary catalyst as illustrated in Figure 7a. Whereas in case of P3 dolomite, in addition to naphthalene, trace amounts of benzene and polyaromatic compounds such as 1, 2-methylnaphthalene, naphthalene, fluorine, phenanthrene, and pyrene were detected under cracking temperatures of 700 and 800 °C and then disappeared after cracking temperature of 800 °C.

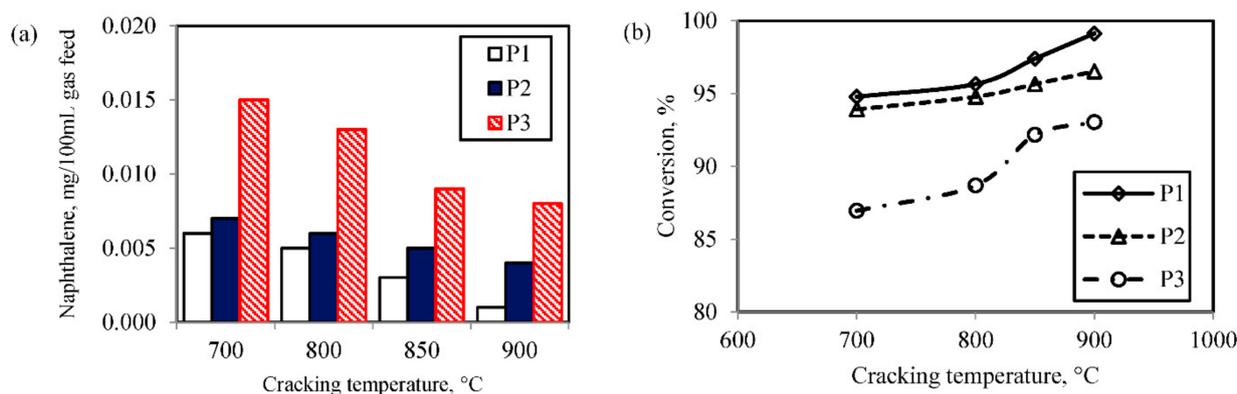


Figure 7. Effect of different catalyst to biomass (C/B) ratio on (a) naphthalene concentration (b) naphthalene conversion.

The conversion of naphthalene over different types of Malaysian dolomites is shown in Figure 7b. It can be observed that the dolomites presented high catalytic activity for naphthalene conversion; the cracking activity was increased with increasing of cracking temperature. At a cracking temperature of 900 °C, P1 dolomite showed highest naphthalene conversion efficiency compared to P2 and P3 dolomites. As shown in Figure 7b, the naphthalene conversion efficiency was ~99%, 97%, and 92% for P1, P2, and P3 dolomites, respectively.

3.4. Dolomite Deactivation-Regeneration

The performances of the dolomites are monitored closely for signs of attrition, deactivation, and ageing. These factors affect the number of re-uses and regeneration cycles that dolomite can undergo. Several research groups reported that the calcined dolomite catalyst deactivated due to carbon deposition and attrition [31]. However, dolomite is cheap and easily replaced. The deactivation of the calcined dolomite is mainly by coke formation since no sulfur or chlorine contents are detectable in produced gas from biomass gasification. Coke can be produced in this process by several simultaneous reactions as shown in equation (Equation (1)), with several factors influencing it, such as high temperatures, basic oxides in the catalyst, tar composition, etc. The produced coke covers active sites and blocks the pores, thus deactivate the calcined dolomite [24].



In this study, the performance of dolomite was explored by examining the P1 dolomite performance after multiple regenerations. The P1 dolomite was selected due to the highest cracking efficiency compared to other types of dolomites used in the catalytic gasification process. Figure 8 shows the H₂ production, total tar yield and tar conversion efficiency of P1 dolomite in the multiple-use and regeneration process. The high activity of P1 dolomite as reported in the previous section as a secondary catalyst is shown to deactivate after each use/regeneration cycle due to coke deposition on the dolomite's surface. The H₂ yield decreased whereas total tar yield increased for every cycle of the regeneration and gasification process. The tar conversion efficiency of P1 dolomite decreased from ~97% at the end of the first (original) cycle to ~92% after the third re-use/regeneration cycle performed in this work.

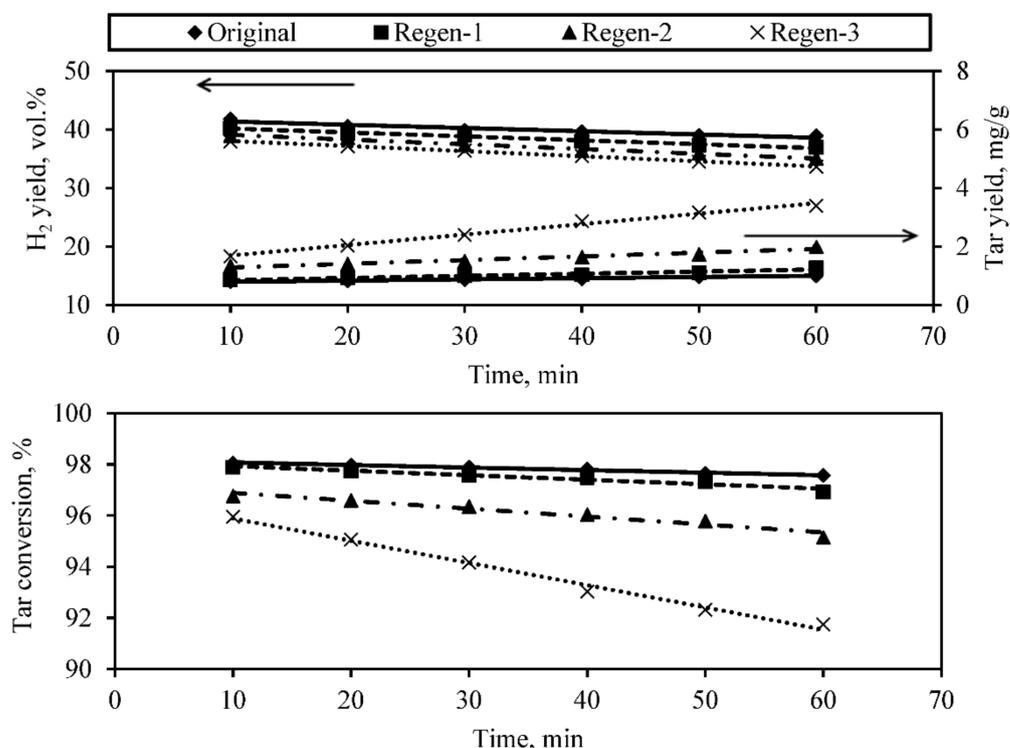


Figure 8. Secondary catalyst performance under multiple re-use/regeneration cycles.

4. Conclusions

In this work, compared to the other Malaysian dolomite catalysts, P1 was the best catalyst in the primary gasification reaction due to the higher surface and pore volume. P1 Malaysian dolomite was proven to be not only the best catalyst in the primary gasification, but also showed a good catalytic activity in the downstream gasification reaction by a 20% increment in gas yield, 99% tar degradation, and ~66% H₂ increment for the following conditions: gasification temperature of 850 °C, equivalence ratio (ER) of 0.25, and cracking temperature of 900 °C. In all cases, significant tar cracking was obtained in the EFB downstream gasification reactor using all the Malaysian dolomites as secondary catalysts. The tar yield values were significantly reduced into 0.08, 0.10, 0.16 mg/g for P1, P2, and P3, respectively as compared to primary catalytic gasification reaction (>10.00 mg/g). Moreover, all phenolic and most of the aromatic compounds in tar were significantly eliminated in this reaction. The naphthalene conversion efficiency was ~99%, 97%, and 92% for P1, P2, and P3 dolomite, respectively.

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Conflicts of Interest: The authors declare no conflict of interest.

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