

# **Hydrogen Production from Catalytic Gasification of Waste Wood Polymer Composite Using Different Low-Cost Catalysts**

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## **Abstract**

Wood polymer composites (WPC) are commonly used as raw materials for flooring and exterior wall materials because of their mechanical strength and environmental friendliness compared to general wood. WPC can be reused many times, but when it loses its durability, it is discharged into solid waste. Hence, looking for efficient and eco-friendly processes to treat WPC is necessary. Notably, WPC can be converted to valuable syngas through thermochemical processes. In this study, The potential of low-cost catalysts (i.e. olivine, dolomite, and red mud) for H<sub>2</sub> generation through air gasification of WPC was investigated. The effects of gasification temperature, Ni loading, catalyst/feedstock (C/F) ratio, and equivalence ratio (ER) were also evaluated. When the temperature was varied (700, 750, 800°C), the highest H<sub>2</sub> selectivity was obtained at 800 °C for all catalysts, and particularly, H<sub>2</sub> selectivity reached a maximum (48.93 vol%) employing red mud. Further, loading 10 wt.% Ni on all catalysts enhanced the gas yield and the volume percentages of H<sub>2</sub> and CO, where red mud with Ni at 10 wt% showed the highest H<sub>2</sub> selectivity at 56.00 vol%. Increasing ER in the range of 0.2 to 0.3 decreased the H<sub>2</sub> selectivity while promoting the gas yield and CO<sub>2</sub> production. Based on these experimental results, air gasification using red mud catalysts can provide a new technology to convert WPC into a more useful and high-energy density substance such as H<sub>2</sub>.

**Keyword**Air Gasification, Hydrogen generation, Dolomite, Olivine, Red Mud, Wood Polymer Composite

## ***1. Introduction***

Wood polymer composites (WPC) are mainly used as materials for flooring and exterior walls due to their mechanical strength and environmental friendliness compared to general wood. WPC can be reused many times, but it has a limitation in that its mechanical properties gradually decrease due to the decomposition of polymers, making it difficult to recycle [1]. According to the Environmental Protection Agency (EPA), the amount of municipal solid waste (MSW) has been increasing in the past few decades, and WPC is a part of MSW, so it is necessary to find efficient and environmentally friendly processes for treating WPC. Interestingly, such WPCs can be converted to valuable syngas through thermochemical processes such as gasification, pyrolysis, and combustion. Furthermore, using catalysts can increase the production of useful gases such as hydrogen ( $H_2$ ) and carbon monoxide (CO) while suppressing the formation of tar during these processes.

Thermo chemical processes are considered a promising technology for producing sustainable energy because they produce less emissions and have high energy efficiency while converting waste into valuable energy sources [2]. In particular, gasification is considered as a more efficient technology for  $H_2$  production owing to its relatively low emission of pollutants and high power and heat generation efficiency, producing high-quality synthesis gases such as  $H_2$  and CO [3]. However, downstream applications of gasification have encountered various problems such as engine and turbine clogging and contamination, cracking of filter pores, low-temperature gas efficiency, the reduced heat value of synthesis gas, and coking and catalyst deactivation [4]. Catalyst application has been proposed as an effective approach to address these important issues in gasification process, enabling effective minimization of tar and enrichment of  $H_2$  proportion in the produced gas [5]. Tar can be effectively converted into hydrocarbons and useful gas products through catalytic cracking. Catalytic treatment is considered the best option for treating syngas and tar elimination [6]. However, the cost of catalyst application remains a major drawback. This problem has been widely explored by numerous researchers over the past 30 years [7]. In this context, using natural minerals (e.g. dolomite and olivine) or waste materials (e.g. red mud) as low-cost catalysts in the gasification process has been growing in popularity in recent years.

Dolomite is a naturally occurring mineral, widely used as a catalyst in tar conversion during gasification. Its general formula is  $MgCO_3 \cdot CaCO_3$ . Generally, Dolomite has the dual functions of tar cracking and  $CO_2$  adsorption during gasification reactions, promoting  $H_2$  generation. Typically, dolomite is calcined prior to use in gasification

which leads to the creation of the CaO–MgO complex. Importantly, only CaO (not MgO) undergoes a carbonation reaction ( $CO_2 + CaO \leftrightarrow CaCO_3$ ) that declines  $CO_2$  content along with enhancing  $H_2$  selectivity [8]. Moreover, the CaO–MgO complex is capable to increase the cleavage of C–C and C–H bonds, stimulating the water-gas shift (WGS) reaction, and adjusting the distribution of various products in the gasification process [9].

Red Mud (RM) is a bulk solid waste generated during the industrial production of alumina. RM has a high surface area, porosity, and reactivity due to its composition including active components ( $Fe_2O_3$ ), inactive carriers ( $Al_2O_3$ ,  $SiO_2$ ), and metal additives ( $Na_2O$ ,  $CaO$ ), which makes it a promising catalyst for gasification reactions. According to research, RM has a specific promoting effect on the WGS and tar steam reforming reactions [10]. Notably, due to its stability, and prevention of sintering and poisoning, RM would be promising catalyst support [11], and its combination with nickel (Ni) can produce an economic and stable catalyst with high decomposition activity, improving the vulnerability of Ni-based catalysts to sintering and poisoning [12]. Overall, The use of red mud as a catalyst for  $H_2$  generation through gasification has several potential benefits, such as reducing the cost of catalyst materials and improving the efficiency and environmental performance of the gasification process.

Olivine with the general formula of  $(Mg,Fe)_2SiO_4$  is a mineral frequently used in gasification to remove tar due to its high catalytic activity and mechanical strength. Previously, the catalytic activity of Olivine was found to be due to the iron content [14]. Indeed, the various oxidation states of Fe, in particular  $Fe^{II}$ , are participated as active sites for augmenting the catalytic activity of olivine for tars decomposition and the WGS reaction. Moreover, the catalytic effect of olivine for increasing gas yield, slightly enhancing  $H_2$  and  $CO_2$  production while decreasing CO and  $CH_4$  content was previously reported. However, the catalytic activity of Olivine is still not sufficient for tar decomposition and  $CH_4$  reforming, leading to the development of NiO/Olivine catalysts [15].

Nickel (Ni) based catalysts have been considered as efficient catalysts in gasification process due to their ability to mitigate tar formation and obtain high activity and selectivity for  $H_2$  production [16], and have high potential for commercialization. The catalytic effect of metal Ni on the cleavage of C-H, O-H, and C-C bonds and intermediates in the gasification process helps to increase gas yield while sacrificing tar yield [17]. Furthermore, the high functionality of metal Ni has been proven to induce carbon-steam, Boudouard, WGS, SMR reactions with appropriate activity for dry reforming of methane (DMR) reaction. It has been approved that metallic Ni ( $Ni^0$ )

is mainly responsible for H<sub>2</sub> production in gasification process. [18].

In this study, air gasification of WPC (waste plastic composite) using dolomite, olivine, and RM was performed with the aim of producing bio-hydrogen through gasification experiments using low-cost catalysts. The effects of C/F ratio, nickel loading, temperature, and ER value were evaluated. This study is expected to provide a promising solution as a clean energy source by producing bio-hydrogen through the gasification of WPC, which is treated as solid waste.

## **2. Materials and methods**

### *2.1. Feedstock*

The wood-plastic composite (WPC) used in this study was a commercially available product manufactured by a domestic company. Applied WPC with the particle sizes of <250 μm was composed of wood components (58%), polyethylene (9%), polypropylene (18%), CaCO<sub>3</sub> (8.5%), and other additives (6.5%). Before performing the experiments, it was dried at 110 °C for 24 h. Proximate analysis using thermogravimetric analysis (TGA) showed that the WPC contained 86.6% volatile matter, 4.0% fixed carbon, 8.0% ash, and 1.4% moisture. According to ultimate analysis in the dry ash-free state, WPC contained of C (57.2%), H (11.6%), N (0.4%), and O (30.8%).

### *Catalysts preparation and characterization*

In this study, commercially available dolomite, olivine, and RM were used from a domestic manufacturer. Calcined dolomite and olivine were used and prepared by calcination at 800 °C for 3 h. RM was activated with KOH and the activated catalyst was prepared by calcination at 800 °C for 3 h. To produce Ni loaded catalyst, Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O was added to the powdered dolomite, olivine, and RM as a metal precursor via wet impregnation method to add 10wt% Ni. Then, it was dried at 110 °C for 8 h and calcined at 550 °C for 3 h. Finally, the obtained catalyst was reduced under H<sub>2</sub> conditions at 800 °C for 3 h.

The produced catalysts were characterized by Brunauer-Emmet-Teller (BET), X-ray diffraction (XRD), and H<sub>2</sub>-temperature-programmed reduction (H<sub>2</sub>-TPR) analysis. BET analysis was performed by the N<sub>2</sub> adsorption/desorption method at 77K after vacuum degassing at 200°C for more than 6 h, and XRD analysis was conducted to confirm the structure of the support and the prepared catalyst. The XRD analysis was performed

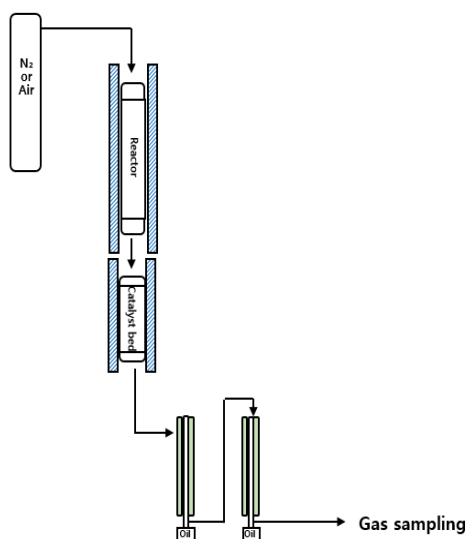
using Cu K $\alpha$  radiation at 40 kV and 40mA in the scanning angle of 5–90° at a scanning speed of 0.5° min<sup>-1</sup>. H<sub>2</sub>-TPR was performed using a chemisorption analyzer (Auto Chem II 2920, Micrometrics) to observe the reduction temperature distribution of the catalyst according to the temperature rise under H<sub>2</sub> gas and the amount of H<sub>2</sub> consumed to determine the reduction degree. It was pretreated for 1 h at 400°C under He gas and then reduced under H<sub>2</sub> gas at 800°C for 3 hours.

### 2.3. Air gasification

In this study, WPC gasification experiments were carried out using a lab-scale fixed bed reactor composed of a control unit, a gasification reaction unit, and a condensation and collection unit. The amount of gasifying agent supplied was controlled using a digital flow controller (KRO-4000) in the control unit. Air was used as the gasifying agent in this study, and the oxygen in the air was considered as an actual reactant in the gasification reaction, so the amount of oxygen required for complete combustion was calculated using the Equivalence ratio (ER) equation (eq.11).

$$ER = \frac{\text{Actual air}}{\text{stoichiometric air}} \quad (\text{eq.11})$$

In the gasification reaction unit, the upper electric part was used to heat the sample for gasification, and the lower electric part was used for catalytic upgrading. Finally, the condensers and gas trapping system in the condensation and collection unit were used to recover the products generated during the reaction. In a typical experiment, certain amounts of WPC and catalyst were located separately in the upper and bottom parts, and the gasification reaction was carried out for 15 min at 800 °C. The effects of varying ER values (0.2, 0.25, 0.3), catalyst/feedstock (C/F) ratio (0.1, 0.2), and temperature (700, 750, 800°C) on the production of H<sub>2</sub>-rich gas were evaluated. Additionally, the effect of Ni addition into catalysts on the yield of gas and H<sub>2</sub> selectivity was also examined.



**Fig1.** Diagram of the gasification reaction setup.

### 3. Results and discussion

#### 3.1. Catalyst characterization

##### 3.1.1. BET

Table 1 shows the specific surface area ( $S_{BET}$ ) and pore volume of calcined catalysts (dolomite, olivine, RM) obtained through BET analysis. The  $S_{BET}$  of calcined RM was  $30.09 \text{ m}^2/\text{g}$ , which is larger than the other two catalysts, indicating that it may have a positive effect on the catalytic activity due to its larger  $S_{BET}$ , pore volume.

**Table1.** BET surface area and Total pore volume analysis of catalysts

Catalyst	$S_{BET}$ ( $\text{m}^2/\text{g}$ )	Total pore volume ( $\text{cm}^3/\text{g}$ )
Dolomite	17.59	0.17
Olivine	0.69	0.002
RM	30.09	0.21

##### 3.1.2. XRD

The XRD patterns of Ni-loaded catalysts were provided (Fig. 2) to confirm the crystallinity of nickel metal

incorporated in each catalyst. The typical peaks of metallic Ni appeared at around 44.5°, 53°, and 76.5° in all catalysts [19]. It is suggested that the high concentration of Ni embedded has influenced the unique structure of each catalyst. Notably, the intensities of peaks relevant to metallic Ni are lower in the case of 10 wt.% Ni/Red mud and 10 wt.% Ni/dolomite compared to those of 10 wt.% Ni/olivine. This can suggest that the larger  $S_{BET}$  and pore size of red mud and dolomite compared to olivine was beneficial for the enhancement of Ni dispersion.

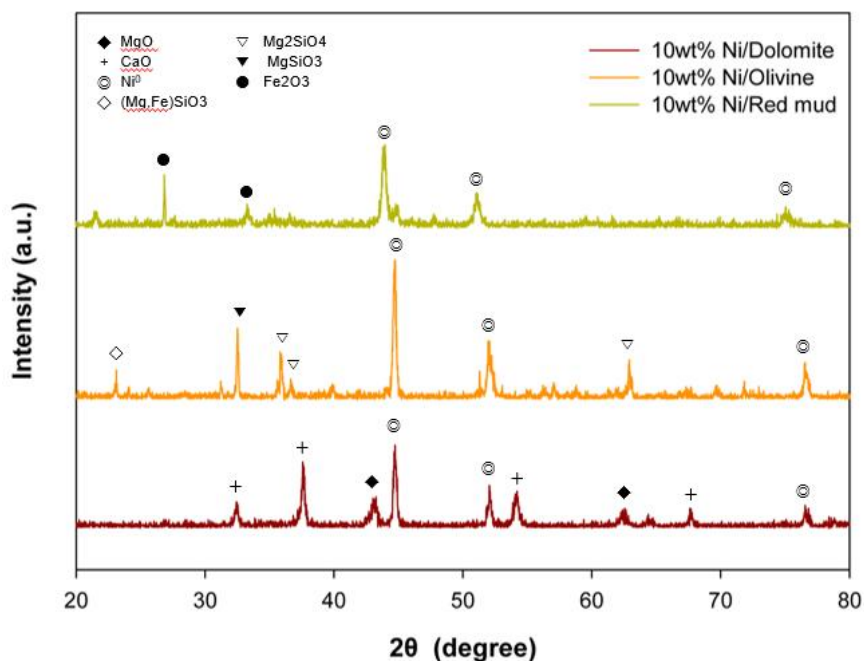
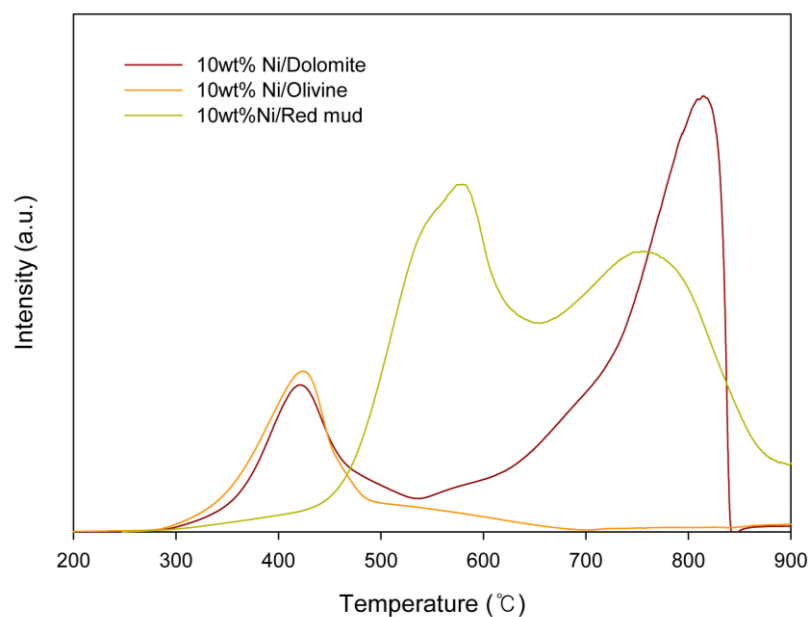


Fig. 2. XRD profile of different catalysts

### 3.1.3. $H_2$ -TPR

The  $H_2$ -TPR profiles of the produced catalysts are presented in Fig. 2. For dolomite and olivine, a low-temperature reduction peak appeared in the range of 330-500 °C, and for RM, this peak shifted to the higher temperature in the range of 500-600°C. Furthermore, while dolomite did not show any peak at high temperatures, RM and dolomite showed a high-temperature peak between 650-850°C. Typically, the peak in the moderate temperature range (400-600 °C) indicates the large volume of NiO located on the external surface of the support [20], while the peak above 600 °C can be attributable to the diffusion of small NiO into the catalyst pore and strong metal-support interaction. Thus, stronger metal-support interaction in the case of 10 wt.% Ni/Red mud and 10 wt.% Ni/Dolomite catalyst is indicative of appropriate dispersion of NiO inside the pores in these two catalysts. This is in agreement with the results of BET and XRD data.





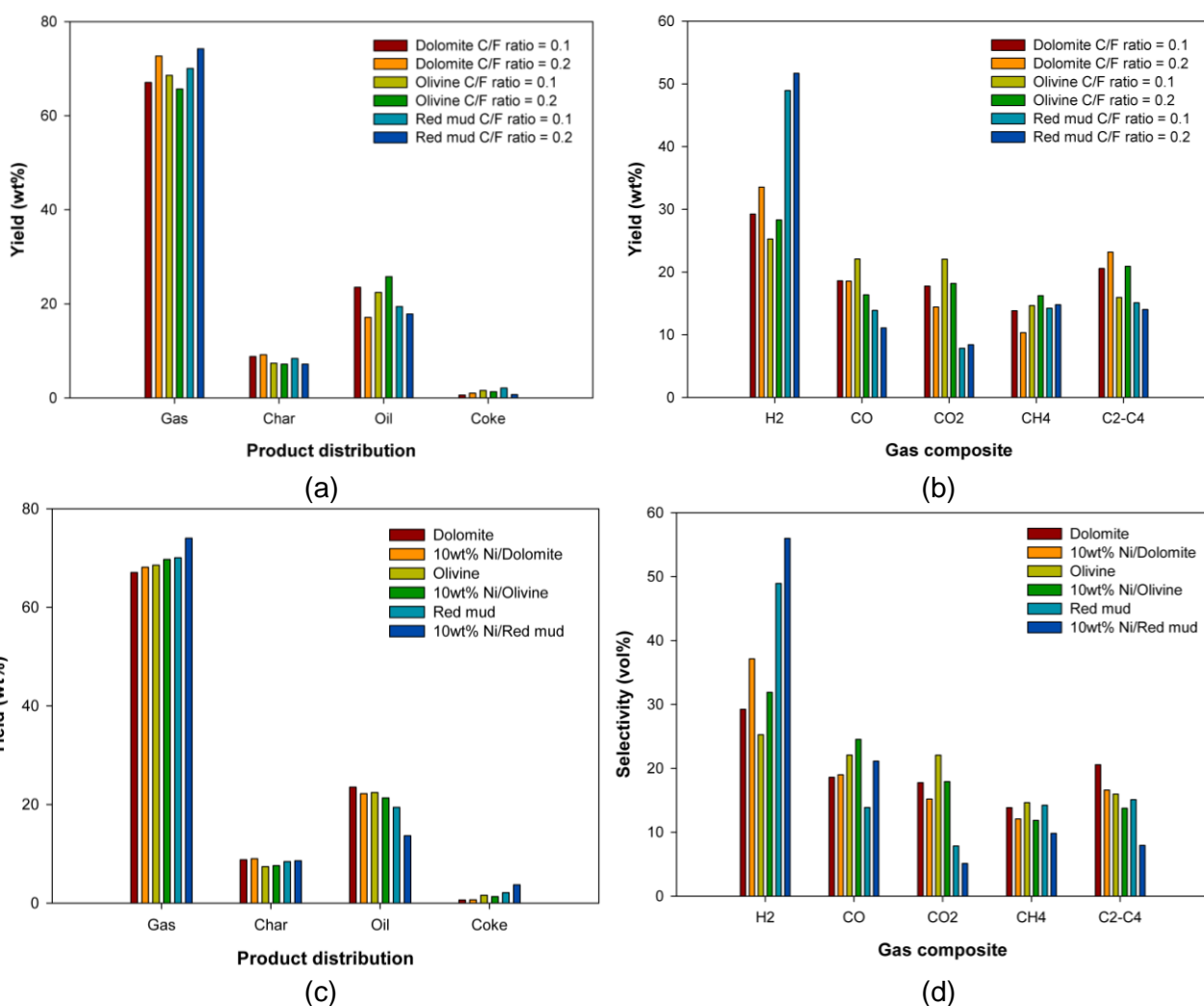
**Fig. 3.** H<sub>2</sub>-TPR profile of different catalysts.

### 3.2. The effect of catalysts

Fig. 4 (a-b), the H<sub>2</sub> production from the gasification of WPC with The catalytic effects of dolomite, olivine, and RM catalysts at C/F ratios of 0.1 and 0.2 were investigated, and the gasification temperature was kept constant. As shown in Fig. 4 (a), as the C/F ratio increased, the gas yield increased and the oil yield decreased. When the C/F ratio was 0.1, the highest gas was obtained using RM (70.04 wt.%), followed by olivine (68.57 wt.%) and dolomite (67.08 wt.%). Also, using RM obtained the lowest oil yield at 19.44 wt.%. In the case of a C/F ratio of 0.2, the gas yield increased compared to when the C/F ratio was 0.1, but the difference was minimal. According to Fig. 4 (b), as the C/F ratio increased from 0.1 to 0.2, the H<sub>2</sub> selectivity increased from 29.23, 25.26, and 48.93 vol% using dolomite, olivine, and red mud, respectively, to 33.53, 28.31, and 51.68 vol%, respectively. In contrast, CO and CO<sub>2</sub> generation showed a decent trend with increasing 0.1 to 0.2 using all catalysts. Overall, it was determined that the influence of the amount of catalyst (varying C/F ratio from 0.1 to 0.2) applied on the gas yield and H<sub>2</sub> selectivity was negligible, and subsequent experiments were conducted with a C/F ratio of 0.1.

Fig. 4 (c-d) indicates the product distribution and gas composition obtained using different supports and their respective Ni-loaded catalysts. As shown in Fig. 4 (c), the gas yield enhanced and the oil yield decreased as the

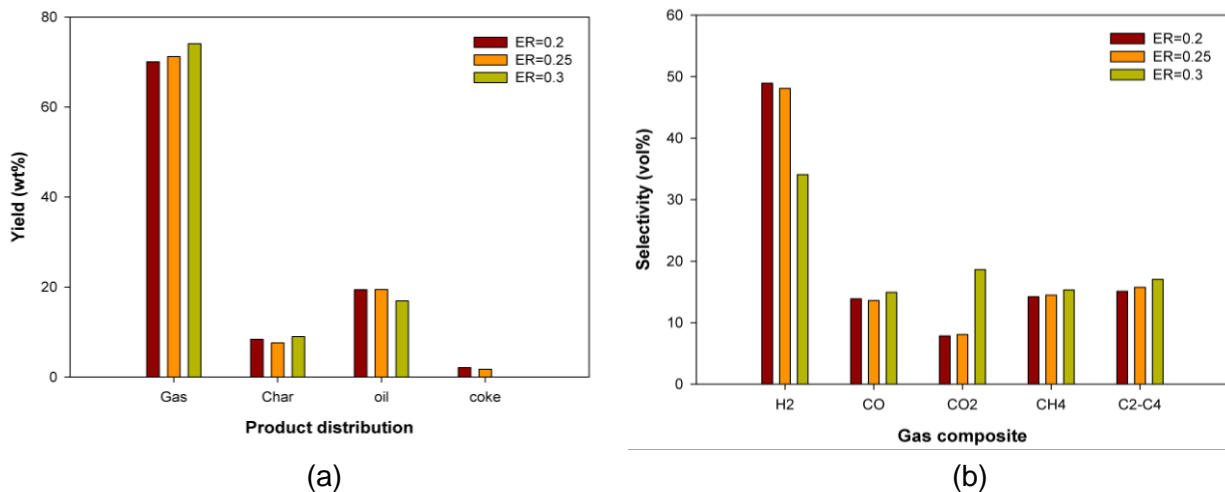
result of Ni (10 wt.%) addition in all tested catalysts. In particular, for olivine, the gas yield increased significantly from 68.57 wt.% to 76.72 wt.% after Ni loading, while the oil yield decreased from 22.43 wt.% to 14.35 wt.%. In Fig. 4 (d), it can be seen that the selectivity of H<sub>2</sub> increased as the result of Ni loading, with dolomite increasing from 29.23 to 37.13 vol%, olivine increasing from 25.26 to 31.90 vol%, and RM increasing from 48.93 to 56.00 vol%. Similarly, CO showed an increasing trend with Ni loading, while CO<sub>2</sub>, CH<sub>4</sub>, and C<sub>2</sub>-C<sub>4</sub> decreased in selectivity. Generally, catalyst activity tends to be proportional to the amount of available active sites, and metallic Ni is considered a major catalyst active site in Ni-based catalysts [25]. It was observed that loading 10 wt.% Ni onto red mud, dolomite, and olivine significantly increased the gas yield and H<sub>2</sub> selectivity, which is attributed to the promoted steam-carbon, WGS, and SMR reactions over active sites provided by the metallic Ni, resulting in increased hydrogen production [26]. In this study, the highest activity was observed when Ni was loaded onto Red mud. This can be ascribable to its higher S<sub>BET</sub> and pore volume (see Table 1) as well as more reducibility and dispersion of Ni particles in the case of 10 wt.% Ni/Red mud, compared to the other two catalysts, as evidenced by XRD and H<sub>2</sub>-TPR results.



**Fig. 4.** Product distribution and gas composition obtained from catalytic air gasification of WPC, using Dolomite, Olivine, RM catalysts at different C/F ratios (a-b), and Ni loading (c-d).

### 3.2. The effect of different Equivalence Ratios (ER).

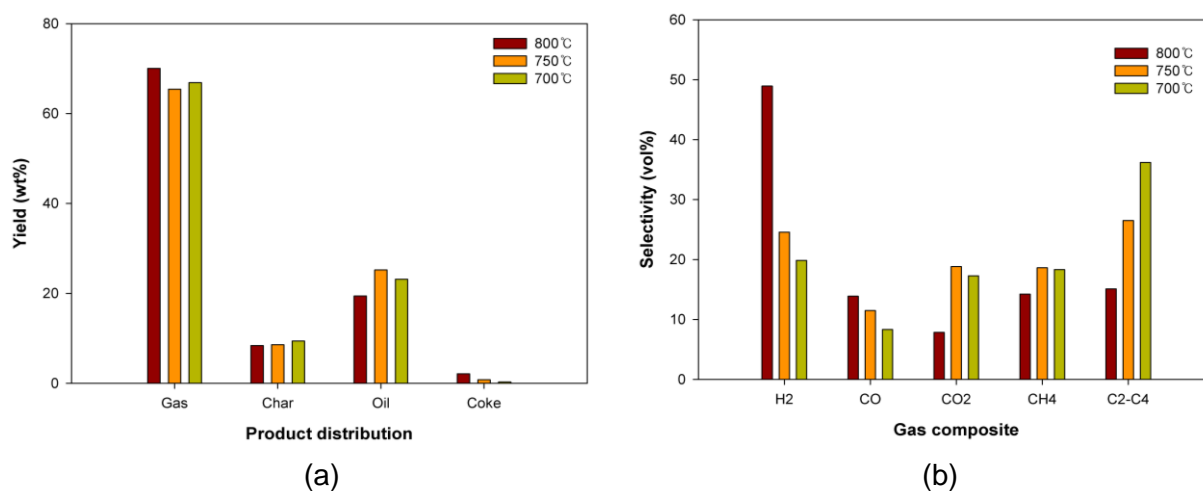
Experiments were conducted by varying the ER ranging from 0.2 to 0.3 at a fixed gasification temperature of 800°C to investigate the effect of ER. The results showed that the amount of products generated during gasification was strongly influenced by the amount of supplied air, i.e., ER. As ER increased, the gas yield increased at the expense of the oil yield, as shown in Fig.5 (a). When the results of the catalyst were compared at ER values of 0.2 and 0.3, the gas yield increased from 70.04 wt.% to 74.06 wt.%, while the oil yield decreased from 19.44 wt.% to 16.92 wt.%. As more air is introduced with higher ER values, combustion inside the reactor increases, generating more heat and promoting gasification reactions of the reactants, resulting in a higher gas yield. On the contrary, Fig.5 (b) shows that the selectivity of H<sub>2</sub> decreased from 48.93 vol% to 34.06 vol% as ER increased from 0.2 to 0.3, whereas the selectivity of CO<sub>2</sub> increased from 7.84 vol% to 18.64 vol%. As more oxygen is supplied to the experiment with increasing ER, the oxidation of hydrogen ( $H_2 + 0.5O_2 \leftrightarrow H_2O$ ) and carbon monoxide ( $CO + 0.5O_2 \leftrightarrow CO_2$ ) gases occurs more frequently [27]. As a result, more carbon dioxide gas is generated instead of hydrogen and carbon monoxide gases.



**Fig. 5.** Catalytic air gasification of WPC, using RM with C/F of 0.1 at 800 °C. Product distribution (a), Gas composition (b)

### 3.3. The effect of different Temperature

To investigate the yield of the products and changes in gas composition with temperature, experiments were conducted under various temperature conditions (700, 750, and 800°C). As indicated in Fig.6 (a), the highest gas yield (70.04 wt%) and the lowest oil yield (19.44 wt%) were achieved at 800°C. Furthermore, Fig.6 (b) shows that the selectivity of H<sub>2</sub> and CO increased as the temperature increased from 700 °C (19.86 and 8.33 vol.%, respectively) to 750 °C (24.56 and 11.49 vol.%, respectively) and 800°C (48.93 vol.% and 13.89 vol.%, respectively). In contrast, CO<sub>2</sub>, CH<sub>4</sub>, and C<sub>2</sub>-C<sub>4</sub> showed a decreasing trend as the temperature increased to 800°C. This result suggests that selectivity for the production of H<sub>2</sub> and CO improves at higher temperatures for several reasons. First, tar can be further decomposed at higher temperatures, as a result, a series of endothermic and exothermic reactions [28]. Second, more H<sub>2</sub> and CO are produced as the temperature increases due to the endothermic properties of the Boudouard ( $C + CO_2 \leftrightarrow 2CO$ ) and steam-gasification ( $C + H_2O \leftrightarrow CO + H_2$ ) reactions and the exothermic properties of the hydrogenation-gasification reaction ( $C + 2H_2 \leftrightarrow CH_4$ ) [29].



**Fig. 6.** Catalytic air gasification of WPC, using RM with ER of 0.2. Product distribution (a), Gas composition (b)

#### 4. Conclusion

In this study, catalytic gasification using low-cost catalysts was conducted to produce H<sub>2</sub> through air gasification process using WPC. Results showed that the highest gas yield and selectivity of H<sub>2</sub> were obtained when the C/F ratio was 0.2 using dolomite, olivine, and RM catalysts. However, compared to a C/F ratio of 0.1, the catalytic activity did not change considerably by raising the C/F ratio to 0.2. When a catalyst containing 10 wt% Ni was used, the selectivity of H<sub>2</sub> and CO and the gas yield increased, due to the catalytic effect of metallic

Ni active sites. Using RM as the catalyst, the gasification results showed the highest gas yield and selectivity of H<sub>2</sub> and CO regardless of the C/F ratio and Ni loading. At the constant condition, using RM as catalyst, changing ER from 0.2 to 0.3 enhanced the gas yield while reduction of H<sub>2</sub> and CO selectivities. Additionally, at a temperature of 800°C, the highest gas yield and volume percentages of H<sub>2</sub> and CO were observed. Therefore, employment of 10 wt.% Ni/Redmud at 800°C with ER of 0.2, and a C/F of 0.1, showed a maximum gas yield and H<sub>2</sub> and CO selectivity in this study.

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