Study of Ni/Char Catalyst for Biomass Gasification in an Updraft Gasifier: Influence of Catalyst Granular Size on Catalytic Performance

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In order to examine whether catalyst granular size was a factor for tar removal and syngas composition enhancement, three Ni/char catalysts with 90-100 mesh (Ni/SC), 50-60 mesh (Ni/MC), and 20-30 mesh (Ni/LC) size were prepared with a mechanical mixing method and tested in an updraft gasifier. Reforming parameters investigated were the reaction temperature (650-850°C), the Ni loading (5-20% of the weight of char support), and the gas residence time (0.1-1.2 s). It was found that the catalyst granular size affected the diffusion of reactants, as well as of products. Ni/SC, Ni/MC, and Ni/LC removed more than 96.5% of the tars in syngas at 800 °C reforming temperature, 15 wt. % Ni loading, and 0.3 s gas residence time. Tar removal efficiencies increased with the decrease of Ni/char granular size, which can be attributed to the higher active surface area with the Ni/SC catalyst. As the catalyst granular size was increased, the H_2 content in the syngas was increased and the CO content was significantly decreased. The total amount of combustible gases (H_2, CO, and CH_4) obtained in the syngas with Ni/MC was higher than with Ni/SC or Ni/LC. It was concluded that Ni/char, especially Ni/MC, can be considered as an effective and inexpensive catalyst for biomass gasification tar removal and syngas conditioning.

Keywords: Gasification; Syngas; Tar reforming; Ni/char; Granular size

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INTRODUCTION

Biomass gasification technologies have been investigated extensively in recent years for the production of sustainable fuels (Torres *et al*. 2007; Stiegel and Maxwell 2001). The main syngas products of biomass gasification are H_2, CO, CH_4, CO_2, and N_2 (if air is used in combustion) in various proportions. This mixture can be directly burned or used for electricity generation, or also turned into liquid transportation fuels (Bridgwater 1995; Li *et al*. 2009). However, in addition to syngas, impurities such as tars are also produced, which is one of the obstacles for the widespread application of syngas. Tar is a generic term comprising all organic compounds present in syngas except gaseous hydrocarbons. In order to reduce the tar content of syngas, several approaches for tar removal, such as physical treatment (Hasler and Nussbaumer 1999), thermal cracking (Fagbemi *et al*. 2001), plasma-assisted cracking (Nair *et al*. 2003), and catalytic reforming (Baker and Mudge 1984), have been reported in the literature. Among these, catalytic reforming is considered the most promising in large-scale applications because of its fast reaction rate and reliability, as well as its ability to increase the quantity of useable gases such as CO and H_2 in syngas (Huber *et al*. 2006).

Wang *et al*. (2010a) prepared and tested NiO supported by hydrothermal carbonization (HTC) chars for syngas cleanup in an updraft gasifier. These authors reported in another article (Wang *et al*. 2011) that 97% tar removal efficiency was achieved with a Ni/coal char catalyst using the optimum condition of 800 °C reaction temperature, 15 wt. % Ni loading, and 0.3 s gas residence time. However, the type and structure of the char support not only influenced the dispersion and activity of nickel catalyst, but it also affected the diffusion of reactants and products towards and away from the catalyst's surface. Several researchers (Rytter *et al*. 2007; Cousins *et al*. 2006; Pis *et al*. 1998) also demonstrated that different catalyst granular sizes affect the diffusion of reactants, as well as products, using simulations and experiments. Therefore, it is a challenging problem to both reduce the costs and increase the efficiency of Ni/char catalyst by optimizing the char size for the support structure of nickel.

In this research, three Ni/char catalysts with different granular sizes were prepared with a mechanical mixing method and tested in an updraft gasifier. The objectives of this study were: (1) to select the suitable char size for nickel catalyst support, and (2) to determine the effect of operating parameters (*i.e.*, catalytic reaction temperature, gas residence time, and Ni loading) on tar removal efficiency and enhancement of the syngas composition.

EXPERIMENTAL

Catalyst Preparation and Characterization

Three catalyst granular sizes were prepared and studied: Ni/small-size char (Ni/SC), Ni/intermediate-size char (Ni/MC), and Ni/large-size char (Ni/LC). The chars were obtained from Royal Oak Enterprises, Inc. (Omaha, AR, US). Chars were crushed and sieved to obtain granular subsamples in the size ranges of 20-30, 50-60, and 90-100 mesh as large-size char, intermediate-size char, and small-size char, respectively. Nickel oxide powders were purchased from Fisher Scientific (Pittsburgh, PA, US). All the Ni/char catalysts were prepared by mechanically mixing char particles and NiO particles in a transparent glass beaker at desired ratios of Ni to char weight (5% to 20% at interval steps of 5% units). All catalysts were characterized with a HITACHI S-3500N scanning electron microscope (SEM; Hitachi, Ibaraki, Japan) through SEM/EDX (energy dispersive X-ray) measurements. For more detailed characterization of the catalysts, hydrogen chemisorption was performed to determine the Ni dispersion ratio, diameter of spherical Ni crystallites, and specific surface area using a Zeton Altamira AMI-200 unit (Altamira Instruments, Pittsburgh, PA). The catalyst (0.1 g) was loaded in a U-shaped quartz reactor fitted with a thermocouple for continuous temperature measurements. Thermal treatment of samples was conducted with 10% H_2/Argon at a flow rate of 50 mL/min and a heating rate of 10 °C/min up to 800 °C. The treatment was holding for 60 min at 800 °C then the catalyst was flushed for 2 h with Argon after the temperature of catalyst returned to room temperature.

Apparatus and Experimental Parameters

The system consisted of three subsystems: (1) an updraft biomass gasifier; (2) a tar reforming unit; and (3) a tar collection unit. The gasification system provided an overall air flow rate of 0.14 to 1.42 m^3/min depending on the voltage of the 15-W blower

(Delibang, Zhejiang, China). The tar reforming system was comprised of a quartz reactor tube that is 2.54 cm in diameter and 61 cm long equipped with a complete vacuum-sealing assembly (MTI Corporation, Richmond, CA, US) and a Thermolyne Economy solid-tube furnace (Fisher Scientific, Pittsburgh, PA, US). The catalyst bed was placed in the center of the tube and horizontally supported by two alumina foam blocks (MTI Corporation, Richmond, CA, US). The tar sampling unit was comprised of four 250-mL flasks dipped into an insulated box filled with dry ice. A detailed description of this system has been previously reported (Wang *et al*. 2011). In a typical tar reforming experiment, a small fraction of the syngas was directed to the reforming tube, and the syngas flow rate was adjusted and measured with a gas valve and a gas flow meter (Brooks Instrument, Hatfield, PA, US). Table 1 shows the experimental parameters of tar reforming. Reaction temperatures were in the range of 650 °C to 850 °C (at incremental steps of 50 °C), and Ni loadings ranged from 0% to 20% (at incremental steps of 5% units). Gas residence times were set at 0.1, 0.3, 0.6, 0.9, and 1.2 s by adjusting the syngas flow rate into the reforming tube through the flow control valve.

Table 1. Experimental Parameters of Tar Reforming

Condition	**Parameter**
Gasification agent	Air
Biomass feed	Sawdust
Catalytic bed temperature, °C	650 to 850
Gas residence time, s	0.1 to 1.2
Ni loading in catalysts	0 to 20%
Gas flow rate, L/min	9.12
Reforming time, min	15
Char granular size, mesh (mm)	20 to 100 mesh (0.15 to 0.85 mm)
Catalyst bed length, cm	9
Amount of catalysts used, g	18

Each sampling was conducted for over 100 liters of syngas in order to obtain sufficient quantities of tars. The tars released during processing were separated from the gas phase by directing the flow through cold flasks dipping into an insulated box filled with dry ice. All tars and vapors were quenched and collected in the flasks, which were then dried at 105 °C in an oven for 2 h. The residue was considered as tars. Other researchers have used similar or slightly modified forms of this method (Narváez *et al.* 1997; Leppala *et al.* 1991). It must be noted that only heavy tars (boiling point above 105°C) were measured in this study, considering that heavy tars (*e.g.*, heterocyclic compounds, heavy poly-aromatics, and asphalt) are more destructive to the downstream equipment of the gasifier and difficult to remove than light tars in catalytic cracking. Syngas was collected from the sampling port or the outlet of the vacuum pump with a 500-mL Tedlar sampling bag. Analysis of syngas was carried out with an SRI 8610s gas chromatograph equipped with a thermal conductivity detector (SRI Instruments, Torrance, CA).

RESULTS AND DISCUSSION

Characterization of Catalysts

In this research, the same quantity of nickel catalysts was supported by the same quantity of chars with different granular sizes. For the same weight of catalyst, large-size char possessed fewer interspaces among the granules of chars than the others. Therefore, it should be noted that the nickel catalyst was more concentrated on the surface of large-size char *vs*. the smaller size chars. As expected, Ni particles at the surface of the large-size char (Fig. 1c) were distributed more uniformly and better dispersed than the others (Fig. 1a and 1b) at the condition of 15 wt. % nickel loading. Table 2 shows once again that the Ni distribution on the large-size char was more uniform, in terms of the higher Ni dispersion ratio on the Ni/LC. The specific surface area of Ni on the small-size char was slightly bigger than that of the median-size char and significantly bigger than that of the large-size char.

The elemental compositions of char used in the experiments were also analyzed by SEM/EDX. The composition of char was: 79.06% C; 19.56% O; 0.61% Na; 0.08% Si; 0.09% K; and 0.56% Ca. The insignificant amounts of Na, K, Ca, and Si were helpful in tar removal (Abu *et al*. 2008).

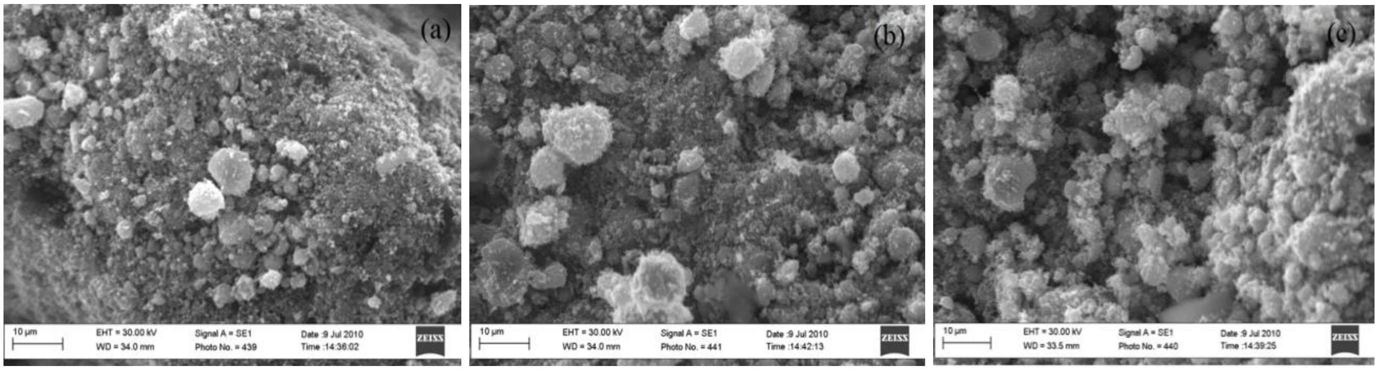

Fig. 1. SEM images of Ni/small-size char (a), Ni/intermediate-size char (b) and Ni/large-size char (c). All catalysts contained 15% nickel loading. The bright dots are Ni.

Table 2. Metal Dispersion Ratio, Diameter of Spherical Ni Crystallites, and Specific Surface Area of Nickel from H_2 Chemisorption Analysis of Ni Catalysts with 15% Ni Loading

Catalyst	Ni dispersion ratio (%)	Diameter of spherical Ni crystallites (nm)	Specific surface area of Ni (m^2/g_{Ni})
Ni/small size-char	41.3	22.6	57.2
Ni/intermediate-char	46.1	21.7	53.3
Ni/large size-char	52.8	23.9	42.2

The Catalytic Performance of Ni/char with Different Granular Sizes

Effect of reforming temperature

The effect of reforming temperature on tar removal and syngas composition were investigated for the Ni loading with different size chars within the temperature range of 650 °C to 850 °C, while the Ni loading and gas residence time were fixed at 15 wt. % and 0.3 s, respectively. As shown in Fig. 2, tar removal efficiency increased with increasing reaction temperature in all cases. Under the same reaction conditions, the Ni/SC catalyst showed the highest tar removal efficiency at all tested temperatures, ranging from 91.27% to 98.29% removal. The Ni/LC catalyst had tar removal efficiencies ranging from 89.55% to 97.44% for reaction temperatures between 650 °C and 850 °C; these efficiencies were less than those for Ni/SC and Ni/MC. The surface area of the small-size char was much greater than that of the large-size char for the same quantity of catalyst. The greater surface areas allowed for more opportunities for the tar to contact the active sites throughout the catalyst bed. Therefore, nickel catalysts supported by a small-size char should be considered the best choice for the purpose of tar removal. However, too small catalyst granular size will increase the flow resistance of the syngas, which can lead to inhomogeneous distribution of reactants within the catalyst bed. Furthermore, a reaction temperature above 800 °C may not be beneficial, considering that overheating usually causes sintering of the catalyst and consumes more energy. For this reason, 800 °C was considered an appropriate temperature for tar removal in this study.

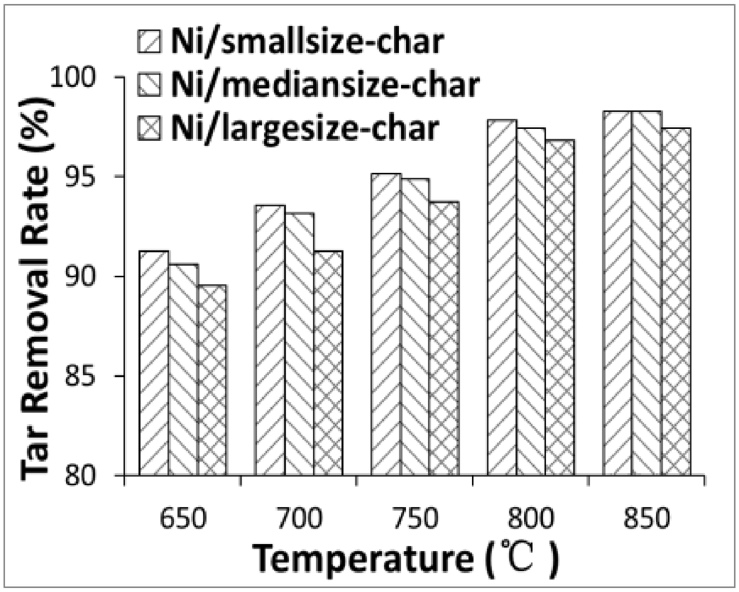

Fig. 2. Effect of catalytic reaction temperature on tar removal (Reaction conditions: 15 wt. % Ni loading when applicable; and gas residence time 0.3 s)

Figure 3 shows the H_2, CO, and CH_4 concentrations as a function of reaction temperature for various char sizes of Ni/char catalysts. As expected, the H_2 concentration increased with increasing temperature for all the catalysts. The H_2 concentration from the Ni/MC was increased significantly over the Ni/LC catalyst from 29.86 to 34.82 vol. % and from 30.05 to 35.58 vol. %, respectively. However, the H_2 concentration increased slowly with the Ni/SC catalyst from 27.62 to 30.12 vol.% as the reaction temperatuere increased. The CO concentration increased significantly, with increasing reaction temperature, from 30.15 to 34.53 vol. % and from 29.74 to 32.73 vol.% for the Ni/SC and the Ni/MC catalyst, respectively. Meanwhile, CO concentraion also increased from 25.08 to 28.34 vol. % with the Ni/LC catalyst. Abu El-Rub *et al.* (2008) investigated the catalytic effect of biomass char for phenol conversion in a simulated experiment, and reported that the CO concentration considerably increased from 0.0025 to 9.2 vol. % at 900 °C. Gilbert *et al.* (2009) investigated tar reduction from pyrolysis vapors from biomass using char as catalysts. They reported that the heavy tar decreased from 19.3 to 8.33 wt. % and CO increased from 8.98 to 14 wt. % with the reforming temperature increasing from 600 °C to 700 °C. This indicated that biomass char itself contributed to the catalytic ability relative to tars, and CO concentration increased simultaneously in this process. Several researchers (Wang *et al*. 2010b; Huber *et al.* 2006) verified that nickel-based loading confers higher catalytic activity towards tars, and favors a water-gas shift reaction for increased H_2 concentration. In terms of characterization of catalysts, there is much more surface area on chars not covered by nickel in the Ni/SC, which indicated that char also plays a key role in tar reforming together with nickel catalysts. Char as catalysts in tar reforming increase CO production, thus, it could be observed that CO content in syngas in the presence of Ni/SC was higher than otherwise.

Depending on the sum of H_2, CO, and CH_4 from Fig. 3, the total amount of combustible gas (H_2, CO, and CH_4) increased from 63.29 to 70.27 vol.% by Ni/MC, higher than that of 61.48 to 68.99 vol.% by Ni/SC and 57.8 to 67.17 vol.% by Ni/LC, while the reaction temperature increasing from 650 to 850 °C. Therefore, Ni/MC should be considered as the most effective and inexpensive catalysts when compared with Ni/SC and Ni/LC for the purpose of increasing combustible gases.

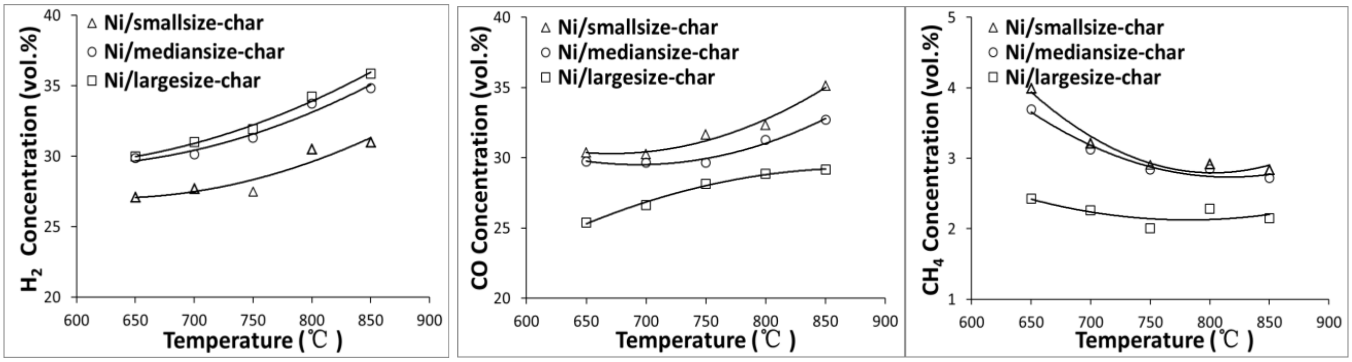

Fig. 3. Effect of catalytic reaction temperature on H_2, CO, and CH_4 concentrations (Reaction conditions: Ni loading 15 wt. %; and gas residence time 0.3 s)

Effect of nickel loading

The effect of Ni loading on tar removal and syngas reforming was studied with three different sizes of Ni/char catalysts at 0.3 s gas residence time and 800 °C reaction temperature. As shown in Fig. 4, the tar removal efficiency steadily increased with increasing Ni loading, which indicated that Ni plays a significant role in tar removal.

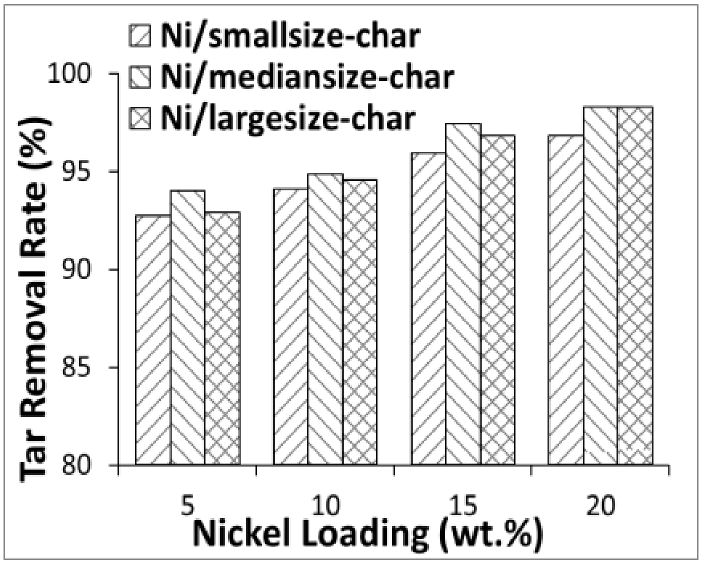

Fig. 4. Effect of nickel loading on tar removal (Reaction conditions: reaction temperature 800 °C; and gas residence time 0.3 s)

The Ni/MC showed better performance than other catalyst sizes when the Ni loading was increased from 5 to 20 wt. %. This can be explained by the fact that Ni/MC had a higher active surface area and dispersion ratio. Figure 4 also indicates that the char itself played an important role when the nickel content was less than 10 wt. %; thus, Ni/MC yielded higher tar removal efficiency (94.87%) even at 5 wt. % nickel loading. With the increase of nickel content above 10 wt. %, nickel catalyst gradually became the dominant factor for tar removal, where the Ni/LC had almost the same tar removal efficiency as Ni/MC. It can be noted that there was similar catalytic performance for the Ni/MC (*i.e.* >97% tar removal efficiency) between 15 wt. % and 20 wt. %. Thus, 15 wt. % could be an optimum Ni loading level for the char support used in this study.

The effect of Ni loading on syngas composition is shown Fig. 5. With the increase of nickel content from 5 to 20 wt. %, H_2 concentration significantly increased from 30.12 to 37.46 vol.% for the Ni/LC, which suggested that it is mainly Ni that enhanced H_2 generation. This can be explained that with the increase of char granular size, the relative surface area of char decreases, Ni was distributed more uniformly and better dispersed on the surface of the Ni/LC; therefore, the nickel catalyst played the main role with H_2 generation during syngas reforming. In contrast, with the Ni/SC, higher CO concentration (from 28.67 to 34.23 vol.%) was observed. With the increase of nickel loading, the CH_4 concentration in syngas decreased for all catalysts. CH_4 concentration decreased significantly in the presence of Ni/LC, which indicated that the nickel catalyst played a main role with the lower CH_4 levels.

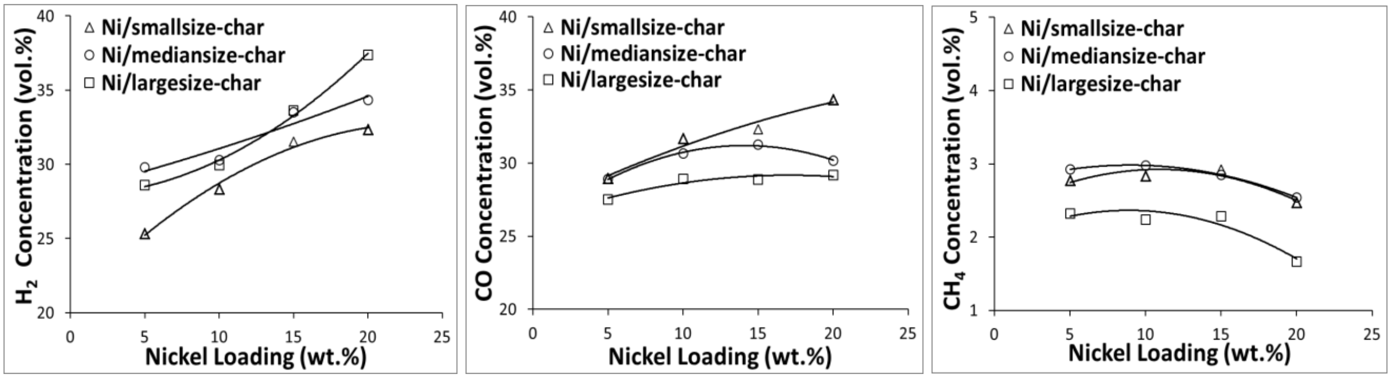

Fig. 5. Effect of nickel loading on H_2, CO, and CH_4 concentrations (Reaction conditions: reaction temperature 800 °C; and gas residence time 0.3 s).

Effect of gas residence time

Figure 6 shows the tar removal efficiency as a function of gas residence time. It can be seen that tar removal efficiency remained high when gas residence time was greater than 0.3 s; however, the removal efficiency significantly decreased below 0.3 s. This indicated that the tar needed sufficient time to decompose. When gas residence time increased over 0.3 s, the increase of tar removal efficiency gradually slowed down. It also can be observed tar removal efficiency of Ni/SC and Ni/MC was basically the same when the gas residence time was greater than 0.3 s, which indicated that the size of the char had negligible effect on the catalytic performance of the Ni/char.

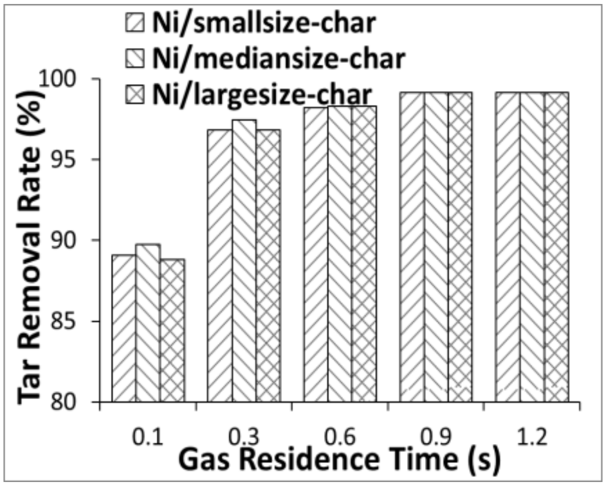

Fig. 6. Effect of gas residence time on tar removal (Reaction conditions: Ni loading 15 wt. %; and temperature 800 °C)

Figure 7 shows the change of concentration of H_2, CO, and CH_4 at various gas residence times. It can be found that the H_2 concentration increased significantly when the gas residence time was increased from 0.1 s to 0.3 s. After 0.3 s, the increase of H_2 concentration slowed down, similar to the trend observed for the tar removal efficiency. H_2 concentration approached its highest value at 0.9 s gas residence time, 33.25 vol. % for Ni/SC, 37.42 vol. % for Ni/MC, and 38.39 vol. % for Ni/LC, respectively. CO concentration also significantly increased for all Ni/char catalysts, but peaked at 0.9 s. Baker and Mudge (1984) found that 0.2 s gas residence time was enough to convert more than 90% of the phenol at 750 °C. Kinoshita *et al.* (1995) found that syngas composition remained stable after 1.3 s gas residence time using commercial Ni catalysts at 750 °C. Because tar removal is the main purpose of catalytic reforming, and longer gas residence times require slower syngas flow rates or the use of more catalysts, a minimal but reasonably effective time, such as 0.3 s, is desired.

CH_4 concentration decreased as the gas residence time increased, especially for Ni/LC. All of the results suggested that a sufficient gas residence time is required to enable the catalytic reactions to reach equilibrium; however, longer gas residence times did not enhance combustible gas generation.

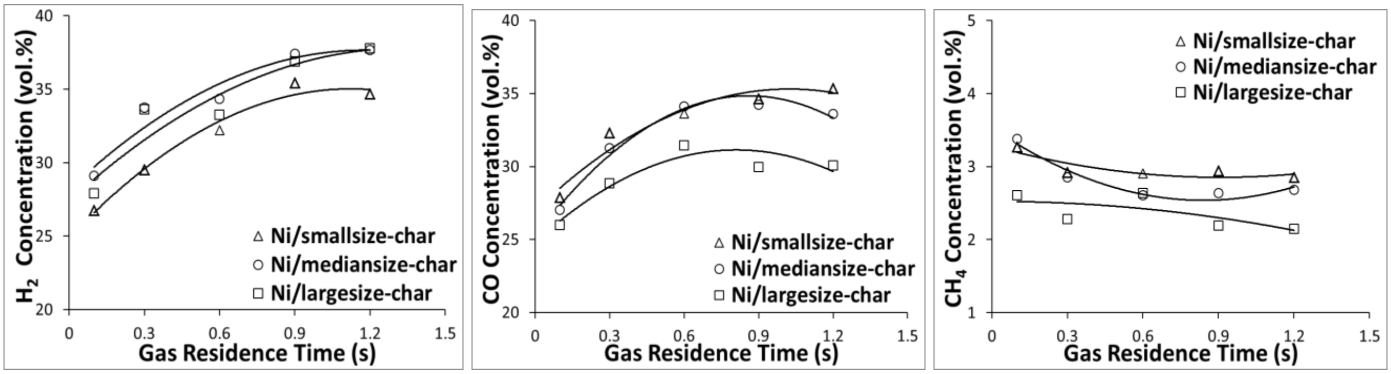

Fig. 7. Effect of gas residence time on H2, CO, and CH_4 concentrations (Reaction conditions: Ni loading 15 wt.%; gas residence time 0.3 s)

Chars as the catalyst support have several advantages. First, Ni can be saved by using char as the support. Ni particles stay only on the outer surface of the char support. The outer surface of catalysts are active sites. Second, mechanical mixing of Ni and char saves energy and time in catalyst preparation. While using chars with small-size as the support structure of Ni, these chars showed the highest tar removal efficiency at all tested temperatures; however, the H_2 content in syngas was lower. Higher H_2 content can be obtained by use of the large-size chars supporting the Ni catalyst, but these chars were not ideal for maximum tar removal. Chars with the intermediate-size as the support of Ni not only can obtain higher tar removal efficiency, but also higher total amounts of generated combustible gases (H_2, CO, and CH_4) in the syngas. Therefore, chars with the size of 50 to 60 mesh should be considered as the optimum support for the Ni/char catalyst.

CONCLUSIONS

1. Nickel and three different sizes of chars were mechanically mixed at various ratios and tested in an updraft gasifier. Catalyst characterizations showed that the Ni particles were distributed at the surface of large-size char more uniformly and more fully dispersed than others.
2. A reaction temperature of 800 °C, Ni loading of 15 wt. %, and gas residence time of 0.3 s were deemed optimum. Ni/SC, Ni/MC, and Ni/LC removed 97.8%, 97.4%, and 96.8% of the tars in the syngas at the optimum reforming conditions, respectively.

3. As the catalyst granular size increased, the H_2 content in syngas increased from 30.52 to 34.22 vol. %; meanwhile, the CO content decreased from 32.31 to 28.86 vol. % at 800 °C reforming temperature, 15% Ni loading, and 0.3 s gas residence time. The total amount of combustible gas (H_2, CO, and CH_4) obtained in the syngas with the Ni/MC was higher than with Ni/SC or Ni/LC.
4. Based on the results from this study, it was evident that the Ni/char, especially Ni/MC, can be an effective and inexpensive catalyst for biomass gasification tar removal and syngas conditioning.

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