In-situ reforming of the volatiles from fast pyrolysis of ligno-cellulosic biomass over zeolite catalysts for aromatic compounds production

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ABSTRACT

Characteristics of in-situ catalytic reforming of the products derived from fast pyrolysis of biomass were studied with an originally designed analytical pyrolysis technique. The volatile products derived from ligno-cellulosic biomass as well as cellulose, xylan, and lignin were converted using a two-stage tubular reactor at 550 °C over various zeolite catalysts with different acidities and pore structures. HZSM-5 exhibited the best performance for converting the cedar derived volatiles to arenes, which mainly composed of benzene, toluene, and naphthalene with a selectivity of 26% on carbon basis. The HZSM-5 had a little effect in increasing the yields of the

1 arenes for xylan and lignin, whilst it had a significant effect for cellulose, showing that more than
2 30% of carbon in cellulose was converted into arenes. A reaction pathway analysis for reforming
3 of volatiles suggested that alkyne and diene such as acetylene, propyne, and cyclopentadiene are
4 the important precursors of the major aromatic hydrocarbon products such as benzene, toluene,
5 and naphthalene. The formations of those intermediates were also confirmed experimentally in
6 an early stage of the in-situ reforming.

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10 **Keywords**

11 Catalytic reforming; Reaction mechanism; Nascent volatiles from ligno-cellulosic biomass;
12 Arenes; Zeolite catalysts

1. Introduction

Ligno-cellulosic biomass such as agricultural residues, short rotation woody crops, herbaceous crops, forestry residues, and waste paper is considered to be a promising source for renewable liquid fuel [1-3]. The fast pyrolysis of biomass is one of the viable processes to convert it into liquid fuels, which are referred to as bio-oil. Bio-oil is considered to be as a promising alternative fuel, produced by simple methods with high efficiency [4, 5]. However, the quality of bio-oil is much lower than that of the conventional petroleum-based oil because of its thermal instability, acidic nature, and low calorific value [6]. The effective upgrading techniques are needed to meet the standards of the conventional fuel [7-9].

Catalytic reforming is one of the promising ways to upgrade crude bio-oil. There are few reports that investigate in-situ reforming of volatiles derived from the fast pyrolysis of biomass [10-12]. For example, Carlson et al. [11], investigated the conversion of biomass into aromatics by thermal decomposition in the presence of zeolite catalysts using pyroprobe analytical pyrolyzer. They reported that more than 30% aromatic compounds were obtained from cellulose with zeolite catalyst (ZSM-5, $\text{SiO}_2/\text{Al}_2\text{O}_3 = 60$) at the optimal reactor conditions. The high conversion efficiency from those carbohydrates which are free from aromatic structures to aromatics should be of great interest. However, the mechanistic interpretations for this process are limited since the complex processes such as primary pyrolysis and reforming of volatiles over catalysts as well as nascent char are simultaneously occurred in the pyroprobe reactor. The volatile-char interaction is known to have a positive effect on tar destruction [13, 14].

The objective of this work is to study experimentally the in-situ reforming characteristics of the products from the fast pyrolysis of biomass over various zeolite catalysts under minimized volatile-char interactions by using a two-stage tubular reactor which is directly connected to a

gas analysis system. The tubular reactor was divided into two zones by a filter, the first zone is used for fast pyrolysis of biomass, and the other zone is used for catalytic reforming. The reactor has a set-up to control the contact times of the nascent volatiles over the catalyst. Moreover, a reaction pathway included in the conversion into arenes is also proposed based on the detailed chemical kinetic model proposed previously [15].

2. Experimental

Biomass pyrolysis with in-situ catalytic reforming experiments were carried out in a U- shaped two-stage tubular reactor (TS-TR) at 550 °C. All the fast pyrolysis with reforming experiments were carried out at 550 °C in the presence of inert gas helium or nitrogen. A schematic representation of the reactor is shown in Fig. 1. The TS-TR was made-up of quartz and divided into two zones by a quartz wool filter, the first zone is used for fast pyrolysis of the biomass and second is used for catalytic reforming of volatiles derived from the biomass on zeolite catalysts. The second zone of the reactor is loaded with catalyst zeolite particles. Table 1 shows the characteristics of the five zeolites with different acidity and pore size. All catalysts are purchased from Zeolyst International and Tosoh. The inner diameters of the pyrolysis zone and the reforming zone were 6 and 4 mm, respectively.

Japanese cedar (C: 50.2; H: 6.3; O: 42.3 wt%, dry basis), cellulose (Sigma Aldrich), lignin (Kishida Chemical Co., Japan), and xylan (Tokyo Chemical Industry, Japan) were used as samples in this study. Sawdust of cedar was used and the diameter was in the range of 0.50 to 1.18 mm. Xylan was used as a model compound for hemicellulose. Approximately 1.0 mg of the sample was wrapped with a sheet of stainless (SUS316) wire mesh with a mesh opening of 45 µm and dried in vacuum oven at room temperature overnight. The mass of the wrapped sample was measured immediately after drying to determine the exact dry mass of the sample, which

1 was subsequently fixed at the upper part of the TS-TR. The reactor was heated to 550 °C by
2 external furnace, after stabilizing the heated section of the TS-TR, the sample was dropped into
3 the bottom of the first tube, where the biomass sample was fast-pyrolyzed. The volatiles and char
4 were formed in the first zone of the reactor. The volatiles released were subsequently swept off
5 into the catalyst bed for reforming. The residence time (0.02 to 0.16 s based on catalyst bed
6 length) of the volatiles in the catalytic zone was controlled by the amount of loaded catalyst
7 varying from 0.012 to 0.11 g in the second zone of the reactor.

8 The reformed products were introduced into gas chromatographs (Shimadzu GC 2014 or GC
9 2010) with helium or nitrogen used as carrier gas. The products were exhaustively analyzed by
10 using three different columns and two different detectors (TCD and FID). More than 40
11 compounds were identified and quantified. Inorganic gasses such as H₂, CO, CO₂, and H₂O were
12 separated by Gaskuropack 54 column (GL Sciences Co. Ltd) and detected with TCD. The
13 packed column Gaskuropack 54, consists of polymer beads with highly cross linked styrene-
14 divinylbenzene copolymer, can able to separate these products. Light hydrocarbons such as
15 alkanes, alkenes, and alkynes, and oxygenated compounds such as methanol, ethanol,
16 acetaldehyde, furan, acetone, propanol, acetic acid, hydroxyl acetone, furfural, and furan were
17 quantified by the same column with FID. Another packed column VZN-1 (GL Sciences Inc.) is
18 used for separating alkene, alkyne, and diene with FID. Since the packed columns have some
19 limitations in detecting high-molecular weight compounds, these compounds were quantified by
20 GC equipped with a capillary column (CP PoraBOND, Agilent Technology, Co. Ltd). High
21 molecular weight compounds such as mono- and bicyclic aromatic compounds were detected by
22 PoraBOND column. The quantifications of the products were conducted based on the external
23 calibration method which uses response vs amount functions that are subsequently used to

estimate amounts of analyte in a separately analyzed sample as long as the standard compounds for the target products were available. The effective carbon number concept [16] was also employed for estimation of the amounts of products of which standards were unavailable. The detailed procedure was published elsewhere [17]. The product yields were calculated based on the carbon content in the feedstock. The yields were calculated according to the equation

$$Y_i = \frac{A_i}{\left(\frac{A_{CH_4}}{N_{CH_4}}\right) * R} * M_i * x_i * \frac{1}{W_{c-feed}}$$

Where, Y_i is the yield of i^{th} component (mol-C/ mol-C in feed), A_i is peak area in the chromatogram, R is the relative response, x_i is the ratio carbon weight to the molecular weight of i^{th} component, and W_{c-feed} is the carbon weight in cedar, and N_{CH_4} is the moles of CH_4 in the standard gas.

3. Results and Discussion

3.1. Non-catalytic experiment

The volatile products derived from the fast pyrolysis of cedar at 550 °C without catalyst in the second zone of the reactor were analyzed by different columns. Typical GC/FID chromatograms along with the assigned peaks are shown in Fig. 2. Fig. 2a shows the Gasukuropak54 chromatogram of inorganic compounds that are identified by the TCD. The same column was also used with FID to detect light hydrocarbons such as alkanes, alkenes, alkynes, and oxygenated compounds such as alcohol, aldehyde, and furan and respective chromatograms are shown in Fig. 2b. However, this column cannot separate acetylene with ethylene. Another packed column (VZN-1) was employed to separate and quantification of the alkyne and dienes

which are shown in Fig. 2(c). As shown in Fig. 2d, CP PoraBOND column is able to detect compounds larger than toluene such as alkylbenzene, phenol and naphthalene.

3.2. Effect of catalyst on catalytic reforming of cedar volatiles

The volatiles from the fast pyrolysis produce water and dehydrated species by hydration reactions, which can occur in the gas phase or in the catalytic surface. These hydrated species are further converted into aromatics, hydrocarbons, water, and so on by a series of reactions in the presence of zeolite catalysts [11]. Fig. 3 shows the chromatographs of catalytic reforming of volatiles derived from cedar in the presence of 0.11 g of HZSM-5 ($\text{SiO}_2/\text{Al}_2\text{O}_3 = 23$) at 550 °C. These chromatograms show (Fig 3) the very large intensities for hydrocarbons and aromatic compounds such as benzene, toluene, and naphthalene. For instance, benzene peaks in Fig 2 are very low compared with the peak intensity in Fig 3, and similar type of observations can be drawn even for hydrocarbons. The peak intensity areas for hydrocarbons are also increased in the presence of zeolite catalysts. The large differences in these chromatographs indicate that the presence of zeolite catalysts have obvious effects on the conversion of volatiles derived from the fast pyrolysis of cedar.

The catalytic reforming experiments were carried out with different zeolite compositions loaded in the second zone of the reactor. In order to get the quantitative information as well as the effect of catalysts on the product distributions, five kinds of zeolite catalysts were used in this study. Fig. 4 shows the product yields (carbon basis) of volatiles derived from fast pyrolysis of cedar on different zeolite type catalysts at 550 °C. The product yields without catalyst is also shown for comparison in Fig. 4. It is obviously observed that the product yields such as hydrocarbons and aromatics showed higher values in the presence of catalysts. HZSM-5 is more active for promoting the formation of arenes than other zeolite catalysts used. When HZSM-5 was used,

the arenes yield increased from 4.6 to 26%, while the oxygenated compounds yield decreased from 6.6 to 0.3%, in comparison with non-catalytic experiment. The catalyst Y-type-2 ($\text{SiO}_2/\text{Al}_2\text{O}_3 = 400$) showed lower aromatic yield than any other catalysts. The yield of aromatics for the different zeolite catalysts is in the order of HZSM-5 > Mordenite > Y-type-1 > β -type > Y-type-2. Fig.4. also reveals that the catalyst Y-type-2 is less effective in converting the oxygenated compounds than any other zeolite catalysts.

The catalyst HZSM-5 is more effective in converting the oxygenated compounds and decreasing conversion in the order of Y-type-1, mordenite, β -type, and Y-type-2. This is mainly due to higher silica to alumina ratio in the zeolite Y-type-2 ($\text{SiO}_2/\text{Al}_2\text{O}_3 = 400$) catalyst. The low ratio of silica to alumina catalyst has more effective in decomposition of oxygenated compounds. It is also inferred from the Fig. 4, the conversion characteristics of HZSM- 5 was different from those with mordenite, even though both the catalysts have similar silica to alumina ratio. This indicates that the frame structure of zeolite is also an important parameter in the catalyst reforming.

3.3. Effect of catalyst on catalytic reforming of model compound

The in-situ catalytic reforming on HZSM-5 catalyst was also carried with individual components of cedar volatiles derived from fast pyrolysis. Ligno-cellulosic biomass such as cedar mainly consists of three components namely cellulose, hemicellulose, and lignin. Xylan was used as a model compound for hemicellulose. The catalytic reforming products distribution of fast pyrolysis of individual components of cedar such as cellulose, lignin, and xylan on HZSM-5 were shown in Fig. 5. The presence of catalyst increases the aromatic product yields in all the individual components of cedar. The similar trends are also observed in the yields hydrocarbon ($\text{C}_1 - \text{C}_5$). The presence of catalyst converts the most of the oxygenated compounds

1 present in the individual components of cedar. This also reconfirms the catalytic effect on
2 volatiles derived from the fast pyrolysis of cedar.

3 The cedar component of cellulose has shown maximum aromatic yield, whereas lignin and
4 xylan showed lower aromatic compounds yields in the presence of catalyst HZSM-5. The
5 aromatic yields showed maximum for cellulose, intermediate for lignin, and minimum for xylan.
6 It is most likely that the cellulose content in biomass contributes to the formation of aromatic
7 compounds. The ligno-cellulosic biomass such as cedar mainly contains about 40-47 wt% of
8 cellulose [18, 19].

9 Thring et al. [21] converted the organosolv lignin dissolved in the double amount of acetone
10 over HZSM-5 at 500–650 °C and WHSV of 2.5–7.5 h⁻¹ using a fixed bed flow reactor. One of
11 the results showed that they succeeded to get aromatic of lignin with 38% yield at 550 °C and
12 WHSV = 5 h⁻¹. In our case of the cracking of nascent volatiles derived from lignin over HZSM-
13 5, we could get aromatic hydrocarbons with the yield of 12 mol-C % (corresponds to 7 wt% of
14 lignin fed) as shown in Fig.5. The possible reason for the lower yield observed in our case is
15 likely to be a very different experimental condition. As evidenced by Thring et al. [21], acetone
16 converted into gaseous products such as ethylene, propylene, and propane almost completely
17 over HZSM-5 at a condition 600 °C and WHSV = 5 h⁻¹ typically used in their experiments. This
18 means that reactive radicals are occurring during lignin decomposition process and likely
19 contribute to quench radicals formed from lignin depolymerization, thereby reducing charring
20 and increasing the liquid product yields. On the other hand, in our experiments, reforming of
21 nascent volatiles from lignin fast pyrolysis over HZSM-5 underwent in the presence of inert
22 carrier gas like helium. This results in the higher char and lower aromatic hydrocarbons (liquid

products) yields when compared with the conversion experiments over HZSM-5 using lignin/acetone mixture as a feed by Thring et al. [21].

3.4. Reaction pathways leading to arenes

There are no interpretations available for the arenes formations in the in-situ reforming of multi-component mixtures derived from cellulose fast pyrolysis over a zeolite catalyst at mechanistic level. The authors developed a detailed chemical kinetic model for describing the vapor phase i.e., non-catalytic reforming of cellulose [15]. Based on this kinetic model, a reaction pathway for aromatic compounds formation is proposed and shown in Fig. 6. The fast pyrolysis of cellulose produces mainly levoglucosan, furfural, D-glyceraldehyde, hydroxyl acetaldehyde, 5-HMF, and hydroxyl acetone, which were also confirmed by Shin et al [20]. These volatiles are further converted into reforming products in the presence of zeolite catalysts. It is suggested that dehydrogenation and cracking of volatiles take place on the surface of the zeolite catalyst. These reactions produce intermediates mainly short chains of alkanes and alkenes. According to this proposed mechanism, alkenes and dienes such as acetylene, propyne, and cyclopentadiene are the important intermediates to the arenes formation. The formation of intermediates such as alkenes and dienes were confirmed from the vapor phase cracking of nascent volatiles derived from cellulose [15,17].

The proposed reaction pathway shows that aromatic benzene is formed mainly from two routes; the first one is the decomposition of toluene, and the second is from C₃ hydrocarbons such as propadiene and propyne. Toluene is formed almost exclusively by the combination of cyclopentadiene with ethylene. Cyclopentadiene is formed mainly by the combination of allyl radicals and acetylene. Allyl radical is formed from butane and propylene, both of which are major constituents of the primary pyrolysis products of cedar. Acetylene is likely produced from

the dehydrogenation of ethylene. Benzene formation from C₃ hydrocarbons is formed from the propylene, which is from the decomposition of furan.

The reforming experiments with fast pyrolysis were also carried out with varying amount of catalyst HZSM-5 in the second zone of the reactor to follow the formation profiles of these important intermediates kinetically. Fig. 7 shows the yields (carbon basis) of alkynes and cyclopentadiene. The yields are increased with increased catalyst loading, and further increase the catalyst loading, the yields were decreased. When the catalyst loading is 0.05 g, acetylene and propyne showed maximum yields. The yield of cyclopentadiene is maximum when the catalyst loading is 0.02 g. The formation of alkyne and diene compounds in the catalytic reforming of multicomponent mixture derived from cellulose fast pyrolysis play a major role in the formation of aromatics. It should be noted that many monomers in lignin are considered to have a C₆-C₃ (phenyl propanoid) structure [21]. It is thus possible that ethylene and propylene are also produced not only from cellulose but also from lignin by cleavage of lignin side chains (C₃ portion) to give aromatic compounds.

4. Conclusions

In-situ reforming of volatiles derived from the fast pyrolysis of cedar were studied at 550 °C. Different types of zeolite catalysts were used in this study. The volatiles from the fast pyrolysis undergo reforming on zeolite catalysts by a series of reactions produce aromatics and hydrocarbons. The catalyst HZSM-5 had showed higher efficiency than other catalysts. The experiments with individual components of cedar, one of the component cellulose showed the maximum conversion of biomass into aromatics products. The reaction pathway is also proposed to understand the in-situ reforming kinetics and indicates that aromatic benzene is formed from

two dominating routes. The experiments with varying catalyst loading (HZSM-5) revealed that the formation of alkynes and dienes were playing important roles in the formation of aromatics.

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List figure captions

Fig. 1. A schematic representation of the experimental set-up for studying the kinetics of in-situ reforming of volatiles derived from the fast pyrolysis of cedar.

Fig. 2. GC chromatograms of volatiles derived from cedar pyrolysis at 550 °C without catalyst. (A) Packed column (Gaskuropack 54)-TCD, (B) Packed column (Gaskuropack 54)-FID, (C) Packed column (VZN-1)-FID, (D) Capillary column (PoraBOND)-FID.

Fig. 3. GC chromatograms of the in-situ reformed products from cedar fast pyrolysis reformed on catalyst (HZSM-5, 0.11 g) at 550 °C. (A) Packed column (Gaskuropack 54)-TCD, (B) Packed column (Gaskuropack 54)-FID, (C) Packed column (VZN-1)-FID, (D) Capillary column (PoraBOND)-FID.

Fig. 4. Product distributions of the reforming of the products derived from fast pyrolysis of cedar at 550 °C on different zeolite catalysts (0.11 g).

Fig. 5. Product distributions of the reforming of the products derived from fast pyrolysis of individual components of cedar at 550 °C on HZSM-5 catalyst (0.11 g).

Fig. 6. A proposed reaction pathway leading to aromatic compounds in in-situ reforming of volatiles derived from the fast pyrolysis of cedar on zeolite catalysts.

Fig. 7. Product distribution of alkyne and diene as a function of catalyst loaded amount of HZSM-5 for reforming of products from fast pyrolysis of cedar at 550 °C.

1 **Table 1**

2 Characteristics of the zeolite catalysts

Zeolite (Frame structure)	SiO ₂ / Al ₂ O ₃ , mol/mol	Na ₂ O, wt%	Unit Cell Size, Å	Surface Area, m ² /g
HZSM-5	23	0.05	-	425
Mordenite	20	0.08	-	500
Y-type-1	5.1	2.8	24.5	730
Y-type-2	400	0.05	500	620
β-type	500	0.1	3000	400

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