Local Wood’s Bio-Oil Upgrading Using Non-sulfided (Co, Mo)/USY Catalyst

K D Nugrahaningtyas\(^1\)\(^a\) and E Prasetyorini\(^1\)
\(^1\)Department of Chemistry, Faculty of Mathematics and Natural Sciences, Sebelas Maret University, Jl. Ir. A. Sutami 36A, Kentingan, Surakarta 57126, Indonesia

\(^a\)E-mail: khoirina@mipa.uns.ac.id

Abstract. In this present study, the non-sulfided (Co, Mo)/USY has used as a catalyst on the hydrotreating and hydrocracking (HC) reactions for upgrading the quality of bio-oil. The bio-oils were obtained from sengon, coconut, meranti, and bangkirai woody biomass by pyrolysis method at a temperature of 300 °C and atmospheric pressure. Meanwhile, the hydrotreating and HC reactions were done with a flow reactor at 300 °C, under H\(_2\) gas flow (20 mL/min). The composition of bio-oil and upgrading bio-oil were analyzed by Gas Chromatography-Mass Spectrometry (GC-MS). The resulted show that the main component of bio-oil is levoglucosan, while the main product of upgrading bio-oil is methyl format. Bio-oil with the highest yield obtained from meranti on the HC reaction that used Co-Mo/USY catalyst.

1. Introduction
The supply of fossil fuels in the world requires alternative energy to replace fossil fuels. Biomass is one of the renewable fuels that have the potential to replace fossil fuels. One example of biomass is woody biomass from agriculture or forests. Woody biomass can convert into fuel by pyrolysis process which will produce bio-oil [1-4]. Based on the cellulose, hemicellulose and lignin composition, woody biomass divided into hardwood and softwood [5]. Therefore, the bio-oil composition, properties and characteristics that obtained from hardwood will be different with the bio-oil from softwood.

As an alternative fuel, bio-oils have the same combustion characteristics with light fraction oil [5]. Moreover, bio-oil is environmentally friendly because the nitrogen and sulfur components in biomass are less than fossil fuels [5-8]. Meanwhile, bio-oil has properties such as heating value [6], viscosity, corrosive, oxygen, water and ash content [9] poor than fossil fuels [6,9]. These characters cause bio-oil cannot use directly as fuel, so they need to be upgraded.

Bio-oil consists of many compounds such as ketone, phenols, aldehyde, ester, alcohol, acid, and hydrocarbon. The multi-chemical compounds in the bio-oil making its upgrading reaction is complicated. Oxygen removal in the bio-oil constituent components can be through several reactions. The reactions that occur can be decarbonization, decarboxylation [8], cracking, hydrocracking (HC) [10-12], hydrodeoxygenation (HDO), hydrogenation (HYD) and polymerization reactions [8,13]. Bimetallic catalyst has higher activity and lower rate deactivation that compare with the monometallic catalyst on bio-oil conversion [14]. HDO reaction of bio-oil that use bimetallic catalyst Fe-Co/SiO\(_2\) has higher activity compared than monometallic catalyst Fe/SiO\(_2\) and Co/SiO\(_2\) respectively [8].

The sulfided CoMo and NiMo that supported by alumina (Al\(_2\)O\(_3\)) is a commercial catalyst for hydrotreating reactions [3]. The sulfided CoMo/Al\(_2\)O\(_3\) has higher activity than the sulfided NiMo/Al\(_2\)O\(_3\), on HDO of anisoole and guaiacol [15]. Ariyani and coworkers [16] have been used free-sulfide
The pyrolysis of sengon, coconut, meranti and bangkirai bio-oil component

The pyrolysis of sengon, coconut, meranti and bangkirai produces three types of products in the form of charcoal (biochar), bio-oil, and gas. The characteristics of bio-oil are dark brown, very thick and smell like smoke. The percentages of bio-oil that is produced from pyrolysis of sengon, coconut, meranti and bangkirai are 36.98%, 35.76%, 38.76% and 32.97%, respectively. The main component that is contained in bio-oil is levoglucosan. Levoglucosan is the main product of the pyrolysis of cellulose[20]. Bio-oil of each woody biomass has a different percentage of levoglucosan because of each woody biomass has a different cellulose content [5,21]. The higher levels of levoglucosan in bio-oil show the higher cellulose content in the woody biomass. The order of woody biomass that produces high amounts of levoglucosan is meranti, bangkirai, sengon, and coconut, respectively.

The percentage of levoglucosan component is also influenced by the temperature that used on the pyrolysis process [2]. The pyrolysis temperature at ± 300°C is optimum thermal decomposition of cellulose and hemicellulose components, but that is not optimum temperature for the lignin decomposition. That is according to Mohan et al. [5], which said that the decomposes of cellulose occurs at 240-350°C, hemicellulose decomposes at 200-260°C, while the lignin decomposition occurs at 280-500 °C.

Overall, the components of bio-oil derived from woody biomass are consisting of ethers, esters, carboxylic acids, aldehydes, ketones (cyclic and aromatic), alcohols (aliphatic and cyclic), furan, phenol, glucose, and impurity compounds such as S and N. Those compounds are the reaction product of cracking, dehydration, decarboxylation, ring opening, and degradation of cellulose, hemicellulose, and lignin [22]. Ketone, carboxylic acids, hydrocarbons, aromatics, and phenolic compounds are hemicellulose degradation products. The other products such as simple phenols which is substituted methyl groups, ketones (especially cyclic), aldehydes, and alcohol, are obtained from cellulose degradation. The lignin decomposed products are a complex phenol compound (a heavy molecular weight and substituted of methoxy group) and acid [22].
Table 1. Distribution of bio-oil compounds from pyrolysis of sengon, coconut, meranti and bangkirai woody biomass.

| Compound            | Percentage (%) |
|---------------------|----------------|
|                     | Sengon | Coconut | Meranti | Bangkirai |
| Ester               | 11.03  | 13.52   | 9.55    | 2.55      |
| Ether               | 7.39   | 1.15    | 3.82    | 2.24      |
| Carboxylic Acid     | 5.57   | 10.66   | 1.88    | 2.75      |
| Ketone              | 14.85  | 7.85    | 8.78    | 18.21     |
| Alcohol             | 7.27   | 2.72    | 3.18    | 6.42      |
| Alcohol             | 6.15   | 1.19    | 5.11    | 6.26      |
| Phenolic            | 11.85  | 27.62   | 19.69   | 18.85     |
| Hydrocarbons        | -      | -       | 1.05    | 1.22      |
| Levoglucosan        | 30.71  | 17.57   | 46.94   | 35.17     |
| Another Component Contain N and S | 5.2    | 17.71   | 0.9     | 6.36      |

3.2. Product of Upgrade Bio-oil

3.2.1. Product Distribution of Upgraded Bio-oil

Table 2 shows the the liquid products composition of upgraded sengon bio-oil. Liquid product from upgraded sengon bio-oil is dominated by ester compounds (methyl format), and other products such as carboxylic acid, ketones, and phenols. There are not found levoglucosan, aldehyde, and impurities compounds such as N. Compounds of 2-hydroxy-3-methyl-2-cyclopentene-1-one and 2-methoxy phenol are only present in thermal reaction products. That's maybe, in the catalytic reaction, both compounds disappear and undergo further reaction into other compounds. According to Bui et al. [23], 2-hydroxy-3-methyl-2-cyclopentene-1-one compound can be converted into 2-cyclopentene-1-one through a cracked pathway of the methyl groups and HDO by broken of CO bonds, while 2-methyl-2-cyclopentenone is formed through the HDO pathway by CO bonds broken and transfer methyl groups. Meanwhile, 2-methoxy phenol can be converted into phenol by demethoxylation (DMO) pathway of the Csp2-OMe bond or through a demethylation pathway (DME) of the ArO-Me bond.

The composition of upgraded coconut bio-oil is presented in Table 3, where the product contains esters, carboxylic acids, and phenols. The main content bio-oil from coconut woody biomass after pyrolysis is levoglucosan (Table 1), while the liquid from upgraded coconut bio-oil is an ester (methyl format) and a high amount of phenol. The high phenol compound in this product was due to the amount of phenolic derivatives compounds that found in the coconut bio-oil before the upgrading reaction relatively higher than another woody biomass bio-oils. Whereas levoglucosan, alcohol, ketone, aldehyde, and heteroatom (S and N) compounds didn't found in liquid products of upgraded coconut bio-oil.
Table 2. Distribution of liquid product components from upgraded sengon bio-oil (% area).

| Compound               | Catalyst       |
|------------------------|----------------|
|                        | Thermal | Co-Mo/USY | CoMo/USY | Mo-Co/USY |
| Ester                  |          |           |          |           |
| Methyl formate         | 75.01   | 75.01     | 86.41    | 89.75     |
| Carboxylic Acid        |          |           |          |           |
| Propanoic acid         | 9.78    | 8.05      | 9.34     | 6.76      |
| Ketone                 |          |           |          |           |
| 2-hydroxy-3-methyl-2-Cyclopent-1-one | 2.97 | -         | -        | -         |
| 2-Cyclopent-1-one      | -       | 1.54      | -        | -         |
| 2-Methyl-2-cyclopentone-1-one | - | 2.9      | -        | -         |
| Phenolic               |          |           |          |           |
| 2-methoxy Phenol       | 2.98    | -         | -        | -         |
| Phenol                 | 2.99    | -         | 5.75     | -         |

Table 3. Distribution of liquid product components from upgraded coconut bio-oil (% area).

| Compound               | Catalyst       |
|------------------------|----------------|
|                        | Thermal | Co-Mo/USY | CoMo/USY | Mo-Co/USY |
| Ester                  |          |           |          |           |
| Methyl formate         | 71.15   | 81.19     | 82.3     | 77.53     |
| Carboxylic Acid        |          |           |          |           |
| Propanoic acid         | 4.7     | -         | 3.58     | 4.23      |
| Phenyl acetate         | 3.61    | -         | -        | -         |
| Phenolic               |          |           |          |           |
| Phenol                 | 18.69   | 16.96     | 12.27    | 16.39     |

Compound distribution of upgraded meranti bio-oil is presented in Table 4. The main compound product is an ester (methyl format), and other products are carboxylic acids, ketones, and phenols. Levoglucosan, ketones, alcohols, aldehydes, and impurities compounds that contained N elements disappeared in upgrading catalytic of meranti bio-oil. Meanwhile, ketones are only a product from upgrading bio-oil by thermal.

Table 4. Distribution of liquid product components from upgraded meranti bio-oil (% area).

| Compound               | Catalyst       |
|------------------------|----------------|
|                        | Thermal | Co-Mo/USY | CoMo/USY | Mo-Co/USY |
| Ester                  |          |           |          |           |
| Methyl formate         | 79.9    | 83.78     | 91.1     | 81.38     |
| Carboxylic Acid        |          |           |          |           |
| Propionic acid         | 7.56    | 7.99      | 7.55     | 9.66      |
| Ethylene diacetate     | 2.51    | -         | -        | -         |
| Ketone                 |          |           |          |           |
| 2-Methyl-2-cyclopentone-1-one | 6.13 | -        | -        | -         |
| Phenolic               |          |           |          |           |
| Phenol                 | 2.55    | 6.87      | -        | 7.61      |
Compounds distribution of thermal and catalytic upgraded bio-oil from bangkirai woody biomass presented in Table 5. Similar to the previous discussion, it is found that the main compound from upgraded bangkirai bio-oil is esters. Then, other products are carboxylic acids, ketones, furan, phenols, and bicyclic compounds. The thermal products of upgraded bangkirai bio-oil are ketones and furan compounds. Meanwhile, in the bangkirai bio-oil product which improved by using Co-Mo/USY as a catalyst, there is a high percentage of Juniper camphor and trans-caryophyllene which are both bicyclic compounds. The formation of this bicyclic compound shows a C-C coupling reaction [24]. The C-C coupling reaction products will increase at low hydrogen pressure due to low residence time [25]. The percentage of phenol compounds in the liquid product which is upgraded bio-oil from bangkirai woody biomass is high. That is caused by the content of phenolic compounds is relatively high at the feed of bangkirai bio-oil which is 27.4%. The presence of trans-Caryophyllene which is an oxygen-free bicyclic compound indicates the occurrence of a hydrodeoxygenation reaction.

| Compound       | Thermal | Co-Mo/USY | CoMo/USY | Mo-Co/USY |
|----------------|---------|-----------|----------|-----------|
| Ester          |         |           |          |           |
| Methyl formate | 49.25   | 49.25     | 71.56    | 74.1      |
| Carboxylic Acid|         |           |          |           |
| Propanoic acid | 9.83    | -         | 8.31     | 11.14     |
| 3,7-Dimethyl-2,6-octadienoic acid | - | 18.74 | - | - | |
| Ketone         |         |           |          |           |
| 1-Hydroxy-2-Butanone | 1.65 | - | - | - |
| 2,3-Pentanediene | 2.57 | - | - | - |
| 2-hydroxy-3-methyl-2-Cyclopenten-1-one | 2.75 | - | - | - |
| Furan          |         |           |          |           |
| 2-Acetylfuran  | 1.43    | -         | -        | -         |
| 5 Methyl Furfural | 3.81 | - | - | - |
| 2-Furancarboxaldehyde | 2.63 | 3.34 | - | - |
| Phenolic       |         |           |          |           |
| 2-methoxy-Phenol | 4.56 | - | - | - |
| Phenol         | 16.44   | 6.45      | 18.72    | 13.35     |
| Bicyclic       |         |           |          |           |
| trans-Caryophyllene | - | 4.17 | - | - |
| Juniper camphor | - | 25.05 | - | - |

Esters which are the dominant products in liquid products of HDO bio-oil can come from the esterification reaction of carboxylic acids with alcohol compound in bio-oil [8,26]. In addition to esterification reaction, methyl formate can also be a hydrocracking product obtained through the C-C bond breaking of ester compounds in bio-oil, because USY has high acidity and has a large amount of Brønsted acid site [27] so that it leads to hydrocracking reactions. Esters which are the dominant products in upgraded bio-oil come from the esterification reaction of carboxylic acids with alcohol compound in bio-oil [8,26]. By the way, methyl formate can be obtained from hydrocracking pathway, through the C-C bond breaking of ester compounds in bio-oil, because USY has high acidity and has a large amount of Brønsted acid site. Pu and cowokers [27] say that the high acidity property leads to a
hydrocracking reaction. Methyl formate is a levulinic acid derived compound that can use as an additive in fuels such as gasoline [11]. Meanwhile, levulinic acid is a product from the conversion of furan compounds [28] or/and a dehydration product of sugar and cellulose that used an acid catalyst [11]. The high amount of the product of low molecular weight carboxylic acid, because the carboxylic group is less reactive and requires an HDO reaction temperature higher than the reaction temperature in this study, which is more than 300 °C[11]. This shows that the reaction leads to a hydrocracking reaction, rather than a deoxygenation reaction. The nature of hydrocracking comes from a combination of the hydrogenation properties of Co sites and the cracking properties of the USY sites, while the deoxygenation reaction comes from Mo sites. The combination of hydrogenation and cracking properties are stronger than deoxygenation, and the content of oxygenated compounds is imbalanced with the number of Mo sites, cause the catalyst to be ineffective for deoxygenation reactions.

Alcohol, aldehydes, ketones, and ethers are more susceptible to HDO reactions than esters and carboxylic acids. Therefore, these compounds did not found in liquid products from bio-oil upgrading reactions. Hydrocarbon compounds are also not found in liquid products, because the hydrocarbon products formed are very light compounds, even as gases, that cannot be detected. According to Corma et al. [10], the product compounds can be C1-C4 hydrocarbon compounds, which are the product of cracking reactions using USY as catalysts. The disappearance of levoglucosan compound, which is the main compounds of bio-oil, indicated that HC or HDO reactions occur. Levoglucosan which had five oxygen components converts to methyl ester which has two elements of oxygen. Meanwhile, the impurities compounds which contain elements of nitrogen and sulfur had removed. It is indicated that the three catalysts have HDN and HDO activity.

The reaction mechanism in HDO bio-oil is very complicated. This is caused bio-oil contains highly complex organic compounds. So, it is possible that the bio-oil constituent compounds will compete with each other to absorb on the surface of the catalyst, and even react to each other. One example of an unstable compound in bio-oil is an aldehyde, which can react with other bio-oil components during storage [11]. Meanwhile, there are several bio-oil components that can inhibit the reaction on the surface of the catalyst, such as compounds that is containing sulfur or nitrogen.

3.2.2. Yield Total of Upgrading Bio-oil from Sengon, Coconut, Meranti, dan Bangkirai woody biomass

Figure 1 shows Mo-Co/USY, Co-Mo/USY and CoMo/USY catalyst activities in bio-oil upgrading reactions. The use of catalysts in the improvement of meranti and bangkirai bio-oil caused a significant increase in activity, but the increase in bio-oil coconut and sengon did not occur. This is possible caused of the 4-hydroxy benzenesulfonic acid compounds content in bio-oil coconut and sengon is poisoning or disabling the active site of the catalyst. The interaction between sulfur and the surface of the catalyst causes a decrease in the activity and selectivity of the catalyst. Sulfur will cover the active site of the catalyst and reduce the number of reactants that the active site can absorb and also weaken the bonds between metal and reactants [29].
The activity of Co-Mo/USY catalyst was higher than CoMo/USY catalyst, in the upgrading reaction of bio-oil from sengon, coconut, and meranti woody biomass. This is possible because Co-Mo/USY catalysts contain more Mo metal than Co-Mo/USY catalysts. A large amount of Mo causes higher HDO activity, that is, by breaking C-O bonds. Meanwhile, the three types of bio-oil such as bio-oil from sengon, coconut, and meranti woody biomass, contain esters that are more amount than bangkirai, while esters are relatively more difficult to involvement in HDO reactions. Hence the increased reaction of sengon and meranti bio-oil requires a catalyst containing more Mo metal or catalyst with a higher Mo: Co ratio. However, Co-Mo/USY catalyst has lower activity than CoMo/USY in the reaction of upgrading bio-oil from bangkirai woody biomass.

The Mo-Co/USY, Co-Mo/USY and CoMo/USY catalysts have HDO activity for upgrading quality bio-oil with meranti> coconut> sengon> bangkirai sequences. The difference yield for each bio-oil is due to the difference of types and number of bio-oil components. HDO yield of bio-oil meranti is relatively higher than other bio-oils because in bio-oil meranti there is no sulfur-containing compound that can deactivate the catalyst. Besides the sulfur, compounds which contain nitrogen can also deactivate the catalyst [30]. The presence of compounds which contain nitrogen in meranti bio-oil is relatively less compared with other bio-oils, it means the compound components that can interfere with the reaction of HDO from bio-oil meranti decreased. In the reaction of HDO, bio-oil bangkirai has a relatively lower yield than other bio-oils. The low number of co-reactant compounds in bio-oil bangkirai affects the low yield of HDO from bio-oil bangkirai because co-reactant compounds will act as H donors for HDO reactions [31].

3.2.3. Selectivity of Co-Mo/USY, CoMo/USY, and Mo-Co/USY Catalyst toward Methyl Formate

The reaction of upgrading bio-oil of sengon, coconut, meranti, and bangkirai woody biomass, using Mo-Co/USY, Co-Mo/USY and CoMo/USY catalysts, has the main product, methyl. However, in the upgrading reaction, Co-Mo/USY catalyst has lower selectivity than Mo-Co/USY and CoMo/USY catalyst caused by more varied types of particles or in Co-Mo/USY catalyst. The different of particles types provide the different catalytic active sites that allow the different types of reactions to take place. Therefore, the catalyst becomes less selective for one particular reaction type. In general, the

![Figure 1. Correlation of catalyst types to the yield of liquid products from upgrading bio-oil of sengon, coconut, meranti and bangkirai woody biomass.](image_url)
CoMo/USY catalyst has high selectivity of the methyl format formation, because its particles types are more uniform than the others.

![Figure 2. Correlation of catalyst type and the selectivity of the Methyl format on upgrading bio-oil from sengon, coconut, meranti, and bangkirai woody biomass.](image)

The highest selectivity of methyl format shows that the catalytic upgrading of bio-oil tend to hydrocrack reactions, namely the cracking of C-C bonds. The hydrocracking reaction, which is more dominant in upgrading bio-oil, shows that the role of USY and Co metal are more dominant in the catalyst. While, the Mo metal as a deoxygenation site becomes less functional, so it is ineffective to break the CO bonds on the oxygenate compound from bio-oil. In accordance with Huber et al.[11], the reaction that occurs in upgrading bio-oil with zeolite catalyst is a cracking reaction. Besides that, the very high acidity of Mo-Co/USY (8.304 mmol/g), Co-Mo/USY (10.849 mmol/g) and CoMo / USY (11.206 mmol/g) catalysts[19] are also supporting factors for the dominance of bio-oil hydrocracking reactions. The high acidity of the catalyst shows the high number of Bronsted acid sites and Lewis acid sites on the catalyst. USY is a supporting material with a large number of Bronsted acid sites [27,32], where the Bronsted acid site can act as a proton donor [33]. Therefore, the large amount of hydrogen that can be donated by this catalyst will direct the reaction more dominantly against hydrocracking.

4. Conclusion
The upgrading of woody biomass-derived (bio-oil) has been carried out through HC levoglucosan reaction into methyl formate and the reaction of HDO from oxygenated phenol derivatives into phenol. Each bio-oil has different components so that it can cause each catalyst to have different optimal activity and selectivity in each bio-oil. The catalyst with optimal activity in the reaction of upgrading bio-oil from meranti woody biomass was Co-Mo/USY, which was equal to 72.39%. Meanwhile, the highest HC selectivity in the formation of methyl formate was Mo-Co/USY catalyst, which was 93.00%.

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