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KOVAR TUBE AS A POTENTIAL CATALYST FOR CONVERSION OF TAR PRODUCED FROM BIOMASS GASIFICATION

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Abstract. A pre-oxidized Kovar tube was employed as a reforming catalyst for the conversion of naphthalene. Under dry reforming condition, 24.7 % naphthalene conversion could be achieved, whereas 36.6 and 42.3 % naphthalene conversion could be achieved when steam was added to the producer gas at the volume ratio of 0.06 and 0.11, respectively. Increasing the reforming temperature to 1173 K enhanced the catalytic removal of naphthalene to 91.5 %. The activation energy and frequency factor values were found to be 136 kJ/mol and $3.07 \cdot 10^7$ $cm^3 \cdot cm^{-2} \cdot min^{-1}$, respectively.

Keywords: Kovar tube, tar, naphthalene, catalyst, gasification.

1. Introduction

Biomass gasification has been viewed as a promising strategy for renewable energy production.[1,2] It has been considered as one of the best technologies for substituting fossil fuels.[3] Biomass gasification is one of the promising routes for hydrogen production, such a fuel is considered a clean energy carrier.[4,5]

A major problem associated with biomass gasification is the production of tar and particulates. The presence of such contaminants along with the producer gas induces technical problems during the operation of downstream process equipment such as gas engines and turbines.[6,7] Engine or turbine applications require the use of a producer gas with low tar levels. The use of catalyst downstream the gasification zone might assist in gas reforming and tar destruction. Synthetic catalysts have been widely researched for the sake to effectively destruct tar into lower molecular weight compounds and for producer gas reforming. Examples of synthetic catalysts are: nickel-based catalysts[8] and iron-based catalysts.[9] However, the high production cost and the running cost are the main drawback for using these synthetic catalysts in biomass gasification, which may hinder the technology toward commercialization. Several types of naturally occurring catalysts have been proposed for catalytic biomass gasification, such as limestone, dolomite, and olivine.[10] These catalysts are relatively cheaper than synthetic catalysts.[11] In general, natural catalysts might exhibit either lower activity, thermal instability at high temperature operations, poor mechanical properties, or rapid deactivation.

Metal-containing alloy tubes have been exploited as potential catalysts for hydrogen production. Yu *et al.*[12] carried out glucose gasification experiments in supercritical water in capillary tube reactors. The researchers indicated that the yield of the product gases was strongly influenced by the type of reactor wall being Hastelloy C276 or Inconel 625 capillary tubes. Taylor *et al.*[13] utilized a tubular reactor made from Inconel 625 for hydrogen production *via* reforming of methanol in supercritical water. The researchers reported an enhanced reforming of methanol due to the tube reactor catalytic properties.

The catalytic activity of the metal-containing alloy tubes can be improved by high temperature oxidation pretreatment. de la Rama *et. al.*[14] utilized SUS304 alloy tube as a catalyst for hydrocarbon reforming. The SUS304 alloy tube was oxidized at 1273 K and then used as a catalyst for tetradecane reforming. The researchers reported that the pre-oxidized SUS304 alloy tube exhibited a catalytic activity toward tetradecane partial oxidation and CO_2 reforming.

Tagawa *et al.*[15] investigated the promotion effect of several nickel containing alloys as potential catalysts for reforming model tar compounds. Metal alloy tubes made from Hastelloy, Superinvar, SUS304 and Inconel were first calcined under oxygen flow at 1273 K for 2 h then used as catalysts for tetradecane partial oxidation. The

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researchers reported noteworthy high activity of Hastelloy and Superinvar toward tetradecane partial oxidation.

De la Rama *et al.*[16] evaluated the performance of a pre-oxidized Hastelloy tube as a catalytic for steam reforming of several model tar compounds. The catalyst revealed good activity and stability during tetradecane steam reforming. On the other hand, lower activity and increased rate of carbon formation were noticed during naphthalene steam reforming. In another investigation, de la Rama *et al.*[16] investigated the effect of pre-oxidation temperature of Kovar tube on its catalytic activity. The pre-oxidized Kovar tube was evaluated as a potential catalyst for CO_2 reforming of tetradecane at 1073 K. The researchers reported that pre-oxidation at 973 and 1273 K were the optimum temperatures for producing CO and H_2, respectively.

Jiwanuruk*et al.*[17] evaluated the performance of a pre-oxidized Hastelloy tube as a catalyst for steam reforming of methanol. The catalyst revealed good activity under when the catalyst was subjected to oxidation condition at 1273 K for 2 h.

The objective of this study is to investigate the catalytic conversion of a model tar compound, namely: naphthalene contained in a simulated producer gas from wood gasification process. A pretreated Kovar tube will be employed as a reforming catalyst and its catalytic activity will be evaluated under different operating conditions.

2. Experimental

Fig. 1 shows a schematic drawing of the experimental setup.

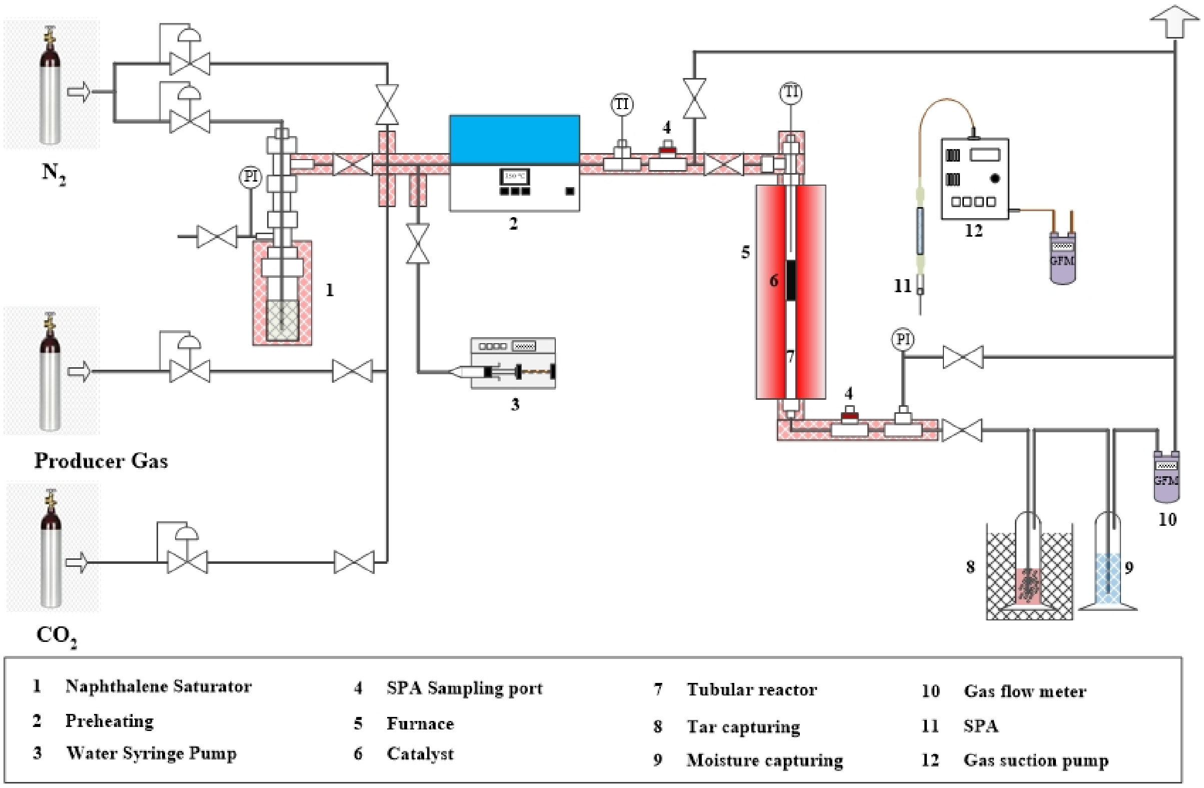

Fig. 1. Experimental apparatus

The catalytic reforming of the producer gas and tar decomposition experiments were carried out in a tubular stainless-steel reactor (12 mm ID). The reactor was housed in an electric heating furnace. The tube catalyst was inserted as a bed within the reactor. The temperature of the catalyst bed was monitored and controlled via a PID controller.

Producer gas from a wood gasification process. A naphthalene saturator heated by means of a mantle heater

A simulated producer gas was used in the experimentation. The producer gas comprised a mixture of gases namely: H_2, CO, CO_2, CH_4, O_2, and N_2. Naphthalene was selected as a model tar compound as it is one of the most stable tars, hence very difficult to remove. In this study, an input naphthalene concentration of 1g/m^3 was used as a typical naphthalene concentration in a was used to generate naphthalene vapor. The naphthalene vapor was swept by a nitrogen stream, then mixed with the simulated producer gas stream. The resulting gas stream was diluted with another nitrogen stream to have the desired producer gas and tar composition. All lines were traced at the temperature of 503 K by means of ribbon heaters to prevent any condensation in the tubes. Water was added to the producer gas stream by means of a syringe pump. The resulting gaseous stream was preheated to the temperature of 653 K.

The reformed gas stream is subjected to tar and moisture removal prior to analysis. Samples of the reformed gas are periodically collected from the downstream line using tedlar sampling bags. The tar-free gas is offline analyzed by two micro-GC 3000 (Agilent Technologies). Detailed description of the gas instrumental analysis system is provided elsewhere.[7]

Naphthalene is sampled by the Solid Phase Adsorption (SPA) method.[18] SPA columns having the size of 3 mL are used (SampliQ Amino, Agilent Technologies). For each sample, 100 mL of the gas are withdrawn from the sampling port at the rate of 50 mL/min for 2 minutes by using gas sampling pump (GSP-250FT, GASTEC Co., Yamato). The captured tar is recovered from the SPA column by elution with 2 mL dichloroethane under positive pressure from a syringe. The collected solution is subjected to quantitative analysis using a GC/MS system (Shimadzu Co.). Three standard solutions are used to produce a calibration curve for naphthalene.

Kovar tube (OD = 5.5 mm) having a composition of 29 wt% Ni, 17 wt% Co and a balance of Fe is used as a catalyst. The Kovar tube was cut into four segments (10 cm each). A squire-pitch configuration was adopted to accommodate the four tube segments within the tubular reactor.

3. Results and Discussion

3.1. Pretreatment of Kovar tube

Oxidation of the Kovar tube segments was carried out under oxygen flow of 100 mL/min. The tube furnace was programmed to increase the temperature at the rate of 50 K/min, then maintaining a final temperature of 1273 K for two hours. Fig. 2 shows the oxidized Kovar tubes.

The formation of scales on the surface of the tubes as a result of the high temperature treatment increased the roughness and surface area. As the Kovar surface is oxidized at high temperatures, it is expected that three types of iron oxides form: FeO, Fe_2O_3, and Fe_3O_4. Owing to its loose structure, several studies into Kovar oxidation revealed that the Fe_2O_3 did not exist.[19] High temperature treatment oxidized Fe_2O_3 to Fe_3O_4. XRD analysis of Kovar tube revealed an increase in Fe_3O_4 peaks when it was oxidized at higher oxidation temperatures.[16] Yates *et al.*[20] investigated the structure of the oxide layer that formed on Kovar wires after they were oxidized in air at 973 and 1073 K. According to the researchers, the oxide that accumulated on the Kovar wire was a layered structure made up of many different oxides. The top layer was verified to be $(Fe,Co)_3O_4$ using XRD, XPS, SEM, STEM, and Raman techniques. The catalytic activity of the oxidized Kovar tube is attributed to the presence of the catalytically active sites, namely: Ni and Co dispersed on the oxide layer.

Fig. 2. Kovar tubes pretreated by high temperature oxidation

3.2. Catalytic steam reforming of naphthalene

3.2.1. Effect of steam addition

Prior investigations on tar removal under thermal reforming conditions (no catalyst), indicated that the addition of steam had no noticeable effect on tar conversion.[6] When the steam to carbon ratio at a gasification-thermal reforming condition was raised from 0 to 2, the concentration of naphthalene remained nearly constant. This finding is in line with other research which suggests that steam has only a minor impact on aromatics thermal conversion.[21] Cheng *et al.*[22] reported that naphthalene was stable up to 1073 K under homogeneous thermal cracking with just 1.9 % conversion.

The use of appropriate reforming catalyst is recommended for tar conversion. Naphthalene may be reformed on a catalyst surface with either steam or carbon dioxide, or both, producing additional carbon monoxide and hydrogen as proposed by equations 1 and 2.[23]

$$C_{10}H_8 + 10H_2O \rightarrow 10CO + 14H_2 \tag{1}$$

$$C_{10}H_8 + 10CO_2 \rightarrow 20CO + 4H_2 \tag{2}$$

Fig. 3 shows the catalytic naphthalene conversion as a function of steam to producer gas volume ratio.

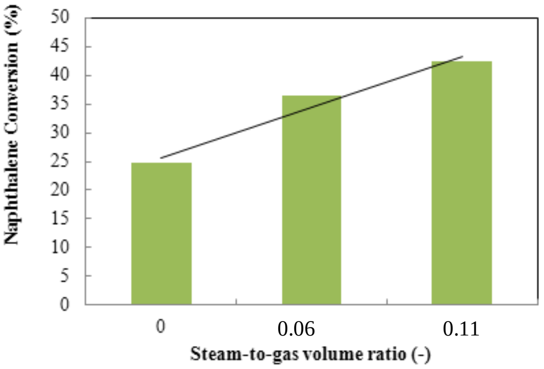

Fig. 3. Catalytic naphthalene conversion as a function of steam to producer gas volume ratio (T = 1023 K, 15.8 % H_2, 9.4 % CO, 4.8 % CO_2, 1.3 % CH_4, 0.6 % O_2, and balance N_2)

An inspection of the results shown in Fig. 3 indicates that the addition of steam improved the catalytic naphthalene conversion. About 24.7 % naphthalene conversion was accomplished under dry reforming conditions, while 36.6 and 42.3 % naphthalene conversions were achieved when steam was applied to the producer gas at volume ratios of 0.06 and 0.11, respectively.

A study carried by Buchireddy *et al.*[23] indicated close range of naphthalene conversions as those reported in this study. The researcher reported catalytic steam conversion of naphthalene in the range of (19 ~ 55) % using different types of zeolite catalysts.

3.2.2. Effect of CO_2 addition

Fig. 4 shows the effect of CO_2 on naphthalene conversion when it is added to the producer gas.

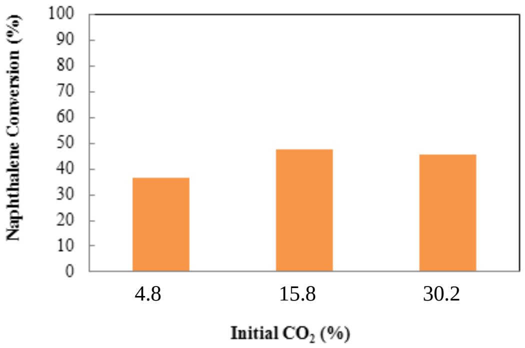

Fig. 4. Catalytic naphthalene conversion as a function of steam initial CO_2 concentration (T = 1023 K, 15.8 % H_2, 9.4 % CO, variable % CO_2, 1.3 % CH_4, 0.6 % O_2, and balance N_2,0.06 steam-to-gas volume ratio)

Increasing CO_2 concentration in the producer gas from 4.8 to 15.8 % increased naphthalene conversion from 36.6 to 47.8 %. However, further increase in the CO_2 concentration had no effect on naphthalene conversion.

3.2.3. Effect of reforming temperature

Fig. 5 shows the conversion of naphthalene as a function of reactor temperature.

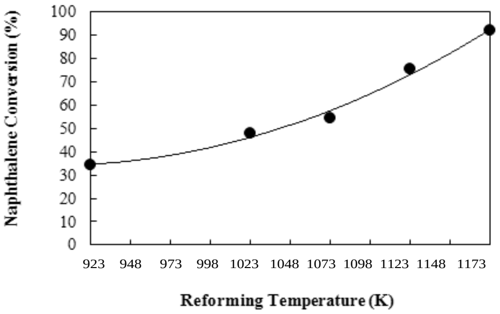

Fig. 5. Naphthalene conversion as a function of reforming temperature (15.8 % H_2, 9.4 % CO, 15.8 % CO_2, 1.3 % CH_4, 0.6 % O_2, and balance N_2, 0.06 steam-to-gas volume ratio)

Increasing the reactor temperature increased naphthalene conversion. Higher reactor temperatures augmented naphthalene conversion more profoundly than at lower temperatures. The catalytic removal of naphthalene was increased to 91.5 % by raising the reforming temperature to 1173 K. Min *et al.*[24] reported the tar yields of a biomass under reforming at various temperatures with and without ilmenite (a natural iron-containing mineral). The tar yield decreased with increasing temperature, regardless of whether an ilmenite catalyst was used, due to increased thermal cracking and potential homogeneous reforming. Aljbour and Kawamoto[6] reported substantial decrease in naphthalene concentration in a biomass producer gas when the thermal reforming temperature increased from 1023 to 1223 K. Di Carlo *et al.*[25] used a nickel-mayenite catalyst for tar reforming in a raw fuel gas from biomass gasification. Temperature plays an important role in the decomposition of tars, according to the researchers. As the temperature was elevated from 973 to 1073 K, the conversion of total hydrocarbons and each tar class improved.

3.3.Kinetics

Studying the kinetics of naphthalene conversion is crucial for proper operational process design. In addition, it provides an indication for the catalyst performance when compared with other catalytic systems.

It has been widely accepted to assume first order kinetics for the rate of catalytic tar conversion.[26-28]The rate of tar conversion has been presented in terms of catalyst weight or catalytic empty bed volume. In this study, the rate of tar removal has been represented in terms of catalyst surface area:

$$-r'_{tar} = k_{app} C_{tar} \tag{3}$$

where $-r_{tar}$ is the rate of tar removal defined as tar removal speed per unit surface area of the catalyst [mg/min · m^2], k_{app} is the apparent rate constant [m^3/min · m^2], and C_{tar} is tar concentration [mg/m^3].

The tar conversion, X, is calculated as follows:

$$X = \frac{C^{o}_{tar} - C_{tar}}{C^{o}_{tar}} \tag{4}$$

where C^{o}_{tar} is tar input concentration [mg/m^3]

$$-r'_{tar} = k_{app} C^{o}_{tar}(1 - X) \tag{5}$$

The apparent rate constant, k_{app} , in a tubular reactor under plug flow conditions is estimated as follows:

$$k_{app} = [-ln(1 - X)]/\tau \tag{6}$$

$$\tau = \frac{A}{v_o} \tag{7}$$

where τ is the time constant based on catalysts surface area [m^2 · min/m^3], v_o is the gas flow rate based on reactor condition [m^3/min] and A is the surface area of the catalyst.

The activation energy E [kJ/mol] is estimated based on Arrhenius' law:

$$k_{app} = Aexp\left(\frac{-E}{RT}\right) \tag{8}$$

where A is the frequency factor [m^3/(m^2·min)], R is the universal gas constant [8.314 J/(K·mol)] and T is reactor temperature [K].

Fig. 6 shows the apparent reaction rate constant as a function of temperature.

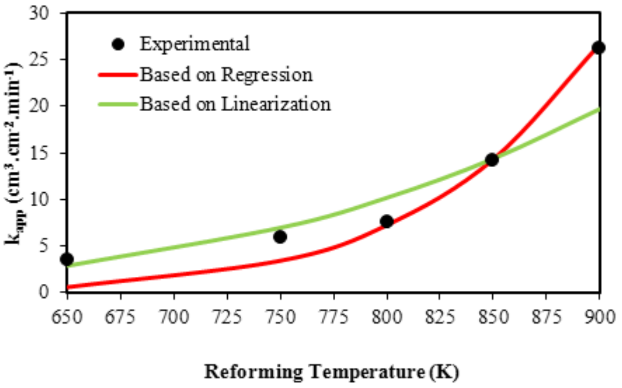

Fig. 6. The apparent reaction rate constant as a function of reactor temperature
(15.8 % H_2, 9.4 % CO, 15.8 % CO_2, 1.3 % CH_4, 0.6 % O_2, and balance N_2,0.06 steam-to-gas volume ratio)

The activation energy E and the frequency factor A are estimated based on Arrhenius' law using the linearized form of the law, and by means of non-linear regression through the minimization of the sum of the squared error (SSR). The results of the two fitting models are also shown in Figure 6 and the fitting parameters are given in Table 1.

Table 1. The activation energy E and the frequency factor A for the catalytic conversion of naphthalene

	Linearized Model	Non-linear regression Model
E (kJ/mol)	69	136
A (cm^3·cm^{-2}·min^{-1})	$2.71 \cdot 10^5$	$3.07 \cdot 10^7$
SSE	49.93	15.28

Model prediction based on kinetic parameters obtained by means of non-linear regression possessed a minimum SSE value compared to the linearized model. The activation energy and the frequency factor by means of non-linear regression are 136 kJ/mol and $3.07 \cdot 10^7$ cm^3 · cm^{-2} · min^{-1}, respectively.

Table 2 shows a comparison of the activation energy for different catalysts used for naphthalene conversion.

Table 2. Activation energy for different catalysts used for naphthalene conversion

Catalyst	E (kJ/mol)
Thermal conversion in the presence of H_2O and H_2 (No catalyst)	350[21]
Char	61[29]
Pretreated olivine	187[30]
Ni-Fe-dolomite	63.96[31]
Coal char	71[32]
Coconut char	72[32]
Dried sewage sludge char	63[32]
Oxidized Kovar tube	136 (This study)

The results indicate that the use of the pre-oxidized Kovar tube catalyst substantially reduced the activation energy required for naphthalene conversion. When looking to its low cost and high mechanical stability, then the Kovar tube catalyst would be a reliable choice for catalytic tar removal in pyrolytic and gasification processes.

4. Conclusions

A pre-treated Kovar tube was successfully employed as a tar-removing catalyst. High temperature treatment under oxygen flow formed mixed metal oxides layer on the surface of the Kovar tube. The formation of metal oxides layer on the surface of tube promoted catalytic activity toward naphthalene conversion. The catalytic promotion of the Kovar tube in the removal of naphthalene was evident under steam reforming, CO_2 reforming and thermal reforming conditions. The activation energy needed for naphthalene conversion was significantly reduced when the Kovar tube catalyst was used. The Kovar tube catalyst will be a good option for catalytic tar removal because of its low cost and high mechanical stability.

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ТРУБКА З КОВАРУ ЯК ПОТЕНЦІЙНИЙ КАТАЛІЗАТОР ДЛЯ ПЕРЕТВОРЕННЯ ДЬОГТЮ, ОТРИМАНОГО В РЕЗУЛЬТАТІ ГАЗИФІКАЦІЇ БІОМАСИ

***Анотація**. Попередньо окиснена трубка з ковару використана як каталізатор риформінгу для перетворення нафталену. За умов сухого риформінгу можна досягти конверсії нафталену 24,7%. За додавання до генераторного газу водяної пари в об'ємному співвідношенні 0,06 і 0,11 досягається конверсія нафталену 36,6 і 42,3%, відповідно. Підвищення температури риформінгу до 1173 К збільшує каталітичне видалення нафталену до 91,5%. Величини енергії активації та передекспоненційного множника становлять 136 кДж/моль і* $3{,}07\cdot10^7$ *см*^{3}*·см*$^{-2}$*·хв*$^{-1}$*, відповідно.*

***Ключові слова**: трубка з ковару, дьоготь, нафтален, каталізатор, газифікація.*