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Catalytic Removal of Tar from Biomass Producer Gas in a Microwave Reactor: Its Effectiveness and Kinetics

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ABSTRACT

The tar in biomass producer gas is problematic and needs to be removed before use as fuel. This study investigated tar removal by using Ni/Mg/Y-Zeolite as an upgraded catalysts in a microwave reactor. The wet impregnation method was used to prepare the upgraded catalyst. Catalytic cracking of tar provided the highest about 99% tar removal, with only little carbon deposition on the catalyst. The data gave credible estimates of activation energy, and the fitted kinetic model had SEE=1.5% and R^2= 0.95. Moreover, the experimental data satisfied overall mass balance to 96% accuracy.

1. Introduction

Global warming, eventual depletion of fossil fuels, and emissions of greenhouse gases are current acknowledged problems [1]. Alternative energy sources are actively sought due to these concerns [2,3]. Biomass is a renewable energy source that can provide clean energy [4,5]. Biomass gasification has been studied for many years with the increasing emphasis on sustainable energy. Biomass gasification is a thermochemical process converting solid biomass into useful fuel gas (producer gas, as distinct from natural gas) and solid char [6]. The biomass producer gas from gasification could be utilized for heat and power generation with gas turbines, IC engines, or fuel cells [7]. However, direct use of biomass producer gas has a high tar concentration [8] that tends to cause equipment problems on use as fuel, and impacts also the gasification [7,9]. "Tar" has been defined as a mixture of condensable organic compounds or hydrocarbon complexes. It is thick, black, highly viscous liquid, and can condense in low-temperature zones of the gasifier. Thus, treating the tar in producer gas is necessary [10,11]. The tar concentration in producer gas typically depends on the type of gasifier,

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namely downdraft, updraft, or fluidized bed type, that respectively give approximately 1, 10 and 100 g/m^3 concentrations [12].

There are two types of tar removal methods: 1) primary, with tar removal within the gasifier; and 2) secondary with tar removal outside the gasifier [9]. Although the primary methods can reduce tar contamination in producer gas, the removal is not sufficient without applying secondary measures [13]. The secondary methods can be divided into physical/mechanical, catalytic cracking, and thermal cracking. Particularly catalytic decomposition or cracking of tar is of high interest as the tar can be converted to a useful gas mix, containing H_2 and CO that can improve the energy content of producer gas [7,14]. Furthermore, in terms of energy consumption the complete tar removal by thermal cracking requires high temperatures of about 1,200-1,300 ° C and is more energy intensive (and more expensive) than catalytic cracking at 500 - 900 ° C [15,16]. Thus, catalytic cracking of tar appears to be an appropriate and economical technology for treating tar in producer gas from biomass gasification [17].

Many studies have shown that the catalytic removal of tar in biomass gasification can use several kinds of catalysts, such as nickel-based catalysts, non-nickel metal catalysts (Rh, Ru, Pd –Nobel catalysts), alkali metal catalysts, natural catalysts, acid catalysts, and activated carbon catalyst [8]. The nickel-based catalysts are very successful in decomposing tar, and especially Ni/Al_2O_3 has shown good efficiency and activity in tar removal, but these catalysts are rapidly deactivated by fouling known as coking [18]. Non-nickel metal catalysts are the best in performance and stability in tar reduction, with also low coke formation, but they are very expensive [19,20]. Alkali metal catalysts also show good tar removal and improve the producer gas quality, but they rapidly evaporate during the gasification [21]. Natural catalysts such as dolomite and olivine are broadly used, being inexpensive and easily available, but these catalysts have the lowest activities. Silica-alumina, Zeolite, etc. are acid catalysts that have been recommended for improving producer gas quality by removing tar. The advantages of acid catalysts include high thermal stability, low price, and easy regeneration, but the main problem is rapid inactivation by coke formation [14,16]. Activated carbons or chars are low-cost catalysts that are naturally produced in gasification, having high specific surface with acidic groups. Thus, activated carbons are good for catalytic cracking, but the blocking of pores by coke formation remains a problem [8]. Among these catalysts, nickel-based and acid catalysts are the most interesting for tar removal, although coke formation can rapidly deactivate them. Many studies have reported that nickel or magnesium loaded onto catalysts has improved catalytic activity and stability in tar cracking, while avoiding carbon deposition and coke formation [21–24]. Anjaneyulu *et al.,* [22] mentioned that also other metals on catalysts can improve catalytic activity and stability.

As mentioned above, tar treatment by catalytic cracking gives useful gases (H_2, CO, and CH_4) from the decomposition [7] at reaction temperatures in 500-900 ° C range. The selection of heating method is also an important factor. Microwave heating has several advantages over conventional heating, such as wide range of temperature generation, volumetric and contactless heating, controllable heating process and high heating rate (not limited by speed of thermal diffusion), thus it was applied in the field of biomass gasification tar treatment [25–28]. In addition, several studies reported the superior tar removal efficiency by using microwave heating compare to methods using conventional heating. [29]

In this study, microwave irradiation was used in the tar removal system. An upgraded catalysts was evaluated in removal of tar from producer gas generated in a laboratory-scale downdraft gasifier from wood pellets. The effects of an upgraded catalyst (Ni/Mg/Y-Zeolite) and reaction temperature on the removal of tar were investigated, along with LHV and gas yield. Further, a kinetic model of catalytic cracking of tar in the producer gas was fitted to the data, which was validated by comparison to prior published studies. The satisfaction of mass balance was also evaluated.

2. Materials and Methods

2.1 Materials

Commercial wood pellets with 0.6 cm diameter and 3 cm length were used as the biomass to generate producer gas in a downdraft gasifier. The proximate and ultimate analyses of the wood pellets were determined by ASTM standard methods are shown in Table 1.

Table 1
Properties of wood pellets

Parameter	Value	Unit
Proximate analysis		wt % dry basis
Fixed carbon	14.3	
Volatile matter	76.2	
Ash	0.5	
Moisture	9	
Elemental analysis		wt % dry ash-free basis
C	44.55	
H	7.54	
N	0.06	
S	0.17	
O	47.67	
HHV	17.8	MJ/kg

2.2 Catalyst Preparation

Y-Zeolite catalyst of commercial grade was prepared through wet impregnation following Liu *et al.,* [30]. The Y-Zeolite catalyst was dried in an oven at 120°C overnight to remove moisture. The dry catalyst was then immersed for 24 h in magnesium nitrate solution at room temperature, and then it was calcined in a furnace for 3 h at 500 °C and cooled to room temperature. Finally, the catalyst (Mg/Y-Zeolite) was immersed in nickel nitrate solution as a metal precursor, and further treated as above.

2.3 Experimental Apparatus Setup

A schematic of the experimental apparatus system is shown in Figure 1. The three units in the system perform 1) biomass gasification; 2) tar decomposition; and 3) tar sampling. The details are described next.

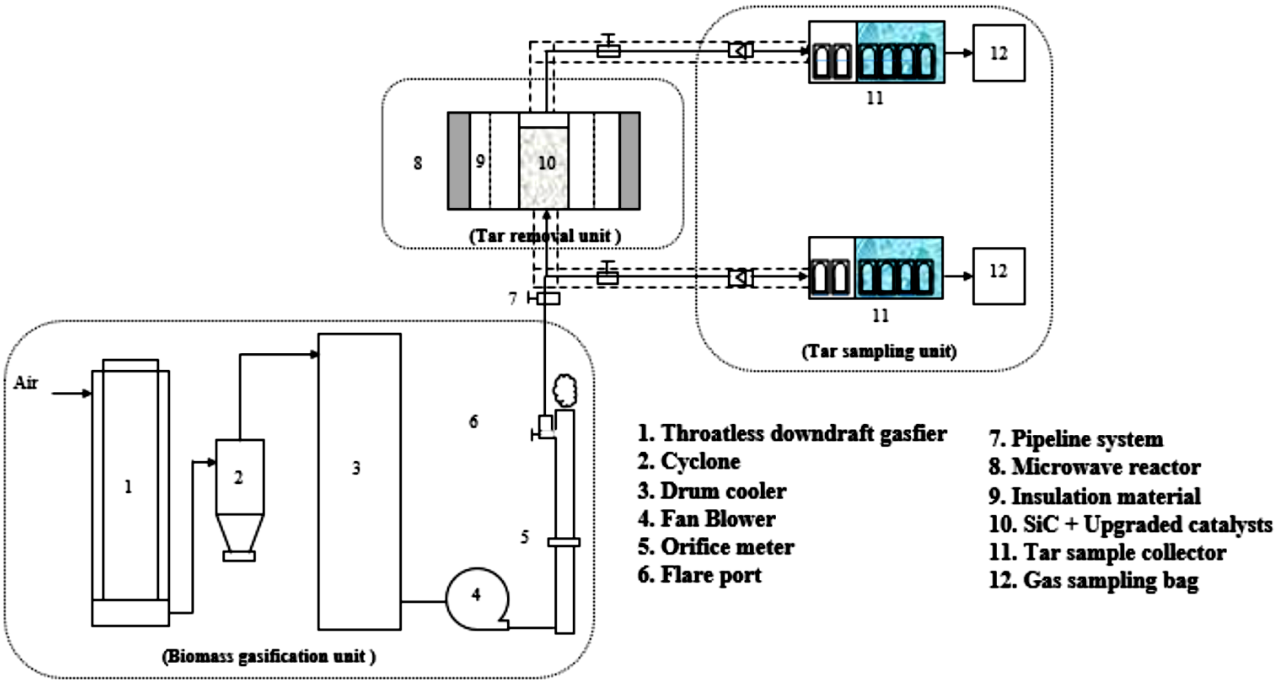

Fig. 1. A schematic diagram of the experimental setup for catalytic decomposition of tar in producer gas generated from biomass

2.3.1 Biomass gasification

The biomass gasification system includes a laboratory-scale throttled fixed bed downdraft gasifier, cyclone, drum cooler, blower, and gas flare. The gasifier has an inner diameter of 150 mm and a height of 1,500 mm. It can produce 8.3 kW_T thermal power output depending on the biomass feed rate, at 5 kg/h. The blower fan moves the gas from gasifier to the gas flare. This system also includes an orifice meter attached to the outlet pipe, with measurement of the pressure drop to indicate flow rate of the producer gas.

2.3.2 Tar decomposition

The biomass tar is decomposed in a reactor within a microwave oven. The microwave oven (Panasonic, NN-SM330M) was modified by drilling holes on top and bottom plates of the oven, in order to install a reactor tube within the microwave chamber as shown in Figure 2**.** The 2450 MHz microwave power output is maximally 700 W at 1125 W power consumption. The reactor tube is 16 cm tall with 2.54 cm internal diameter and 0.5 cm thickness. It was vertically fixed in the microwave chamber. SiC (Silicon carbide) of F10 grade in FEPA standard, with 1.48 g.cm^{-3} bulk density and 2085 µm grain size was used as the susceptor material absorbing microwaves and converting the energy to heat. The temperature was measured with a type K thermocouple. Feedback control of temperature was used to maintain it at desired level in the reactor.

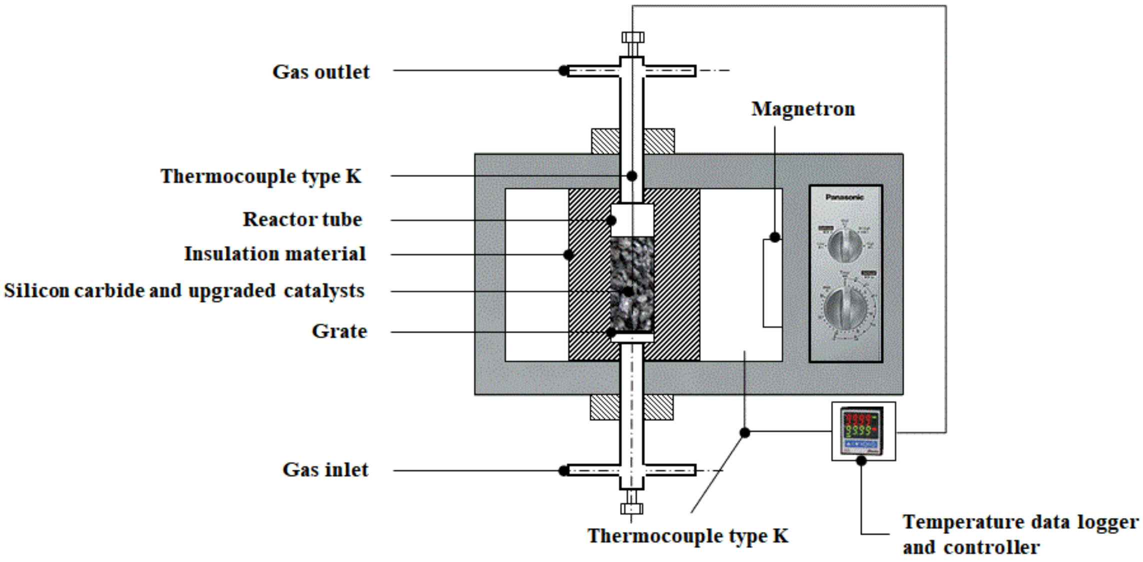

Fig. 2. A modified microwave oven of tar removal reactor

2.3.3 Tar sampling

The tar sampling system includes a tar collector train, vacuum pump, and flow meter. It was modified from the guidelines given by Neeft *et al.,* [31] for tar and by-products in producer gas. The train had six bottles in series with the first two bottles at room temperature, and the others placed in an icebox containing ice and salt for about -22 o C temperature, to condense tar as described by Bergman *et al.,* [13]. Each bottle had 50 ml capacity, and the first five bottles had 50 ml of isopropanol while the last bottle was blank. The producer gas was pulled from the gas flare into the tar removal system, and then to the tar collector train, by suction from a vacuum pump. A flow meter was included to measure and control the flow rate of producer gas.

2.4 Experimental Procedures

2.4.1 Experimental parameters

Table 2 presents the experimental parameters for catalytic decomposition of tar. The temperatures were in the range 500-700 °C. The residence time was fixed at 0.24 s by gas flow rate set at 3.8 LPM.

Table 2
The experimental parameters for catalytic decomposition of biomass tar

Parameter	Value	Unit
Catalyst mass	3	g
Biomass producer gas flow rate	3.8	LPM
Catalyst bed height	3	cm
Residence time	0.24	s
Temperature	500-700	°C
Microwave power	0.7	kW

2.4.2 Catalytic cracking

This study focused on tar decomposition in biomass producer gas by catalytic cracking. The raw producer gas from biomass gasification flowed into the microwave oven for treatment. The bed in the silica tube reactor had three layers: silicon carbide (SiC), SiC+ upgraded catalysts, and SiC. Each experiment had 3 g of the upgraded catalyst mixed with 25 g of SiC. The residence time was fixed at 0.24 s. The catalytic bed temperature for reaction was varied in the range 500–700 °C [8].

2.4.3 Sampling and analysis

The producer gas passing through the reactor was sampled before and after it, with samples run into the tar collector train having isopropanol for tar condensation. The condensate samples were combined and filtered through Whatman filter paper into a flask. The yield of particles was from differential weight of the filter paper. The filtered isopropanol was treated in a BUCHI rotary to get the tar residue that was weighed. The tar conversion efficiency was calculated as follows [32]**.**

$$\text{Biomass tar conversion efficiency (\%)} = [(\text{Tar}_{\text{mass in}} - \text{Tar}_{\text{mass out}})/(\text{Tar}_{\text{mass in}})] \times 100 \quad (1)$$

The clean gas after treatment in tar sampling train was collected in a gas sampling bag and analyzed by gas chromatography (GC–TCD, the Hewlett Packard Module 4890) to determine the amounts of H_2 and CH_4. The used catalyst was tested to i in a thermogravimetric analyzer (TGA).

2.4.4 Kinetic model of biomass tar from producer gas

A kinetic model of biomass tar conversion in the experimental setup was fit to the data, to interpret the experimental results. Such model could also assist in optimizing the tar cracking operation, or in forecasting the reaction behavior [33]. The kinetic model of biomass tar conversion by catalytic decomposition follows Anis and Zainal [33].

The conversion rate of biomass tar in producer gas under catalytic decomposition with Ni/mg/Y-Zeolite is given by:

$$-\mathrm{r}_T = -\frac{dF_T}{dT_R} = kF_T^n \quad (2)$$

where k is the rate constant, and F_T is the remaining condensed tar mass.

Assuming first order kinetics (n = 1), Eq. (2) can be integrated with the initial condition of $F_T(0) = F_{T,0}$ (initial mass of tar model) to provide the tar content:

$$\mathrm{F}_T = F_{T,0}\exp(-kT_R) \quad (3)$$

It is necessary to determine the extent of tar conversion (X_T) and this can be determined as follows:

$$\mathrm{X}_T = 1 - \exp(-kT_R) \quad (4)$$

where X_T is defined as:

$$X_T = \frac{(F_{T,0} - F_T)}{F_{T,0}} = 1 - \frac{F_T}{F_{T,0}} \tag{5}$$

By rearranging Eq. (4), the reaction rate constant at a particular residence time (T_R) can be stated as follows:

$$k = \frac{-ln(1 - X_T)}{T_R} \tag{6}$$

It is assumed that this rate constant in its dependence on temperature follows the Arrhenius's equation:

$$k = k_0 \exp\left(-\frac{E_a}{RT}\right) \tag{7}$$

where k_0 is a pre-exponential factor (s^{-1}), E_a is the activation energy (kJ/mol), R is universal gas constant (0.008314 kJ/mol.K) and T is the reaction temperature (K).

Substituting Eq. (6) into Eq. (7) gives:

$$\frac{-\ln(1 - X_T)}{T_R} = k_0 \exp\left(-\frac{E_a}{RT}\right) \tag{8}$$

or

$$ln\left(\frac{-\ln(1 - X_T)}{T_R}\right) = ln(k_0) - \frac{E_a}{RT} \tag{9}$$

2.4.5 Mass balance

To assess the validity of experimental data, mass balance was checked. The input stream (M_i) for the catalytic decomposition of tar consists of raw producer gas, tar, particles, and water (moisture content in producer gas) while the output stream (M_o) consists of purified producer gas, tar, particles, water and coke deposited on catalyst. The conservation of mass in this cracking process of biomass tar in producer gas requires:

$$\sum M_i = \sum M_o \tag{10}$$

3. Results and Discussion

3.1 Biomass Gasification

The raw producer gas from a laboratory-scale throttled fixed bed downdraft gasifier was used in this study. The gasification of biomass gives producer gas including not only valuable gases such as hydrogen, nitrogen, oxygen, methane and carbon dioxide, but also tar, water, and particulates [34]. Table 3 lists the producer gas components with related data.

Table 3
The products of producer gas from a throatless downdraft gasifier

Parameter	Value	Unit
Gasifier condition		
Equivalence ratio (ER)	0.26	
Low heating value (LHV)	4.32	MJ/Nm
Products yields at sample point		wt.%
Gas	92.96	
Tar	0.34	
Water	6.65	
Particulates	0.05	
The composition of producer gas		vol.%
Hydrogen	10.23	
Oxygen	3.14	
Carbon monoxide	19.5	
Methane	2.12	
Carbon dioxide	10.25	
Nitrogen	54.76	

3.2 Catalytic Activity Test
3.2.1 Catalytic cracking of biomass tar

The cracking of biomass tar by Ni/Mg/Y-Zeolite as the modified catalyst was tested at temperatures in the range 500-700 ° C with a residence time of 0.24 s. When the producer gas flows through the catalyst, the tar can be transformed to gaseous products by several reactions. Known reactions during catalytic cracking of tar are shown in Eq. (11) to Eq. (18) [9,35].

Steam reforming reaction: $C_nH_m + nH_2O \rightarrow \left(n + \frac{m}{2}\right)H_2 + nCO$ (11)

Dry reforming reaction: $C_nH_m + nCO_2 \rightarrow 2nCO + \left(\frac{m}{2}\right)H_2$ (12)

Steam reforming of methane reaction: $CH_4 + H_2O \rightarrow CO + 3H_2$ (13)

Water gas shift reaction: $CO + H_2O \rightarrow CO_2 + H_2$ (14)

Dry reforming of methane reaction: $CH_4 + CO_2 \rightarrow 2CO + 2H_2$ (15)

Water gas reaction: $C + H_2O \rightarrow CO + H_2$ (16)

Methanation reaction: $C + 2H_2 \rightarrow CH_4$ (17)

Boudouard reaction: $C + CO_2 \rightarrow 2CO$ (18)

Figure 3 to 5 show the activities of tar cracking. As shown in Figure 3, at 500° C it seems that most gas components increased while O_2 and H_2 slightly decreased from the initial amounts in the raw producer gas. Figure 4 and Figure 5 present the concentrations of tar and particles that were reduced from initial levels in the raw gas from 2.63 g/Nm^3 to 0.65 g/Nm^3 and from 0.40 g/Nm^3 to 0.09 g/Nm^3, respectively. It was found that both tar and particles are decomposed by 75-78 % at this temperature.

The contents of H_2, CO_2, and CO steadily increased as the reaction temperature was increased from 500 to 600 °C, while CH_4 gradually decreased with temperature. These results could be attributed to water gas reaction and methane reforming reaction, as reported for a previous study [7]. This conforms with prior studies [24,36] in which H_2 and CO concentrations increased three-fold with temperature due to the high effectiveness of Ni loaded Y-Zeolite.

Turning now to Figure 3, it can be seen that the yield of CO tends to decrease with temperatures beyond 600 °C, while the yields of H_2 and CH_4 still grew steadily. It could be reasoned that this reflects the water gas shift reaction and the methanation reaction, Eq. (6) to Eq. (7). As seen in Figure 4, at 900 ° C we observed that most of the tar was cracked by Ni/Mg/Y-Zeolite, about 98%. The final tar residual was 0.044 g/Nm^3, and the lowest particle content was about 0.009 g/Nm^3 corresponding to particle conversion of about 97 %.

The above results on tar and particle conversion (Figure 4 and Figure 5) show that the conversion efficiency significantly increased from 500° C to 650° C, and then slightly improved from 650° C to 700° C. This indicates that less coke was produced as temperature was increased, resulting in high conversion of tar and particles to useful gases.

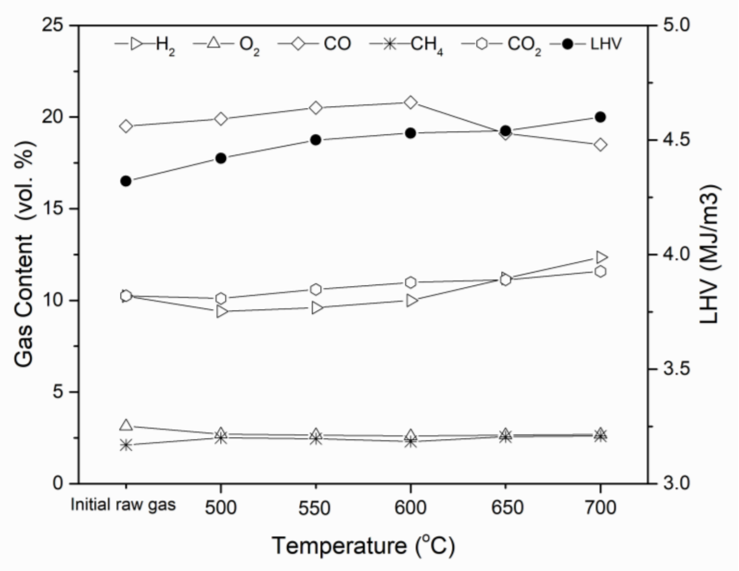

Fig. 3. Gas composition and LHV with catalytic cracking of biomass tar

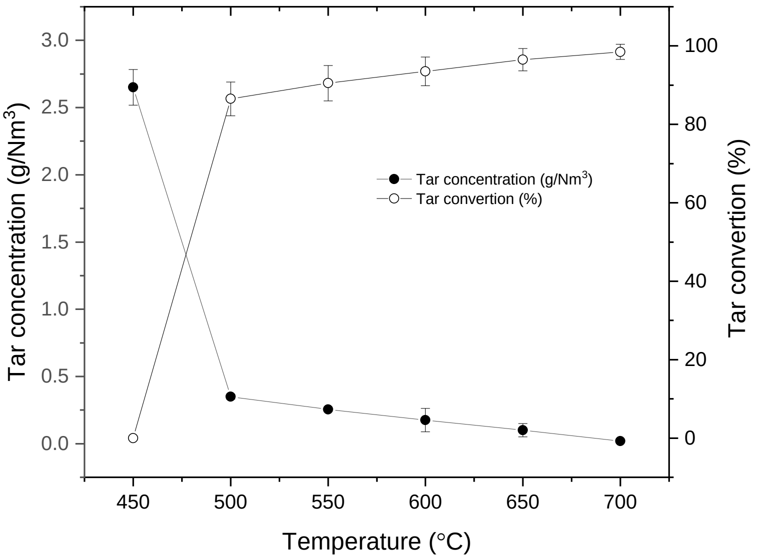

Fig. 4. Tar concentration and conversion efficiency in catalytic cracking of biomass tar

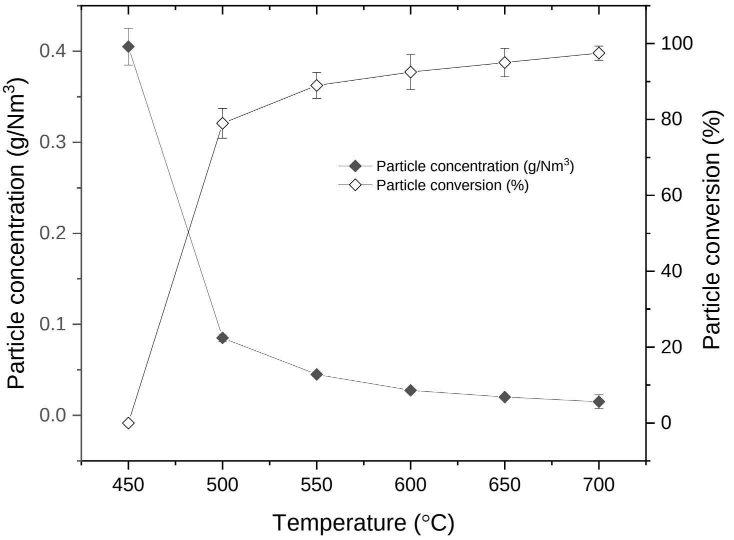

Fig. 5. Particle concentration and conversion efficiency in catalytic cracking of biomass tar

3.2.2 Coke formation

The coke deposition on the catalyst surface is shown in Figure 6, and it appears related to the tar removal efficiency. The deposition of coke (carbon) on catalyst, or coke formation, derives from Boudouard and Methane decomposition reactions, as mentioned by Wang *et al.,* [24]. The results in Figure 6 also demonstrate the smallness of coke condensed structure which is appeared at the low temperature of coke formation, while the coke components at high temperature are more condensed structure of coke, formed inside zeolite pores and deactivate zeolite activity by pore blockage [37,38].

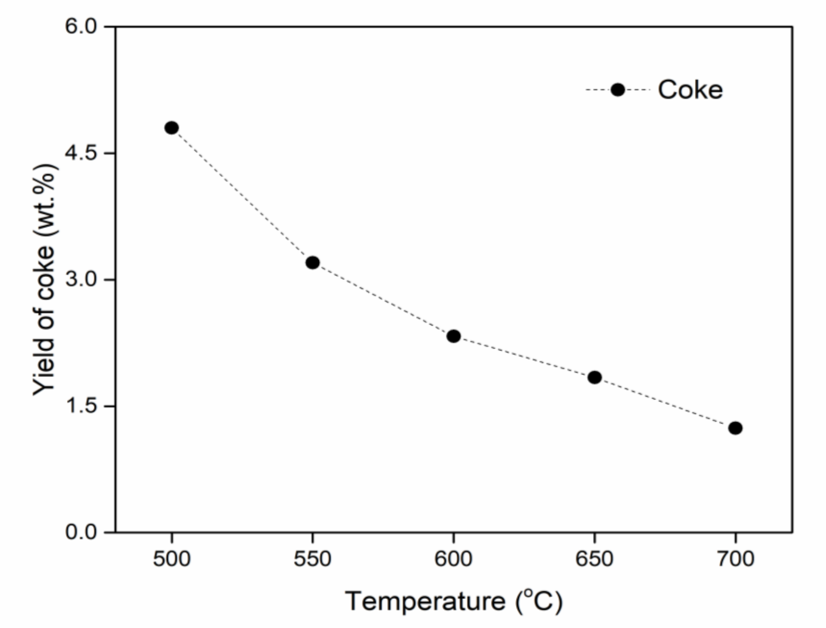

Fig. 6. Coke concentration

The atomic H/C ratio tends to decrease with temperature [39]. As described by Wang and Manos [40] increasing the temperature causes volatile coke precursors to go into the gas phase, whereas condensation is favored by low temperatures causing more coke formation. Furthermore, the declination of coke deposition on catalyst could be reasoned that mg loading on Ni/Y-Zeolite performs as promoter of carbon decreased on catalyst surface. Less fouling of catalyst also improves tar removal efficiency, and this agrees with previous studies [23,24].

3.3 Reaction Kinetics Model of Biomass Tar Reduction
3.3.1 The estimation of kinetic parameters

Fitting a common kinetic model both summarizes and validates the experimental results. Its assessment assists in design of a tar removal operation, helps optimize the conditional operation, or forecasts the behavior of tar removal reactions [33]. The assumptions made for modeling in this current work were: (1) plug flow of the gas phase; (2) steady state; (3) time independent thermal conditions; and (4) negligible increase in gas volume from catalytic conversion. The minimum energy required for reaction on collision of molecules is called the activation energy (E_a). The frequency/pre-exponential factor (K_0) in the Arrhenius equation reflects the rate of molecular collisions. Therefore, the reaction rate constant directly indicates the activity of catalyst, and also the reactivity of tar to be cracked [41].

The Arrhenius plot of the overall reaction rate in catalytic decomposition of tar by Ni/Mg/Y-Zeolite is presented in Figure 7. The kinetic parameters from the fitted straight line are listed in Table 4. From temperatures in range 500–700 °C and residence time of 0.24 s, the activation energy (E_a) and pre-exponential factor (k_0) found are 25 kJ/mol and 3.61 x 10^2 m^3/kg h, respectively.

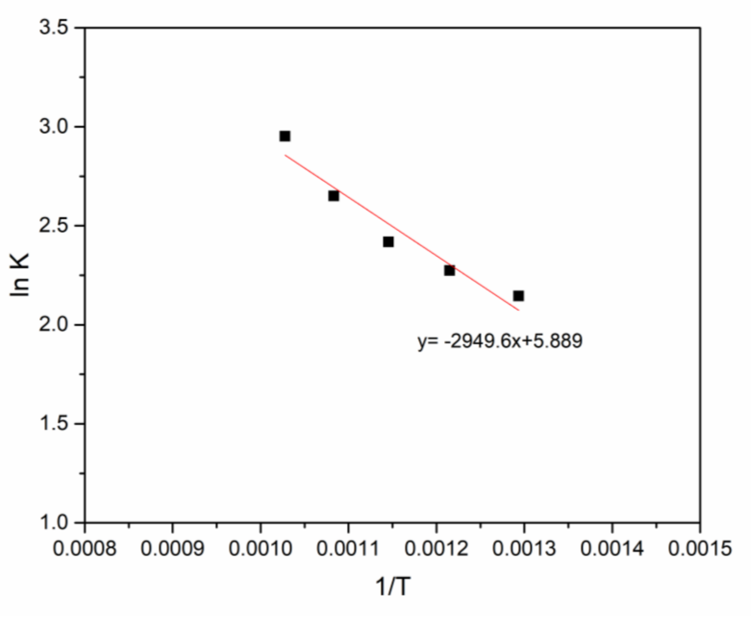

Fig. 7. The Arrhenius plot for E_a and K_0 estimates in biomass tar conversion

Table 4
Kinetic parameters of tar conversion in catalytic treatment

Parameter	Value	Unit
Activation energy (E_a)	25	kJ/mol
Pre-exponential factor (k_0)	3.61×10^2	m^3/kg·h
Temperature	500-700	^{0}C

The kinetic parameter estimates were compared with prior studies due to the strong relation of these parameters with tar conversion efficiency. As shown in Table 5, the activation energy of the upgraded catalyst used in the current study for tar conversion was lower than in the prior references, which is advantageous to the performance.

Table 5
A comparison of kinetic parameters in catalytic cracking of biomass tar

Types of catalysts (catalytic treatment method)	Temperature (^{0}C)	Gasifying agent	E_a (kJ/mol)	K_0 (m^3/kg·h)	Reference
Ni/Mg/Y-zeolite	500-700	Air	25	3.61×10^2	This study
Ruthenium	500-700	Air+Steam	25.61	5.77×10^2	Warsita [42]
Y-zeolite	500-700	Air	46	2.64×10^3	Anis and Zainal [7]
Dolomite	700-900	Air	68	1.01×10^4	Anis and Zainal [7]

3.3.2 Validation of the kinetic model

The kinetic model fitted to data on catalytic removal of biomass tar was assessed for goodness of fit. The standard error of estimate (SEE) used to assess the model fit was calculated as follows:

$$SEE = \sqrt{\frac{\sum_i^n \left(X_{Texperimental,i} - X_{Tmodel,i}\right)^2}{n-2}} \qquad (19)$$

where, $X_{Texperimental,I}$ and $X_{Tmodel,i}$ stand for the conversion efficiency of biomass tar from experiment and from the kinetic model, respectively. Figure 8 shows model predicted values against measured data. The coefficient of determination R^2 is 0.92, indicating good agreement of model with experimental data. The SEE of tar conversion is about 1.3 % and the fitted kinetic model reproduces the data fairly accurately.

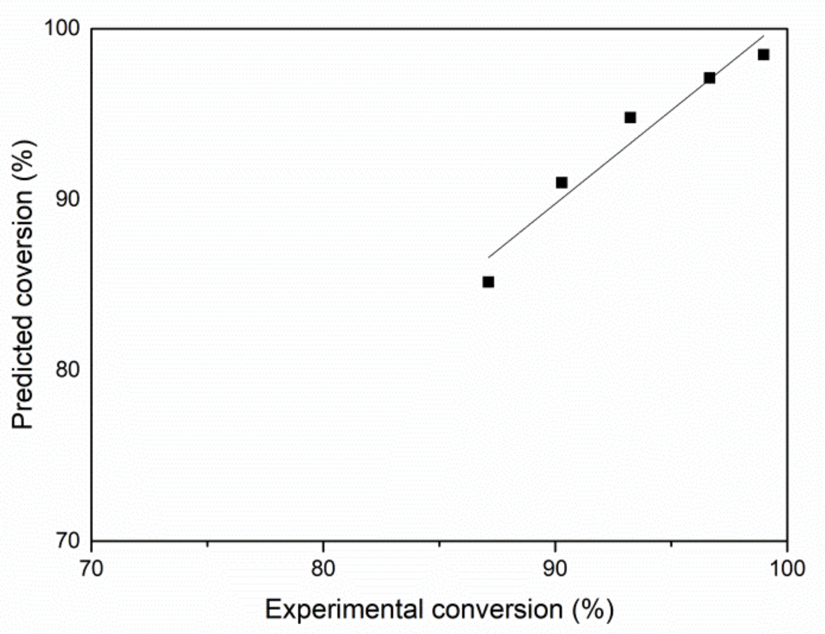

Fig. 8. Model predicted values versus measured data for catalytic treatment of tar

3.4 Mass Balance

The mass flow diagram for catalytic cracking of tar in biomass producer gas is given in Figure 9.

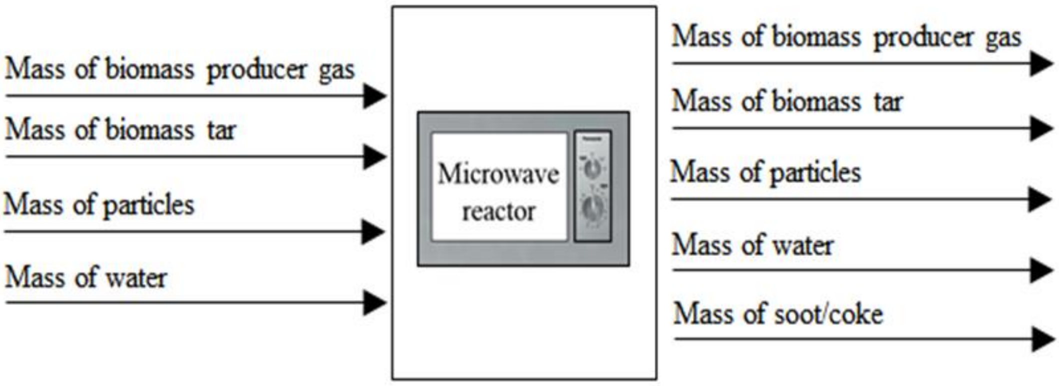

Fig. 9. Flows in the mass balance for catalytic cracking of tar

The flows in mass balance for catalytic decomposition of tar using Ni/Mg/Y-Zeolite were used in Eq. (10), and a summary of the mass balance is shown in Table 6. While from mass conservation we know that the mass balance must be satisfied, the inaccurate measurements from flows in experiments will not precisely conform to this requirement.

Table 6
Summary of the mass balance for catalytic decomposition of tar using Ni/Mg/Y-Zeolite

Components	Mass input (kg/h)	Mass output (kg/h)				
		500 °C	550 °C	600 °C	650 °C	700 °C
Dry producer gas	1.84E-01	1.850E-01	1.852E-01	1.850E-01	1.829E-01	1.813E-01
Biomass tar	6.728E-04	8.666E-05	6.535E-05	4.546E-05	2.245E-05	6.819E-06
Particles	1.034E-04	2.245E-05	1.137E-05	6.819E-06	4.830E-06	3.410E-06
Soot/coke	-	3.995E-05	3.547E-05	3.247E-05	2.979E-05	2.590E-05
Water	1.316E-02	7.017E-03	6.144E-03	5.837E-03	4.762E-03	4.582E-03
Total	1.981E-01	1.922E-01	1.915E-01	1.910E-01	1.877E-01	1.859E-01
Mass Balance closure (%)		97.03	96.68	96.41	94.75	93.86
Average mass Balance closure (%)		95.75				

Based on the mass balances in the above table, it can be seen that on average the mass balance was satisfied with 95.75 % accuracy. This corroborates the experimental data as valid and reliable.

4. Conclusions

The performance in biomass tar removal of Ni/Mg/Y-Zeolite upgraded catalyst was investigated in this study. The downdraft gasifier generated producer gas with LHV 4.32 MJ/Nm^3 and tar concentration 2.63 g/Nm^3. The upgraded catalyst gave the highest 99% tar removal at 700 ° C, when tested with the fixed 0.24 s residence time. Good tar elimination was associated with low coke formation in the stable upgraded catalyst. A fitted first order kinetic model gave slightly lower Arrhenius activation energy (E_a) than in prior references. The kinetic model fit the experimental data very well with SEE of 1.5% and R^2 of 0.95. The experimental measurements are also corroborated as valid by about 96 % accurate satisfaction of the overall mass balance in the experiments.

Acknowledgments

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