

Characterization of Tar Generated During Updraft Gasification of Woody Biomass in Auto-thermal Packed Bed Reactor

ABSTRACT

This study presents characteristics of tar formed during updraft gasification of biomass by using mass spectrometry and thermogravimetric analysis. Tar content in producer gas is higher at the lower-middle part of the reactor which represents a pyrolytic zone. Tar is composed of C, H, N and O by 70.62%, 10.62%, 0.57% and 18.20%, correspondingly and its HHV is around 35.27 MJkg⁻¹. ToF-MS analysis for ion mass-to-charge ratio (mz^{-1}) indicates high intensities from 280 mz^{-1} to around 380 mz^{-1} . In comparison with tar taken from the trap, tar samples captured along the bed heights have relatively low peaks at 500 mz^{-1} , 555 mz^{-1} and 615 mz^{-1} . Devolatilization of tar up to 700 K follows a similar trend regardless of presence or absence of oxidizing agent. Tar has about 22.17% fixed carbon content whose combustion and gasification at heating rate of 20 Kmin⁻¹ occurred above 700 K. Average devolatilization for volatile matter in tar during non-isothermal pyrolysis, combustion and gasification was about 2.29%min⁻¹. Average degradation of fixed carbon during non-isothermal combustion and non-isothermal gasification was found to be 3.45%min⁻¹ and 0.54%min⁻¹, respectively. For isothermal combustion and gasification at 1,273 K, degradation of fixed carbon was about 3.73%min⁻¹ and 0.95%min⁻¹, respectively.

Keywords: tar formation; packed bed; mass spectrometry; gasification.

1. INTRODUCTION

During thermal process such as pyrolysis and gasification volatile matters evolve from solid fuels. Decomposed volatiles can form gases or can undergo condensation and polymerization to form tar [1]. Tar refers to oxygenated aromatic constituents which can condense at low temperatures. Tar is composed of several polyaromatic hydrocarbon species. Compounds which make the major share of tar composition is benzene (C₆H₆), toluene (C₇H₈), o-xylene (C₈H₁₀) and naphthalene (C₁₀H₈) mixed in average ratio of about 4:1:1:1 [2]. Other constituents are indene, acenaphthylene, methylnaphthalene, pyrene, phenol and other phenolic compounds. It can be noted that benzene, naphthalene and pyrene are carcinogenic to humans while toluene is considered as a reproductive toxin [3].

Most of researchers in gasification field have preferred to work with downdraft gasification rather than updraft gasification [4,5]. This is due to relatively lower tar generation during downdraft gasification under which further tar cracking occurs as product gasses pass through the high temperature reduction zone packed with charred biomass [5,6,7]. As opposed to that, pre-mature escaping of product gasses under updraft gasification results into high tar content of the syngas. Nevertheless, updraft gasification produces syngas with favorably slightly higher lower heating value syngas [5,6,8]. In case of producer gas application in engine, tar can get deposited to cause engine troubles [9]. Basic requirement for syngas gas application in the engine is to have tar concentration less than 0.02 gm⁻³ [10]. Tar is also detrimental to gasification catalysts and its formation represents low conversion to gas and hence low gasification efficiency [5,6]. Polyaromatic hydrocarbon can also be formed during combustion of petroleum products [3,11].

Despite of being a hydrocarbon, tar does not find wider use in energy field. It is therefore vital to characterize tar with respect to its formation, composition and decomposition. Mass spectrometry (MS) techniques can be used for analysis molecular weight distribution for a sample composed of various compounds. Hossain *et al.* [12], conducted mass spectrometry determination of carcinogenic naphthalene, anthracene, phenanthrene and fluorene in river water. Herod *et al.* [13], studied molecular mass distributions and structural characterization of coal derived liquids by using mass

spectrometry. In addition, tar decomposition characteristics during combustion and gasification environments have to be understood. Thermogravimetric analysis is widely accepted for such studies.

This paper presents tar formation behavior during updraft gasification of biomass in addition to basic characteristics of tar. In addition to that, mass spectrometry of tar is presented as well as its thermogravimetric characteristics based on pyrolysis, combustion and gasification processes.

2. METHODOLOGY

2.1 Tar generation

Tar formed during updraft gasification of biomass was used for this study. Experimental set-up for updraft gasification from which the tar was captured is presented by Kihedu *et al.* [5]. Pelletized Japanese cedar was used as biomass sample and air or mixture of air and steam was used as gasifying agent. These pellets were about 8.5 mm long and, 6.5 mm in diameter. Proximate and ultimate analysis results for these pellets are presented previously [5,6]. Packed bed height was 1,000 mm and tar sampling ports were allocated along the height of the reactor as well as after reactor exit. Thus, tar samples were captured at 200 mm which represent the lower part of the reactor, 500 mm that is the middle height of the reactor and 800 mm representing upper part of the reactor but also the tar was sample after reactor exit. During the continuous operation modes of the reactor, biomass supply was set at 9 gmin^{-1} and stoichiometric combustion ratio was 0.351. Detailed information on gasification materials and methods can be found elsewhere [5,6].

2.2 Tar sampling procedure

Tar content was captured by using a set of three impingers cooled in the ice bath as described in Kihedu *et al.* [5]. Tar used for analysis of basic characteristics was taken after reactor exit. In addition to that, tar sampling was also done at various bed heights. Before tar sampling, empty beakers were dried in a constant temperature oven at 107°C for 3 hours and weighed. From the sampling ports, sampling lines were then connected to three impingers were filled with 150 mL of dichloromethane to dissolve the tar (Figure 1). Thereafter, all the impingers were disconnected from the sampling line. Tar deposited along the tubes in the sampling line, was rinsed by using dichloromethane and poured in to the beaker. Hence impingers and the collected tar in a beaker were dried at 107°C for 3 hours to vaporize dichloromethane as well as moisture contents [5,6,8]. Weight differences before the tar sampling and after vaporization; indicate tar content in the syngas. Tar measurement process was repeated for every position considered hence average values are presented.

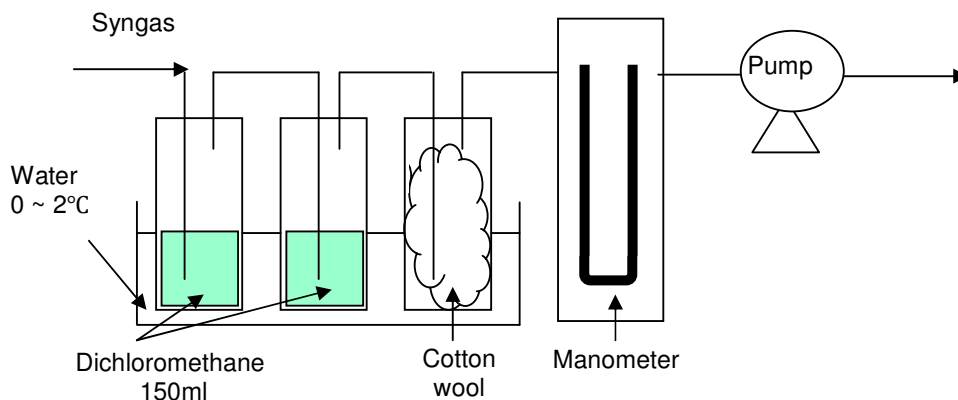


Fig. 1. Tar capturing impinge set-up

2.3 Elemental analysis

Elemental analysis was conducted by using Yamato CHN Coder. Antipyrène which is a standard material with known carbon, hydrogen and nitrogen composition was used to test coder calibration.

Then six samples, each with about 2 mg tar were used for analysis. Carbon, hydrogen and nitrogen composition were directly encoded from the CHN Coder while oxygen was obtained by mass balance.

2.4 Time-of-Flight Mass Spectrometry (ToF-MS)

About 10 mg of tar from the tar trap was thoroughly with 2 mL of dicloromethane. Then 4 μ L of the mixture was dissolved in 2 mL of ethanol as reagent gas. Thereafter, 200 μ L of the solution was sampled for ToF-MS analysis. Ionization of the solution, the analyte, was conducted by using Atmospheric Pressure Chemical Ionization (APCI) method in which the analyte from High Performance Liquid Chromatography (HPLC) was vaporized through a sprayer supplied with high flow rate N_2 as nebulizer gas and surrounded by a heater set at 673 K. Ionization was enabled by using atmospheric pressure corona discharge to produce ions for analysis (Figure 2). Ionization result in fragment ion useful for structural identification however such identification can be extremely difficult if fragmentation is too much as it is usually the case with APCI.

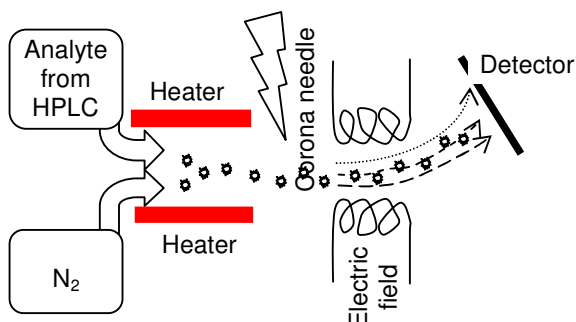


Fig. 2. Schematic diagram for Time-of-Flight Mass Spectrometer

Subjected to magnetic flux density B , ions with elementary charge e and charge number z , will be subjected to Lorentz force F_{lz} and velocity v and a deflection according to Fleming's left hand rule. Lorentz force F_{lz} and centrifugal force F_{cg} acting on the ions are given by;

$$F_{lz} = Bzev \quad (1)$$

$$F_{cg} = \frac{mv^2}{r} \quad (2)$$

Ions pass through magnetic field and reach the detector through a curved path of given radius, therefore Lorentz force F_{lz} and centrifugal force F_{cg} must be balanced.

$$\frac{mv^2}{r} = Bzev \quad (3)$$

Kinetic energy of the ion accelerated by voltage V is described as;

$$\frac{1}{2}mv^2 = zeV \quad (4)$$

Therefore;

$$\frac{m}{z} = \frac{ze^2r^2}{2V} \quad (5)$$

The m/z values can also be expressed as follows:

$$\frac{m}{z} = \frac{(M_w + nH^+)}{n} \quad (6)$$

where M_w is the molecular mass of the sample, n is the integer number of charge on the ions, H is the mass of a proton that is 1.008 Da.

Number of charges is usually not known but it can be calculated if it is assumed that any two adjacent members in the series of multiply charged ions differ by one charge. By keeping acceleration voltage V constant and varying or magnetic flux density B or vice versa, the detector can detect mass of ions. By measuring the time taken for the charged particle to travel to the detector, it is possible to determine its mass, or molecular mass distribution (MWD) when dealing with fluid or gas composed of various compounds. During this study, samples were analyzed for by monitoring the ion mass-to-charge ratio (mz^{-1}) between 260 and 630.

2.5 Thermogravimetric Analysis

Thermogravimetric analysis of about 10 mg tar samples was conducted by using Shimadzu Thermo-Gravimetric Analyzer (TGA). WORKlab water pump was used to supply distilled water to a steam generator set at 573 K. Heating rate from ambient temperature to 1,273 K was set at 20 Kmin⁻¹, however holding at 380 K for 10 minutes. To avoid steam condensation within TGA reactor at lower temperature ranges, only N₂ at 150 mLmin⁻¹ was supplied from ambient temperature to 473 K. Thereafter, N₂ flow was reduced to 75 mLmin⁻¹, and then air or steam at 75 mLmin⁻¹ was supplied to allow non-isothermal combustion or non-isothermal gasification reactions to proceed. N₂ was maintained throughout the combustion or gasification processes as purging gas and as a steam carrier.

Pyrolysis followed by isothermal combustion and isothermal steam gasification at 1,273 K, were conducted by using the same experimental setting. However, only N₂ at 150 mLmin⁻¹ was supplied from ambient temperature to 1,273 K and maintained for 10 minutes. After that, N₂ flow was reduced to 75 mLmin⁻¹, and air or steam at 75 mLmin⁻¹ was added to allow isothermal combustion or isothermal steam gasification to proceed.

2.6 Analytical definitions

According to Channiwala and Parikh [14], higher heating value HHV (MJ/kg), for solid, liquid or gaseous fuels can be found by the following correlation;

$$HHV = 0.3491C + 1.1783H + 0.1005S - 0.1034O - 0.0151N - 0.0211A \quad (7)$$

C, H, O, N and S represent carbon, hydrogen, oxygen, nitrogen and sulphur content of fuel where 0% ≤ C ≤ 92.25%, 0.43% ≤ H ≤ 25.15%, 0% ≤ O ≤ 50%, 0% ≤ N ≤ 5.6%, 0% ≤ S ≤ 94.08%, 0% ≤ A ≤ 71.4%. HHV value obtained may have absolute error of ±1.45% [14].

Conversion ratio X (%), for pyrolysis, gasification and combustion was derived from TGA data as follows;

$$X = (1 - m/m_o) \times 100 \quad (8)$$

where m_o (g) represent dry and ash free mass of the sample at initial condition while m (g) is the instantaneous residual mass of the sample.

Average reactivity R (%/min) was calculated between the two conversion ratios, X_1 and X_2 as follows;

$$r_{comb} = \int_{t_{x_1}}^{t_{x_2}} (((dX/dt) t dt)) / ((1 - X(t)) / (t_{x_2} - t_{x_1})) \quad (9)$$

where t_x is the time when conversion X was attained. In order to avoid obscuring of the global trend, X_1 and X_2 were taken to be 20% and 60% for non-isothermal devolatilization of volatile matter during pyrolysis, combustion and gasification. For non-isothermal and isothermal, combustion and gasification of fixed carbon in tar, X_1 and X_2 were taken as 85% and 95% conversion.

3. RESULTS AND DISCUSSION

3.1 Tar formation in packed bed reactor

During updraft gasification, tar content in producer gas was high at the lower-middle part of the reactor (Figure 3). This is the pyrolytic zone which occurs above the combustion and char reduction zone. At 500 mm bed height tar content reached 75.67 gm^{-3} . Tar content of about 9.56 gm^{-3} observed at 50 mm signifying incomplete pyrolysis at the middle part of the reactor where temperature ranges are medium. At upper part of the reactor, there was slight reduction of tar content. The reduction of tar at this low temperature region can be associated with tar deposition onto the packed fresh biomass, rather than thermal cracking [7,15] or catalytic effect of minerals in biomass [16,17]. At 950 mm bed height tar content reached 30.48 gm^{-3} . At the exit point from the gasifier, tar content in syngas from updraft gasification was about 28.23 gm^{-3} . It can be noted that updraft gasification produced syngas composed of 26.8% CO, 4.9% H_2 and 1.4% CH_4 therefore leading to the lower heating value (LHV) of about 4.43 MJm^{-3} . Cold gas efficiency was around 81.17% and carbon conversion reached about 96.24%. Analytical definitions for LHV, cold gas efficiency and carbon conversion have been reported [5,6].

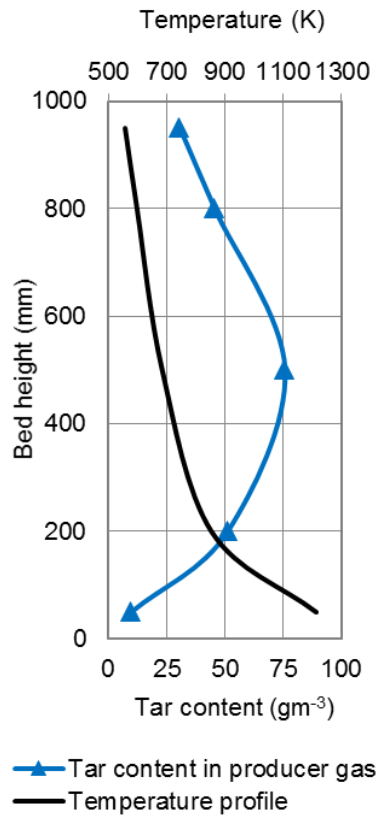


Fig. 3. Tar content in producer gas along the height of a packed bed reactor during updraft gasification

3.2 Basic characteristics of tar

In liquid form, density of the tar taken from tar exit was around 1.14 gcm^{-3} . Elemental analysis indicates that C, H, N and O percentages in this tar was 70.62%, 10.62%, 0.57% and 18.20%, correspondingly (Table 1). By using Equation 7, HHV for tar of was realized to be 35.27 MJkg^{-1} . This result is slightly lower than 40 MJkg^{-1} estimated by Thunman *et al.* [18]. It can be noted that HHV for biodiesel, diesel and petrol is around 42 MJkg^{-1} , 44 MJkg^{-1} and 47 MJkg^{-1} , respectively [19].

Table 1: Elemental analysis for tar from biomass

Test Number	C (%)	H (%)	N (%)	O (%) balance
1.	72.756	10.947	0.582	15.715
2.	68.971	10.375	0.552	20.102

3.	70.264	10.577	0.562	18.597
4.	71.792	10.772	0.576	16.860
5.	69.240	10.428	0.554	19.779
6.	71.495	10.713	0.574	17.218
Average	70.61	10.62	0.57	18.20

3.3 Mass spectrometry for tar

Figure 4(a) through Figure 4(c) shows that mass spectrometry analysis results for tar samples captured at 200 mm which represent the lower part of the reactor, tar at 500 mm i.e. at the middle height of the reactor and tar at 800 mm which representing upper part of the reactor. Also, the tar from gasification by using air was sample after reactor exit (Figure 4(d) and hence compared with tar generated upon introduction of steam at reactor free board area (Figure 4(e)). These figures present mass-to-charge ratio mz^{-1} of the ionized tar compounds against their relative intensities. It can be noted that, mass-to-charge distribution is an indication of molecular weight distribution of the sample in consideration.

All figures show high intensities from 280 mz^{-1} to around 380 mz^{-1} which signifies relatively high concentration of tar compounds with low molecular weight in comparison with low concentration of high molecular compounds in tar. Significant change in distribution of relative intensive of low molecular weight is observe for tar formation along the reactor height. Also, tar samples captured at 200 mm, 500 mm and 100 mm bed heights (Figure 4(a) through Figure 4(c)) have relatively low peaks at 500 mz^{-1} , 555 mz^{-1} and 615 mz^{-1} compared to tar taken after reactor exit (Figure 4(d) and (Figure 4(e)).

No significant change in intensities were observed from introduction of steam for tar taken as compared to gasification by using air. However, concentration of tar compounds with low molecular weight was slightly reduced for tar captured from gasification by using air-steam mixture (Figure 4(d) and Figure 4(e)). This phenomenon suggest that steam contributed to further thermal cracking of tar compound with low molecular weight as reported by [5,6,8].

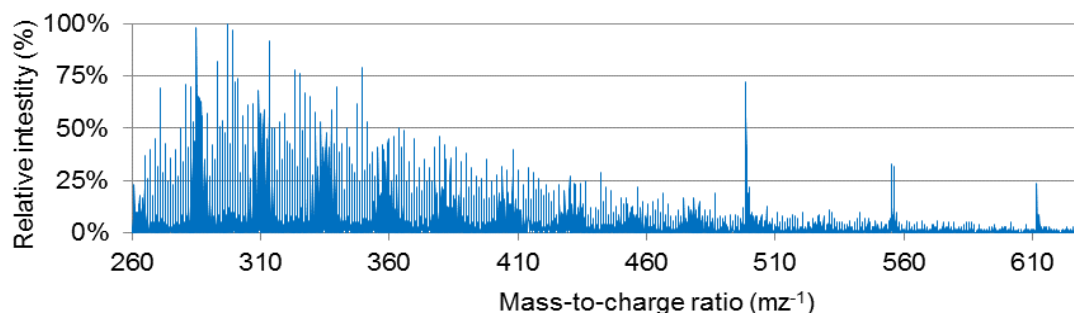


Fig. 4(a). Molecular weight distribution for tar captured at 200 mm bed height during updraft gasification

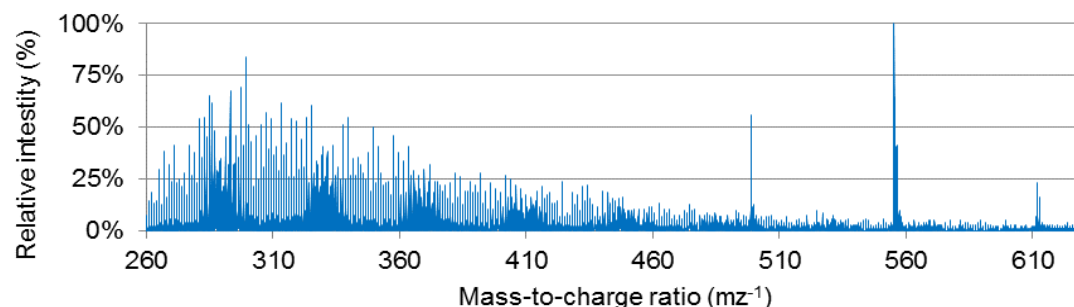


Fig. 4(b). Molecular weight distribution for tar captured at 500 mm bed height during updraft gasification

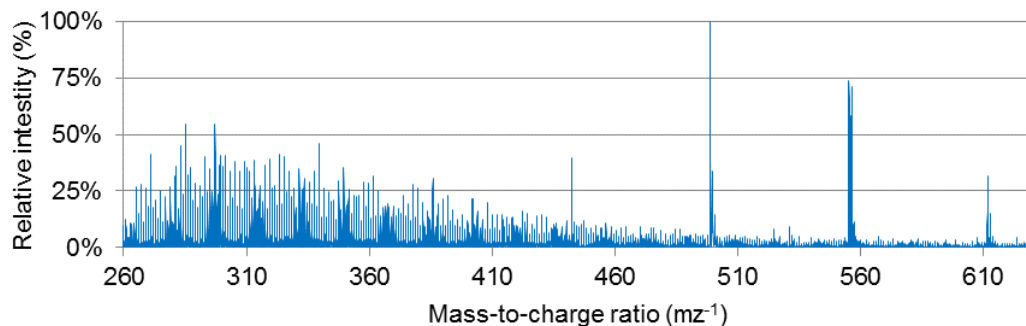


Fig. 4(c). Molecular weight distribution for tar captured at 800 mm bed height during updraft gasification

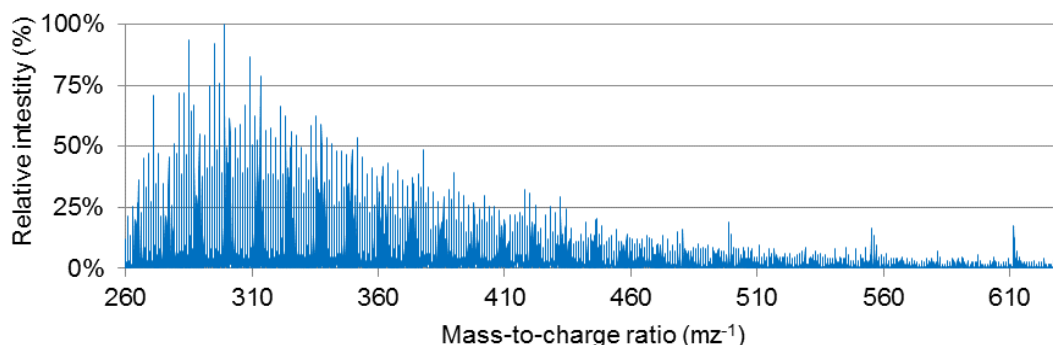


Fig. 4(d). Molecular weight distribution for tar captured during updraft gasification

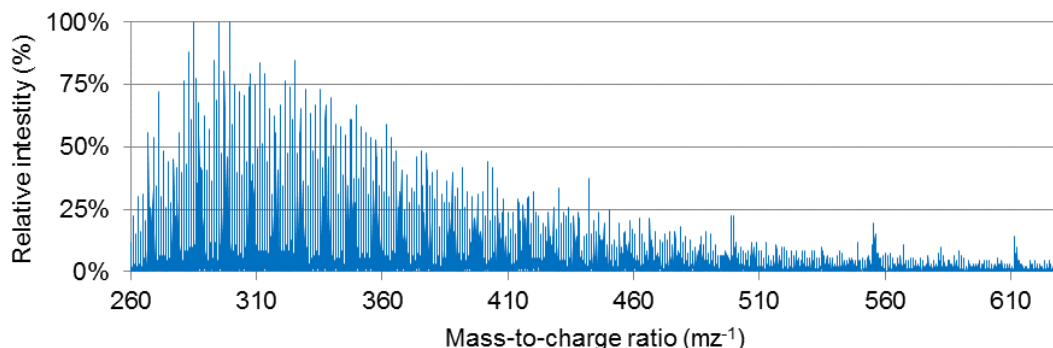


Fig. 4(e). Molecular weight distribution for tar captured during updraft gasification (steam addition)

3.4 Thermal characterization of tar

Figure 5 shows conversion behaviour of tar under pyrolysis, gasification and combustion of tar as derived by using Equation 8. This figure shows that devolatilization for volatile matters (VM) followed a similar trend regardless of presence or absence of oxidizing agent. Significant variation in the trends between pyrolysis versus non-isothermal combustion and non-isothermal gasification was observed from 700 K onwards. Thereafter, combustion or gasification of fixed carbon occurred. Fixed carbon (FC) constitutes of about 22.17% of tar. Obviously, oxidation of fixed carbon in tar was spontaneous in comparison to gasification under both isothermal and non-isothermal conditions. Nevertheless,

isothermal combustion and isothermal gasification of fixed carbon for tar conducted at 1,273 K proceeded faster than non-isothermal processes.

Figure 5 suggests that fixed carbon in tar could not be combusted or gasified below 700 K although devolatilization and hence possible oxidation of the released volatiles was achieved at lower temperature ranges. This implies that consideration such as low tar content or higher operating temperatures should be taken into account during combustion of tar containing gas fuels [1,10].

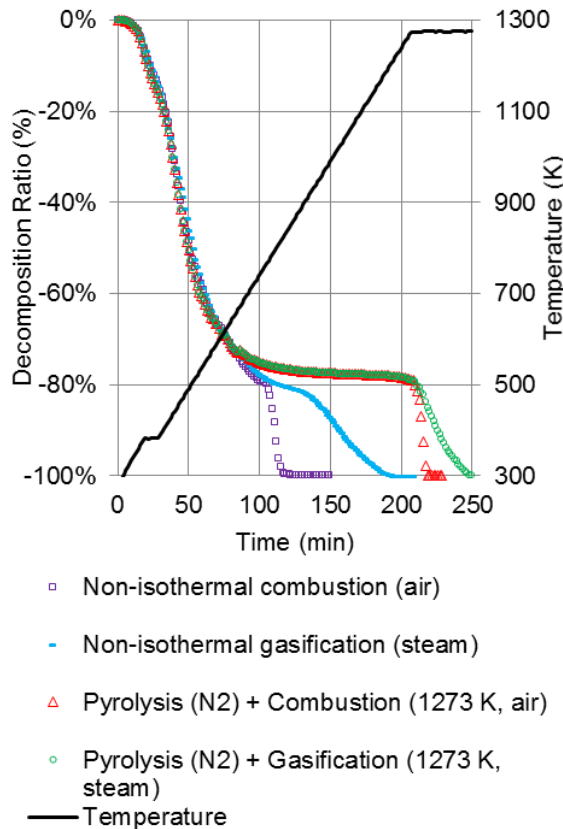


Fig. 5. Pyrolysis, combustion and gasification behavior of tar

Devolatilization reactivity of volatile matter (VM) in tar during non-isothermal pyrolysis, combustion and gasification process are presented in Figure 6. These trends in reactivity were derived by using Equation 9. Corresponding to similar trend in devolatilization processes, this figure shows that reactivity during non-isothermal pyrolysis, combustion and gasification were about $2.29\% \text{min}^{-1}$.

Degradation reactivity of fixed carbon in tar during non-isothermal and isothermal, combustion and gasification are also presented in Figure 6. Degradation reactivity of fixed carbon during non-isothermal combustion and non-isothermal gasification was found to be $3.45\% \text{min}^{-1}$ and $0.54\% \text{min}^{-1}$, respectively. Due to higher temperature used during isothermal combustion and isothermal gasification processes i.e. 1,273 K, degradation reactivity of fixed carbon was enhanced to about $3.73\% \text{min}^{-1}$ and $0.95\% \text{min}^{-1}$, respectively.

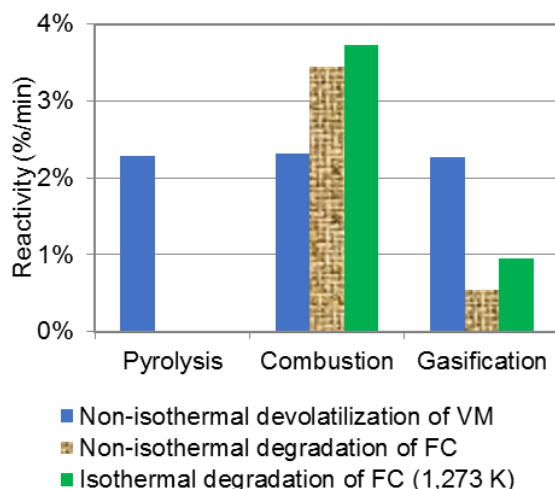


Fig. 6. Tar reactivity during pyrolysis, combustion and gasification

4. CONCLUSION

During updraft gasification, tar content in producer gas was high at the lower-middle part of the reactor which represents a pyrolytic zone. Slight reduction of tar content at upper part of the reactor can be associated with tar deposition onto the packed fresh biomass. Tar is composed of C, H, N and O by 70.62%, 10.62%, 0.57% and 18.20%, correspondingly and its HHV is around 35.27 MJkg^{-1} . ToF-MS analysis for ion mass-to-charge ratio indicates high intensities from 280 m/z to around 380 m/z . In comparison with tar taken from the tar for the gasifier, tar samples captured along the bed heights have relatively low peaks at 500 m/z , 555 m/z and 615 m/z . Concentration of tar compounds with low molecular weight was slightly reduced for tar captured from gasification by using air-steam mixture.

Devolatilization of tar up to 700 K follows a similar trend regardless of presence or absence of oxidizing agent. Combustion or gasification of fixed carbon content in tar occurs at temperature ranges above 700 K. Average devolatilization reactivity for volatile matter in tar during non-isothermal pyrolysis, combustion and gasification was about $2.29\% \text{ min}^{-1}$. Average degradation reactivity of fixed carbon during non-isothermal combustion and non-isothermal gasification was found to be $3.45\% \text{ min}^{-1}$ and $0.54\% \text{ min}^{-1}$, respectively. Due to higher temperature used during isothermal combustion and isothermal gasification processes, degradation reactivity of fixed carbon was enhanced.

Results presented in this paper shows low reactivity of tar at low temperature regime and therefore exhibit low effectiveness of thermal cracking. Further research areas in line with this study include; identification of compounds contained in tar, numerical simulation of tar degradation behavior at high temperature in inert and oxidizing environments as well as reduction of formation through catalysis.

ACKNOWLEDGEMENTS

The author acknowledges support by the University of Dar es Salaam on provision of suitable environment for this research.

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