

MUNICIPAL SOLID WASTE CONVERSION TO ENERGY AND DERIVED CHEMICALS USING PYROLYSIS

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This research work on municipal solid waste conversion to energy and derived chemicals was investigated using pyrolysis. The variables such as time and temperature were used to determine the highest bio-oil yields. Slow pyrolysis was adopted in a drop type chemical vapour depositor (CVD) pyrolyser given the highest bio-oil yields of 32.50 %wt. at 500 °C for 30 min. The bio-oil properties (CHNS-O) at various temperatures were evaluated. Carbon, hydrogen, Nitrogen and calorific values were observed to increase as the temperatures increased, having highest values at 500 °C with a sudden decline at 550 °C. While oxygen, water content, densities and pH values decrease as the temperature increased, with lowest values obtained at 500 °C and sharp increase at 550 °C. Hence, the degrees of de-oxygenation were observed to increase as the temperature increases with 20.25 %wt. at 500 °C and decrease at 550 °C. The results of FTIR analysis of the bio-oils produced at 500 °C indicate functional groups such as alkanes, alkenes, amines, terminal alkynes and aromatic ether with their areas.

Keywords: Pyrolysis, Bio-oil, Derived chemical, Energy

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INTRODUCTION

Back in ages, the amounts of waste generated by humans were insignificant due to low population density and low societal level of natural resource exploitation. Common wastes produced at these times were mainly ashes and human biodegradable waste, and these were sent back to the ground locally to decompose with minimum environmental impact, Bogner *et al.*, (2007) and Alison *et al.*, (2001). However, with the onset of industrialization and the sustained urban growth of large population centres, the build-up of waste in the cities caused a rapid deterioration in the levels of sanitation and general quality of urban life, Bogner *et al.*, (2007) and Alison *et al.*, (2001). These enormous increase in the quantum and diversity of waste material and their potential harmful effects on general environment and public health calls for a pressing need to better waste management methods in which the treatment and handling can lead to safe disposal, cleaner and healthier environment with sustainable use of the earth's resources, Salman, (2009). Also couple with the demand for fuel and various chemicals increasing day by day, the country has been spending lot on refining petroleum and related products putting pressure on the conventional sources of energy which have been depleting at an alarming rate and hence the focus on alternative renewable source of energy. As a result of these twin reasons, biomass as a renewable energy source has continued to attract more attention and a lot of research in this area is in progress using different solid biomass as the feed material, Islam *et al.*, (2010). Needless to say, municipal solid waste (MSW) is a common waste management problem with biomass obtained from any city area of all over the world as well as in Nigeria. Although for many years, opposition to the use of MSW as an energy resource has been nearly universal among activists and regulators. This opposition has been largely based on bad experiences with traditional garbage incineration facilities, which are associated with high levels of toxic emissions, as well as the perception that using MSW for energy will compete with recycling efforts, Brian and Justin, (2009). But for growing climate, energy, and environmental concerns, coupled with technological developments and regulatory changes have ignited a new interest in MSW as an energy source with the potential to provide renewable energy while reducing greenhouse gas emissions and the need for landfill space, Brian and Justin, (2009). Thus, pyrolysis may be considered as a promising option for resource recovery from MSW combined with ARM catalyst, which is abundantly available to produce quality bio-oils. It is therefore, a great potential in converting solid wastes which are very difficult and costly to manage into fuels and chemicals, reducing waste management cost. Due to the low contents of sulphur and nitrogen utilization of the derived energy, it does not add sulphur dioxide, nitrogen oxides and no net carbondioxide, which are greenhouse gases to the atmospheric environment, in contrast to fossil fuels, Ani and Islam, (1997). These also addressed an economical and ecological solution of transforming abundant naturally available materials into something useful to humanity. Considering the above advantages, this research work was undertaken to produce renewable energy and derivable chemicals from MSW using pyrolysis. According to daily assessment report of Niger State Environmental Protection Agency (NISEPA) 300 to 400 tons of MSW are generated daily on an average from Minna metropolitan area, this is a great potential for renewable energy conversion and derived chemicals. Although, these waste are mostly mixtures of substances whose constituents widely vary on location. Thus sorting is required to classify them into groups and their composition such as organic food wastes, papers, broken glasses and ceramics, metals, plastics, rubber and leather, textiles, wood, yard wastes,

e.t.c. The organic solid wastes that are lignocellulosic biomass were the target raw materials in this study.

RESEARCH METHOD

2.1. Feedstock preparation

The MSW used in this study was obtained from Kampalla dump site of Niger state environmental protection agency (NISEPA). The site covers about 20 hectares of lowland area of Maikunkele along Mimma – Zungeru road; headquarter of Bosso local government area of Niger state on latitude 54° W and longitude 108° S. The heterogeneous mixture of the wastes were sorted and separated out. The residues which were lignocellulosic material (papers, wood, yard trim and food wastes) were taken as the feedstock as shown in figure 2. Prior to pyrolysis, some of the wastes were shredded while others passed through a high speed rotary cutting mill and then sieved to obtained the required particle size of 1.75 mm, then dried to 10 wt. % moisture in an electric oven for 24 hrs at 105 °C. The proximate analysis of MSW was performed to measure the moisture content, ash content, fixed carbon contents and volatile matter using the TGA method. The ultimate analysis were also carry out using Liebig's, Kjeldahl's and TGA method of elemental analysis to determine properties of MSW and bio-oils which mainly consist of CHNS, while oxygen contents was determine using calculation difference as shown in Eq. (1).

$$O = 100 - (C\%wt. - H\%wt. - N\%wt. - S\%wt.) \quad \dots (1)$$

The high heating value (HHV) of MSW and bio-oils was calculated from the elemental and ash analysis based on Channiwala and Parikh's formula, Lim *et al.*, (2014) as shown in Eq. (2) below.

$$HHV = 0.3491C + 1.1783H - 1054O - 0.0151N + 0.1005S - 0.0211A \quad \dots (2)$$

Thus, from the elemental compositions of MSW O/C and H/C ratios are to be calculated. The FTIR analysis was performed on bio-oil yields at minimum operating temperature.

2.2. Pyrolysis experiment

2.2.1. Apparatus

Figure 1 below shows the schematic diagram of a batch type vacuum pump reactor called carbon vapour depositor (CVD) used to carry out the non-catalytic and catalytic slow pyrolysis of MSW for this study. The experimental setup consists of a cylindrical reactor made up of horizontal glass (borax) tube furnace with length-1010mm, internal diameter-60mm and outer diameter-65mm. The pyrolyzer was installed in an electric heater and was insulated to enable the heating capacity of the reactor reached 1200 °C. The reactor was equipped with a biomass holder (cubit) and connected to both vacuum and nitrogen source. To record the pyrolysis temperature, a K-type thermocouple was inserted inside the pyrolyzer. The pyrolyzer was connected to ice-trap immersed in ice-water bath to condense the pyrolysis vapours at 0–5 °C. The outlet of the ice-trap was connected with the gas sampling bag to collect the non-condensable gases.

2.2.2. Procedure

Slow pyrolysis was adopted in all the experiments, 20g of MSW was loaded into the cubit and fixed into the reactor. A vacuum pump was set to obtained high vacuum inside the pyrolyser between 0.003 to 0.001 mmHg pressures (< atmospheric pressure). The experiments were carried out to determine the effect of the pyrolysis temperature on products yield. The temperature of the pyrolyser was set between 400-550 °C with intervals of 50 °C for the range of periods 10–40 min with constant heating rate of 20 °C/min and N₂ flow rate of 50 ml/min. the durations of the experiments were held at that temperature until there was no significant gas observed to be released and the CVD reactor automatically terminated at that fixed period. Same procedures were repeated for 20, 30, and 40 min. The products from the pyrolysis process are char, bio-oil and gases. The fluid gas comes out through the vacuum line of the pyrolyser and the solid char remain in the cubit as residue. The fluid from the pyrolyzer was then cool through the condenser. The condensate (bio-oil) was collected and stored in a sample bottle for further analysis, while the uncondenseable gas was given off as syngas (flue gas) and collected in a sample bag. Mass balance calculations were used to estimate the yield; char (wt. %) + bio-oil (wt. %) + gas (wt. %) = 100. The yields of char and bio-oil were determined based on change in weight of the reactor and the condenser respectively, before and after the experiment. The mass of liquid and solid are given within the experimental error of ± 2 .

2.2.3. Analysis of pyrolysis products

At the end of each pyrolysis experiments; the bio-oil collected was found to be in a single phase dark brown colour with a distinct odour similar to smoke from a wood fire and was quite pungent. The pyrolysis oils obtained at pyrolysis temperature of 500 °C for 30 min was characterized by using the following methods. The elemental analysis of the biomass and bio-oils were determined through Liebig's, Kjeldahl's and TGA method of estimating CHNS-O and their calorific values were calculated by difference. The water content of bio-oils was measured using proximate analysis. The pH value was obtained by a pH probe with a digital meter. The densities were calculated by mass/volume ratio in the lab. According to Naqvi *et al.*, (2014), a formula to estimate the degree of de-oxygenation was proposed by Zabeti as shown Eq. (3) below:

$$\text{Degree of deoxygenation} = \left(1 - \left(\frac{[O]_{\text{bio-oil}}}{[O]_{\text{biomass}}} \right) \right) \times 100 \quad (3)$$

Where [O] is oxygen content (mass)

Degree of de-oxygenation is used to judge the amount of oxygen that has been rejected from the biomass and retained in the bio-oil.

2.3. Figures and Tables

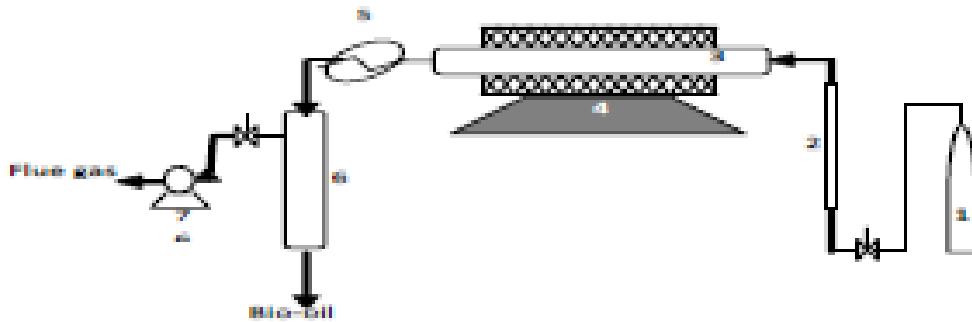


Figure 1: Layout of the pyrolysis process

1-N₂ gas cylinder, 2-Flow meter, 3-Glass pyrolyser, 4-Reactor Controls, 5-Coolant, 6-Condenser, 7-Suction Pump.

FINDINGS AND DISCUSSION OF RESULTS

3.1. Analysis on feedstock

The heterogenous nature of the different materials involved in MSW made it nearest to impossible, to use it as raw material for this study. This was due to presence of metals, broken glasses and ceramics. After the raw materials were manually sorted and separated into their various compositions; out of 1 kg of the raw sample collected 71.7g of it contains lignocellulosic materials as demonstrated in figure 2 below.

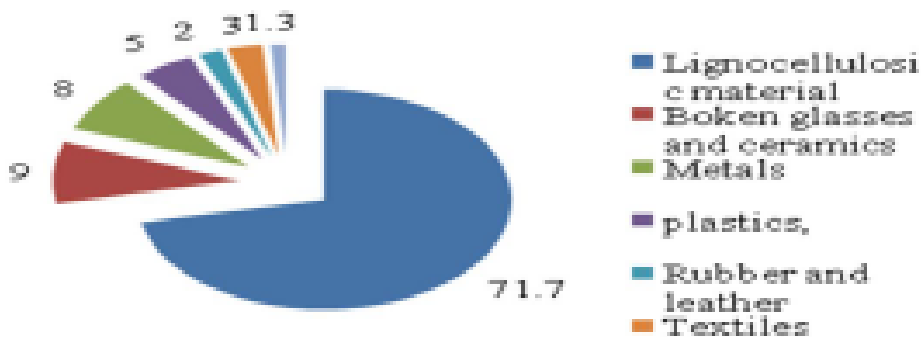


Figure 2: Compositions in 1 kg sample of MSW

The major components of the lignocellulosic material in 71.7g of MSW are shown in figure 3.

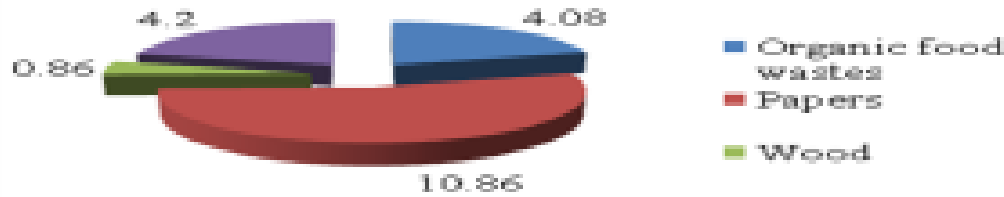


Figure 3: Component of lignocellulosic material in 71.7g of sample MSW

3.1.1. Thermal gravimetric analysis:

TGA4000 was used to determine the mass change of a sample with increasing temperature and time. The main aim of the TGA analysis on MSW is to find (1) the degradation profile, (2) calculate the proximate analysis by monitoring the weight loss. The plot between temperature and percent weight loss for MSW at heating rate of 20 °C/min and the result is as shown in Figure 4 below.

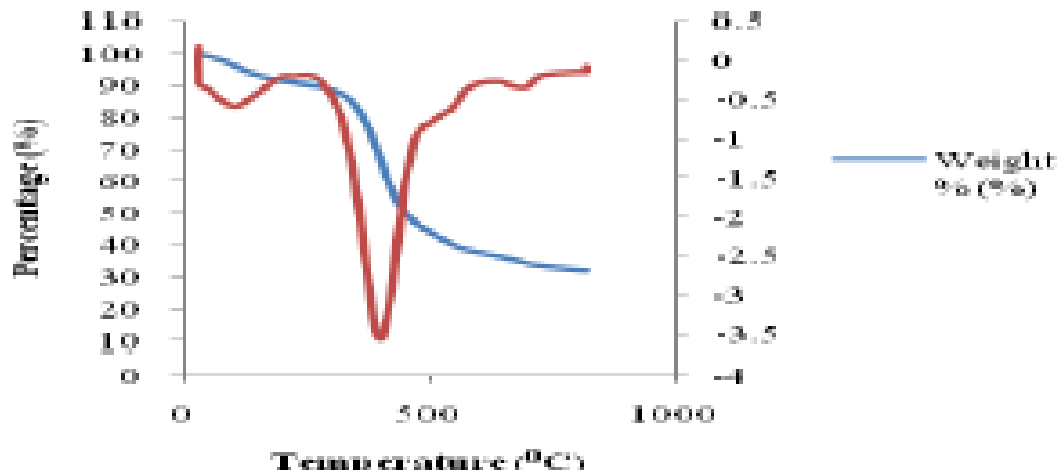


Figure 4: Thermo-Gravimetric Analysis of MSW

The graphical illustration of the TGA–DTA curves of MSW is as shown in figure 4 above. The TGA result shows three distinct weight losses. The first weight loss occurred between 28.98 and 200 °C corresponding to the endothermic peak at 100.55 °C in the DTA curve. These observations related to the vaporization of light volatile matters and water. The second weight losses occurred between 264.71 and 576.64°C corresponding to the endothermic peak at 390.80°C in the DTA curve. This observation can be attributed to the defragmentation of the polysaccharides (hemicelluloses and celluloses). The third weight loss occurred at the temperature higher than 500°C and this correspond to the endothermic peak at 688.05 °C in the DTA curve. This may be attributed to the burned and evaporated (lignin) carbon char.

The graphical interpretation agrees with Yathavan, (2013) and Surwanmaneechot *et al.*, (2015).

According to Yathavan (2013), lignocellulosic biomass usually comprises of three major components namely cellulose, hemicelluloses and lignin. Hemicelluloses which is characterized by amorphous and irregular shape degrades at low temperature 250 – 400°C; cellulose which has crystalline structure degrades between 310 – 430 °C; and lignin which is a complex aromatic heteropolymer, degrades in a wide range of temperature 300 – 500 °C. Table 1 below shows 8.087 %wt. moisture content, 54.141%wt. volatile matter, 36.315 %wt. fixed carbon and 1.612 %wt. ash content of the biomass estimated from the weight loss curve. These results were also in line with those obtained by Surwanmaneechot *et al.*, (2015) and Gopakumar *et al.*, (2011). The ultimate analyses were done separately done using classical methods and the results are equally presented in Table 1 below.

Table 1: Proximate analysis from TGA weight loss thermogram and ultimate analysis of MSW

Properties	MSW
<i>Proximate analysis</i>	
Moisture (%)	8.087
Ash (%)	1.612
Volatile component (%)	54.141
Combustible matter (%)	36.160
<i>Ultimate Analysis</i>	
C (%)	46.81
H (%)	13.30
O (%)	37.92
S (%)	0.05
N (%)	0.58
Ash content (%)	1.34
Caloric value (MJ/kg)	27.98
O/C (molar ratio)	0.61
H/C (molar ratio)	3.41
Empirical formula	$\text{CH}_{3.41}\text{O}_{0.61}\text{N}_{0.0108}$

3.2. Analysis of pyrolysis products

3.2.1. Effect of time on oil yields

To determine the effect of time on bio-oil yields, the series of experimental pyrolysis performed at constant times of 10, 20, 30 and 40 min between the ranges of 400–550 °C were plotted. The results are as shown in figure 5 which agrees with those obtained by Islam *et al.*, (2010).

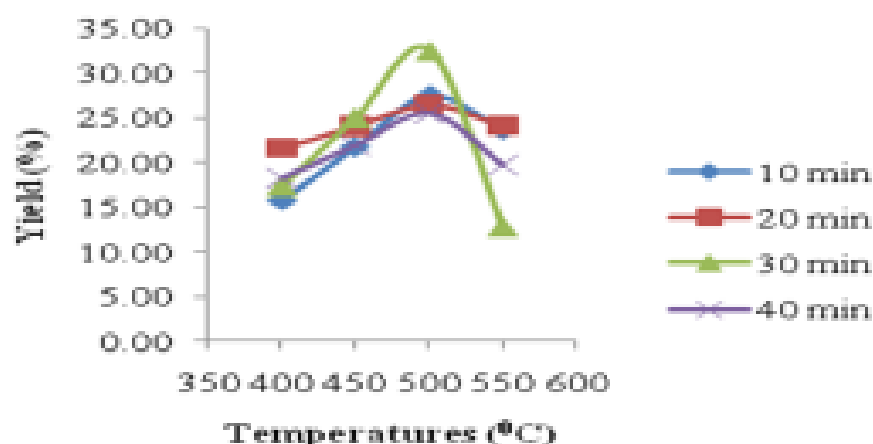


Figure 5: Effect of constant pyrolysis time on oil yields at various temperatures

The best time of 30 min pyrolysis produced the highest oil yield of 32.50 %wt. at temperature of 500 °C as shown in figure 5 above, and this was used to study the impact of temperature on product yield at 500 °C.

3.2.2. Impact of temperature on product yields

It is a well established fact that the main parameter among the operating factors is the temperature; hence the impact of pyrolysis temperature on products yields were investigated and illustrated in figure 6 below. To do this, the pyrolysis experiments conducted for 30 min at the various temperature range of 400-550 °C with intervals of 50 °C was isolated and under study. On a general note, higher gaseous yield were obtained at all temperatures as shown figure 6. These were due to continuous decomposition of bio-char as it remained for too long in the furnace. An increased in the bio-oil yields were observed from 400-500 °C and a sudden decreased at 550 °C. Thus the highest bio-oil yield of 32.50 %wt. was recorded at the pyrolysis temperature of 500 °C and termed as the best temperature. At lower temperatures (<500 °C), the oil yield was lesser and char yield was more. On the other hand, at higher temperature (>500°C) the char and the oil yield were less; however the gas yield was observed to be higher. The reason for less oil yield at lower temperatures (<500 °C) may be due to incomplete pyrolysis which result from insufficient energy required, thereby yielding less oil and more char. On the contrary, the high gaseous yields at higher temperature (>500 °C) may be attributed to the secondary cracking of the pyrolyzed vapour and decomposition of the bio-char, which lower oil and char yield. Similar pattern was observed by other researchers, Salman, (2014), Naqvi *et al.*, (2014) and Nikos, (2012).

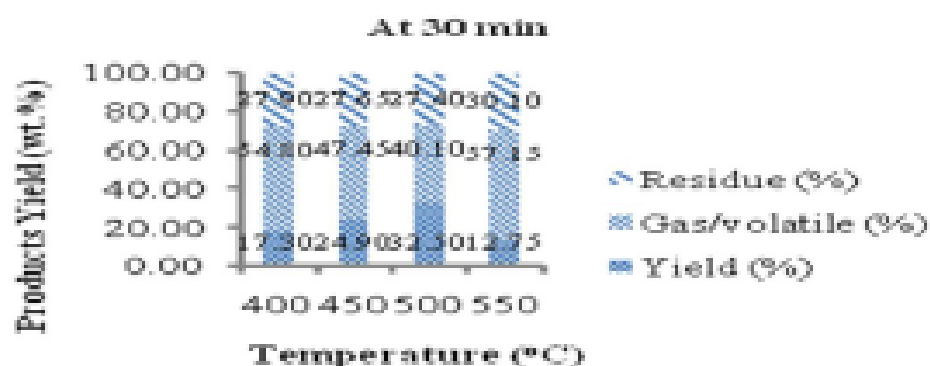


Figure 6: Impact of temperatures on products yield

According to Jahurul *et al.*, (2012), Brown (2009) and Naqvi *et al.*, (2014), the gases formed during pyrolysis experiments comprise of H_2 , CH_4 , CO and CO_2 . These gases were used to calculate the compositions (vol. %) as the temperature increases and the results are as shown in Table 3 below. The total gases yield at 400 °C is 54.80 vol. %, which decreased to 47.45% vol. % at 450 °C and the least volume of 40.10 vol. % was recorded at 500 °C, while further increase in temperature to 550 °C lead to an increase of 57.94 vol. %. At 500 °C where the least volume was obtained it shows that some of the gases were converted to straight chain hydrocarbon thereby given rise to more energy density of the bio-oil.

Table 3: Composition of gases (Vol. %) for non-catalyst and catalyst

Gases	400	450	500	550
H_2	1.22	1.05	0.89	1.27
CH_4	9.74	8.44	7.13	10.16
CO	17.05	14.76	12.48	17.78
CO_2	26.79	23.20	19.60	27.94
Total	54.80	47.45	40.10	57.15

As the temperatures increases the ability to de-oxygenate undesirable constituents of bio-oil to form CO_2 and H_2O increases. While as the temperature of the system cools below 300 °C there is reduction of CO_2 and H_2O leading formation of straight chain hydrocarbon. This is demonstrated in table 3 above, where the volume of CO and CO_2 were least recorded at 500 °C compared to others, this clearly shows that de-oxygenation of harmful oxygenates from the vaporized bio-oil has been achieved by converting them to CO and CO_2 at a temperature below 500 °C. The volume of CO and CO_2 decreasing at temperature below 500 °C, which indicates inability of the temperatures to de-oxygenate harmful oxygenates and increased above 500 °C.

3.3. Analysis of the oil yield

3.3.1. Thermo-physical properties of bio-oil

The key bio-oil properties obtained varies with pyrolysis temperatures as depicted in Figure 7(a-c). It appeared dark brownish free from visible sediments; highly acidic with high water content, low heating value and very low sulphur content. The ash content in the oil is very negligible which agrees with other research conducted by Naqvi *et al.*, (2014). The sulphur contents of the bio-oil produced was relatively lower than petroleum fuels. The low sulphur contents are positive property for application as fuel. Compared with all pyrolysis temperatures, the bio-oil produced at the 500 °C had higher Carbon (21.70%wt.), Hydrogen (11.7%wt.), Nitrogen (1.86%wt.) and lower energy density (18.15%wt) than others as shown in figure 7(a). The higher nitrogen contents are greater than petroleum irrespective of pyrolysis temperature and are not favourable due to causing NO_x emission in fuel use. These results imply that the bio-oils should be de-nitrogenated to improve their applicability as fuels. The lower heating value of the oil is due to high water content and presence of carbonyl compounds. These results agreed with an earlier study by (Islam *et al.*, 2014), (Naqi, *et al.*, 2014) and (Kim *et al.*, 2014).

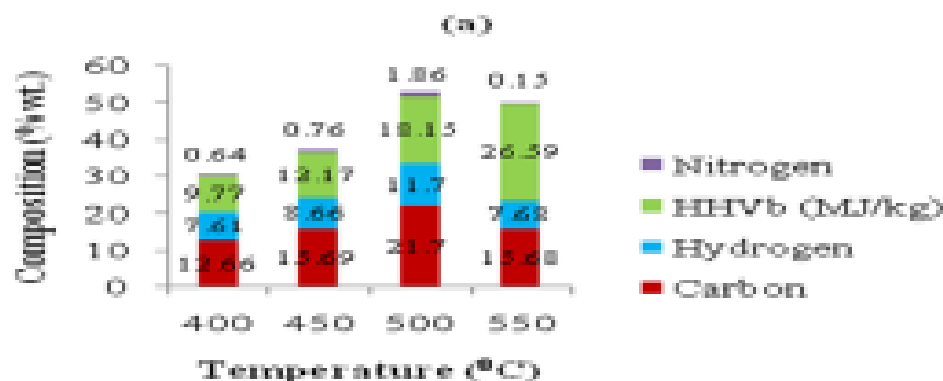


Figure 7(a): Properties of bio-oil yields

The density and oxygen content decreases as the pyrolysis temperatures increased from 400–500 °C, and at 500 °C the bio-oil has minimum values as demonstrated in figure 7(b). Compared with petroleum standard the minimum oxygen content is higher in the bio-oil which results in acidic, unstable and corrosiveness with a relatively low energy density. The varying quantities of water formation which forms a stable single-phase mixture decreasing as the temperature also increases, ranging from 44.86, 41.71, 34.50 to 41.22 %wt. water. The high water content of bio-oil is due to formation of water in pyrolysis process. These all agrees with the early study by Naqvi *et al.*, (2014).

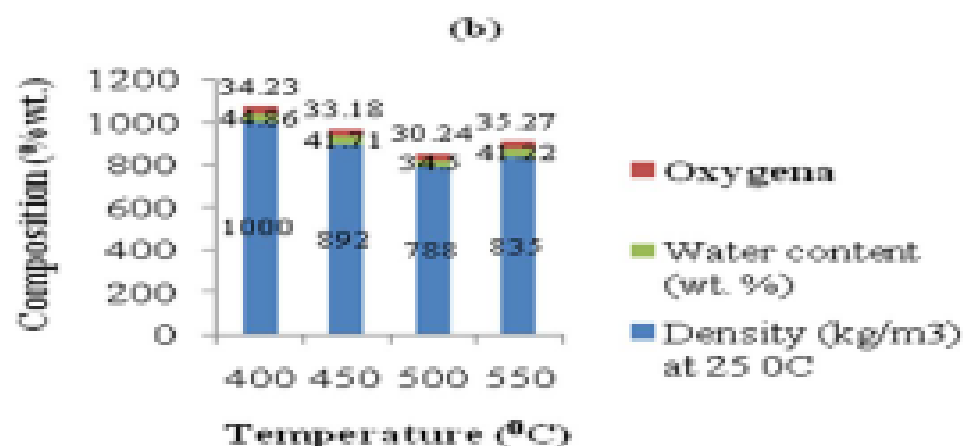


Figure 7(b): Properties of bio-oil yield

In all experiments, the bio-oils consisted of higher acidity (pH) values and decreases as the temperatures increases, while at 500 °C it recorded the lowest acidity (pH) value (3.42) as shown in figure 7(c). This value is lower than 4 indicating it's corrosive nature and resulted from high oxygen content from the biomass, this also agreed with early study by Naqvi *et al.*, (2014).

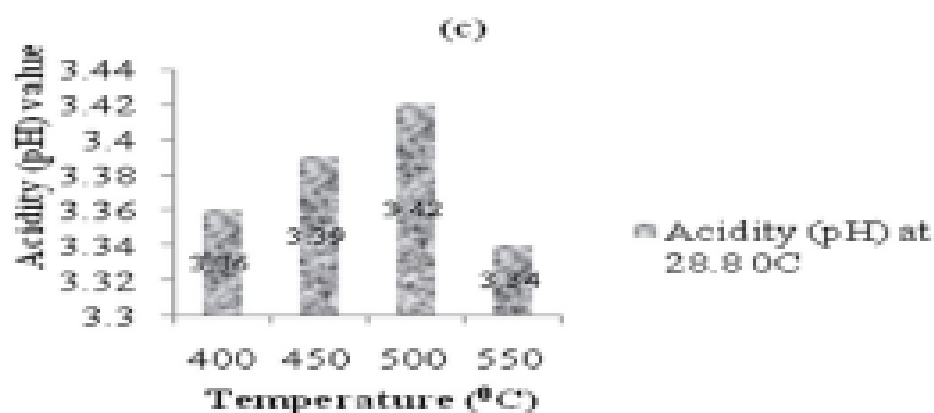


Figure 7(c): Properties of bio-oil yield

3.3.2. Impact of temperature on the degree of de-oxygenation of bio-oil

The degree of de-oxygenation of bio-oil at various pyrolysis temperatures are shown in figure 8. This is aimed at reducing the oxygen content of biomass through various pyrolysis temperatures to get high quality bio-oil. It focuses on how much oxygen has been removed from biomass and retained in the bio-oil.

At temperatures below 500 °C, the oxygen removal was less compared to at 500 °C. Higher temperatures helped to remove more oxygen from the original biomass. The results showed

that more oxygen is been removed ~20.25%wt. at 500 °C as compared to ~9.73%wt. 400 °C, ~12.50%wt. 450 °C, and ~6.99%wt. at 550 °C pyrolysis temperature. It was also observed that the oxygen in the biomass was removed mostly as H₂O and as CO₂/CO, the result also agreed with the study carried out by Naqvi *et al.*, (2014).

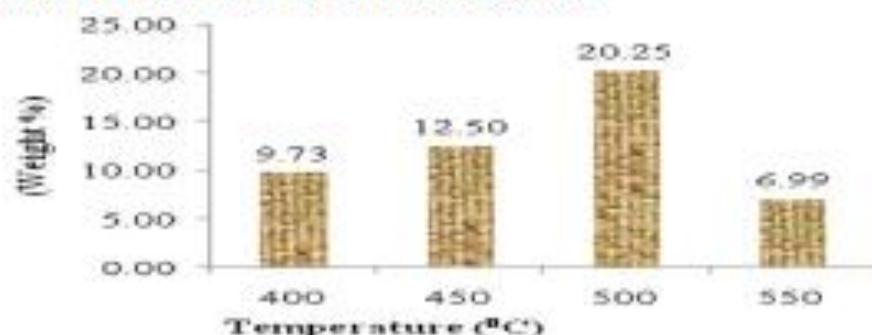


Figure 8: Degree of de-oxygenation of catalytic and non-catalytic bio-oil

3.3.3 Impact of Temperature on the composition of bio-oil

The bio-oil is a mixture of different compounds and reflects the decomposition of the lignocellulosic contents of MSW. To understand how temperature of 500 °C influences the pyrolysis of MSW; the functional groups present in the bio-oils were analyzed using FTIR. These were classified into different groups (such as alkanes (C-H), alkenes (C=C), amines (N-H), alkanol (O-H) e.t.c.) and their areas. The total peak areas of these groups for bio-oils are as shown in figure 9.

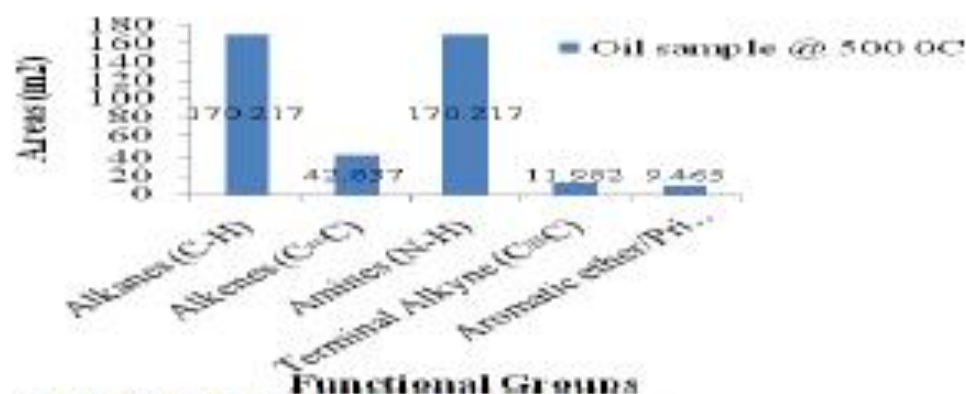


Figure 9: Functional groups of bio-oils and their areas at 500 °C

From figure 9, the functional groups and areas of the bio-oil revealed the different components of MSW with nitrogen-containing compounds and complex carbohydrates. This may suggest that food waste component of MSW contains high source of amino acid (high crude protein). At 500 °C where the degree of de-oxygenation is high, carboxylic oxygenates

are formed through decarboxylation of organic acids (such as acetic, formic, fatty and amino acids) from cellulose, hemicelluloses, esters and nitrogen-containing compounds. Decarbonylation of aldehyde, phenol, ketones and carbohydrates compounds to form aliphatic and aromatic hydrocarbons. Amines are also formed through depolymerization of amino acids (protein and amides), catalytic hydrogenation of nitriles groups ($R-C\equiv N$) and nitro-alkanes ($CH_2-CH-NH_2$). The areas of the functional groups are 42.837m² alkenes, 170.217m² amines, 11.982m² terminal alkynes and 9.465m² aromatic.

CONCLUSION

Much attention has been given to biomass pyrolysis lately, because of clean energy production. The bio-oil yields for the best operating temperature (500 °C) was 32.50%wt. whereas the highest degree of de-oxygenation was 20.25%wt., respectively. Meanwhile, the carbonyl and acidic components that cause instability and acidity of the bio-oil were reduced to the form of H₂O and CO₂ by oxygen removal. The results of FTIR analysis of bio-oils at 500 °C indicate functional groups (such as alkanes, alkenes, amines, terminal alkynes and aromatic ether) with their areas. The presence of amines may likely be from amino acid and amides in the food wastes which make it unfavourable due to emission of NO_x for the oil use. Thus, food wastes can be isolated from the feedstock or better still further study may revealed an efficient way of nitrogen removal in the bio-oil in consideration to a specific fuel use.

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