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# Characterization of Char from Waste Tyre Pyrolysis

Yaru, S. S[1], Satope, P. O[1], Akinola, A. O[1*]

[1]Department of Mechanical Engineering, The Federal University of Technology, Akure, Nigeria

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***Corresponding author:** Akinola, A. O

## Abstract

The characterization of char from waste tyre pyrolysis is evaluated in this work. Disused tyres retrieved from vehicle users and dump hills in Akure and its environment in South West Nigeria were pyrolysed in a refurbished 17.4 litre capacity fixed-bed batch thermochemical reactor. The waste tyres were cut into sample sizes of 20 mm × 30 mm manually and then weighed into various portions of 1kg each. 1kg mass of the washed waste tyres was pyrolysed in each batch of pyrolysis experiment to obtain char at different pyrolysis temperatures of 250$^o$C, 350$^o$C, 450$^o$C, and 600$^o$C respectively. Proximate and ultimate analyses of the char resulting from the pyrolysis were carried out. The pyrolysis process produced char with an average of 83.30% carbon composition 5.45% hydrogen, 0.52% nitrogen, 1.17% sulphur and 1.90% oxygen composition. The analysis of percentage composition of the raw scrap tyre samples gave 79.86% for carbon, 7.35% hydrogen, 0.39% nitrogen, 1.57% for sulphur and 6.50% for oxygen. The ultimate analysis of the char resulting from pyrolysis gave an average value of 38.83 kJ/kg for the heating value and 470.00 kg/m$^3$ for specific gravity. The average fixed carbon content was 33.38% while the volatile matters gave an average of 58.40%. The ultimate analysis of the raw tyre samples gave values of 36.10 kJ/kg for the heating value and 343.00 kg/m$^3$ for specific gravity.
**Keywords:** Pyrolysis, waste tyres, proximate, ultimate, heating value, thermochemical reactor.



## INTRODUCTION

Man has started to grabble with the crises resulting from the usage of the fossil fuels and from the wastes generated from human activities. These fuels generate a lot of carbon dioxide, a greenhouse gas (GHG) during combustion which forms a shield in the atmosphere thus causing global warming with the consequent climate change. At the same time the fossil fuels are depleting at an unprecedented rate with eventual projection that they would be exhausted in some decades to come [1]. As a result the world is turning towards renewable energy as an alternative energy sources. These energy sources are inexhaustible because as they are being used so they are produced and even at faster or higher rate in some cases. Examples of the renewable energy sources are solar and hydropower among others, and in recent times solid wastes produced from the activities of man [2]. These wastes are either biodegradable or non-biodegradable. The non-biodegradable wastes like scrap tyres do not degrade easily in the soil because of their complex polymer mixture of very different materials, which include several rubbers, carbon black, steel cord and other organic and inorganic components [3]; pollutes the atmosphere, soil and water both surface and underground; become breeding places for diseases and their vectors like mosquitoes, cockroaches, scorpions and even rodents; yet their production is on the increase as a result of industrialization and motorization of the world. Wojtowicz and Serio [4], reported that the durable property of tyres makes them biologically non-degradable and at the same time makes their disposal and reprocessing difficult. They asserted further that these tyres also have potential fire hazards as they have pockets of air stored in their piles [5] noted that the production of waste tyres throughout the world is estimated to be 1billion tons tyres per year while the global annual tyre production is about 1200 million [6].

The disposal of solid tyre wastes from human activity is a growing environmental problem for the modern society, especially in developing countries. The only common way of disposal of these scrap tyres is land filling and this requires a large space because the volume of tyres cannot be compacted [3]. The United States Environmental Protection Agency reported in 1991 that about 242 million automotive, truck, and off-road tyres are discarded in the United States each year. This is approximately one waste tyre per person per year. In the report, about 33.5 million tyres are rethreaded and an estimated 10 million are reused each year as second-hand tyres. It is estimated that 7% of the discarded tyres are currently being recycled into new products and 11 percent are converted to energy. Nearly 78% are being land-filled, stockpiled, or illegally dumped, with the remainder being exported. This is largely almost the case is every developing countries resulting in a larger chunk of the scrap tyres being stockpiled, land-filled or illegally dumped.

In response to the environmental problems and health hazards caused by countless scrap tyre piles around the globe, most industrialized countries have instigated legal guidelines addressing this issue [7]. The legal guidelines which are stringent in some cases are all geared towards finding a better alternative to stockpiling such solid wastes. However, recycling of solid wastes to useful end products is a sustainable and reliable approach for solving such environmental and energy need. Different recycling processes are being used such as reclaiming, incineration, rethreading, grinding etc. but these different recycling processes have some drawbacks [8].

Nigeria is not left out in its share of the global menace of illegal solid waste. The country is littered with a host of them. Used tyres represent a major part of such wastes. Obtaining a clean and healthy environment and atmosphere is therefore a necessity [9]. It has become necessary to recycle these waste tyres through pyrolysis to recover sources thermal energy and other products for use in the industries [10], defined pyrolysis as the thermal degradation of waste in an oxygen free environment, or in an environment in which the oxygen content is too low for combustion or gasification to take place [11], defined pyrolysis as a thermal degradation of biomass under moderate temperature in the absence of oxygen. The work described its products to consist of bio-oil (condensable gas), synthetic gas (non-condensable gas), and char [12], described it as the conversion of biomass to liquid (bio-oil or bio-crude), solid (char) and gaseous fractions, by heating the biomass in the absence of air to around 500 $^{o}$C [13], opined that pyrolysis is the thermal break down of biomass in the limited amount of air to produce solid residue, condensable organic liquids and non-condensable gases.

Pyrolysis- the thermochemical conversion process (400 to 600$^{o}$C) of biomass or other organic matters into primarily liquid (oils) and solid (char products) and some gaseous (methane, carbon monoxide, carbon dioxide, and other organic compound) products in the absence of oxygen [2, 14], is a way of reprocessing waste tyres. Some advantages of pyrolysis include: reduction in greenhouse gas emissions and waste going to landfill; produces renewable electricity and a useful by-product, bio-char etc.; low risk of odours; high recovery rate of energy; and minimal risk of health consequences [9, 15].

The solid residue (tyre char), as reported by [16], contains carbon black, high boiling points tar and inorganic ash [17], grouped pyrolysis products into two fractions; the char which is the solid residue and the volatile fraction. The char consists principally of the carbon black with the properties depending on the pyrolysis condition while the volatile fraction is a complex mixture of hydrocarbons from methane to higher molecular compounds; and the non-hydrocarbons like carbon dioxide [14, 18].

A number of studies have been reported in literature related to tyre pyrolysis for its conversion into valuable compounds [19] used fixed bed and fluidized bed reactors to maximize the selective determination of single ring aromatic hydrocarbons [20], pyrolyzed car tyre and truck tyre in a fixed bed reactor and reported that tyre pyrolysis liquids were lighter than diesel but heavier than naphtha.

The heating rate and the particle size of the feedstock have been reported not affect the pyrolysis process significantly. However, when the feedstock is heated faster to a given temperature, the quantity of the char produced is low and the liquid and gaseous fractions are higher. High heating rate decreases the oil yield, but the reverse is the case when the high heating rate is associated with short residence time [21]. They further reported that the carbon black yield is almost constant but the oil fragment pyrolysis product increases with the particle size of the feedstock at high temperature range.

Akinola and Adewole [9], reported on the gaseous product obtained from tyre pyrolysis. The gaseous product derived from the pyrolysis was characterized; the fuel property of the gaseous products was investigated for flammability and the chemical constituents ascertained with the heating value of butane (domestic cooking gas). They concluded that the gaseous products contain energy that can be harnessed for use. The objective of this paper therefore is to characterise the char obtained from vehicle tyre pyrolysis for energy use.

## MATERIALS AND METHOD

### Materials

The materials used for the pyrolysis are the scrap tyres retrieved from vehicle users and dump hills in Akure and its environment in South West Nigeria, refurbished thermal reactor that consists of the furnace, the thermal reactor, condensing unit, digital electronic control unit, water reservoir, water pump, flow meter, control valve, interconnected pipes and gas collection point. The plant can withstand a pressure of 2.3 MN/m$^2$ and a temperature of 1200 $^{o}$C [2].

The scrap tyre was cut into sample sizes of 20 mm × 30 mm manually and each sample weighed 1kg each. A portion of the samples was taken for proximate and ultimate analyses to ascertain the chemical contents of the samples as received before pyrolysis. One kilogram (1kg) of the sample was introduced into the heating chamber of a batch reactor (Figure 1). The tests were conducted using the American Society for Testing and Materials Standards ASTM E1756 – 08 [22].

The heating chamber was well positioned into the furnace of the reactor and all necessary checks are made on the line. All the other components were well positioned and arranged to allow easy movement and access to the various units of the batch reactor. The condensate and gas receivers were positioned in ice baths separately to enhance condensation and gas trapping.

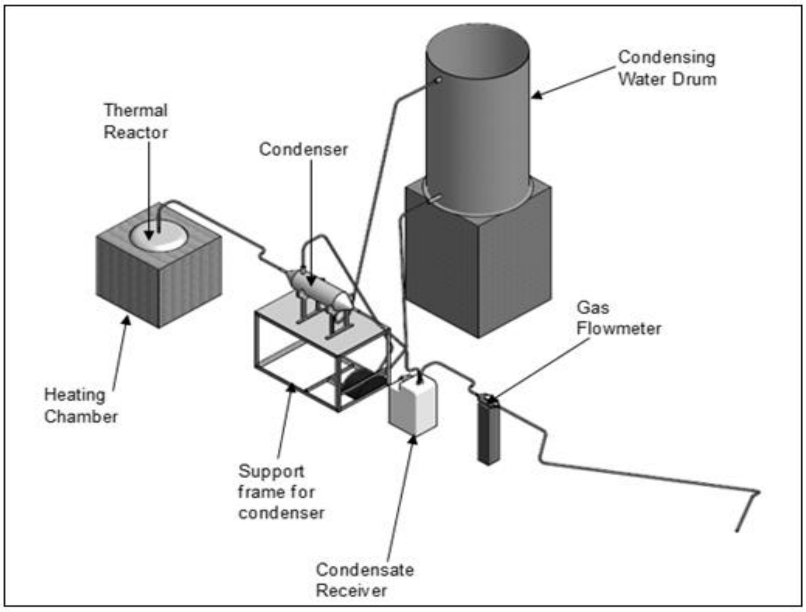


**Fig-1: Batch Reactor for Scrap Tyre Pyrolysis Experiment**
**Source: [2]**

The connections were tightly fitted, and the thermocouple measuring the temperature in the heating chamber was pre-set to 250 °C. The furnace was then electrically heated. The system was monitored after every 15 minutes and the temperature of the heating chamber and the furnace materials were recorded off the digital displays of the thermocouples. As soon as the pre-set temperature was reached, the system was allowed to stay for a retention time of 30 minutes. The system was allowed to naturally cool and the weight of the resulting solid product measured. The procedure was repeated for reactor temperatures of 350 °C, 450 °C, and 600 °C. At each pyrolysing temperature, the solid residue resulting from the process was collected and weighed.

All the solid product samples collected for each temperature was subjected to proximate analysis which included tests for volatile matter, ash content, fixed carbon content, heating value, and specific gravity. The ultimate analysis of each sample was also carried out and the heating values determined with the help of a bomb calorimeter.

The weight of the tyre samples were determined using the equation (1)

$$W_{st} = W_{Pst} - W_{pan} \quad \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

where, $W_{st}$ is the weight of the tyre sample; $W_{Pst}$ is the weight of measuring pan together with the scrap tyre sample; and $W_{pan}$ is the weight of measuring pan.

The char produced at the end of pyrolysis was recovered from the reactor, then weighed and its mass worked out using equation (2).

$$W_{char} = W_{Pchar} - W_{pan} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

Where $W_{char}$ is the weight of the char produced; and $W_{Pchar}$ is the combined weight of the char and the measuring pan.

### The char product yield

Product yield is the ratio of the weight of product to the sample in percentage. It was determined for the solid product by weighing each solid product of the pyrolysis collected at each pyrolysing temperature against the weight of each batch of sample.

The char yield $\bar{Y}_{char}$ was determined using equation (3)

$$\bar{Y}_{char} = \frac{W_{char}}{W_{st}} \times 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

### Analysis of Samples

Both ultimate and proximate analyses were done where applicable on the tyres before their pyrolysis and the pyrolytic products were also analysed after the pyrolysis of the tyre samples.

**Ultimate Analysis**

The elemental analysis was carried out to determine the percentage composition of the samples by weight. This was done in accordance with the ASTM E1757 – 01 standards [23] by using Leibig-Pragle Chamber containing magnesium percolate and sodium hydroxide. The percentage distribution of the constituents of carbon, hydrogen, oxygen and sulphur were determined. The Nitrogen content was determined using the Kjedahl method.

**Proximate Analysis**

All the samples collected (both pyrolysed samples and raw sample) were tested for volatile matter, ash content, moisture content and fixed carbon content. These were done according to ASTM D7582 – 15 standards [24].

**Percentage Ash Content**

A mass of 2g of sample was introduced into a previously weighed crucible and placed in a muffle furnace set at 900˚C for 6 hours till a white greyish matter was obtained. The weight of the residue was obtained as a percentage of ash using equation (4)

$$\%\text{Ash} = \frac{M_a - M_o}{M_s} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (4)$$

Where, $M_a$ is the mass of the crucible with ash; $M_o$ is the mass of the crucible; and $M_s$ is the mass of the sample.

**Percentage Moisture Content**

A mass of 2g of sample was introduced into a previously weighed crucible and placed in a GallenKamp drying oven up to and maintained at 105˚C. The change in weight was taken at every 6 hours till a constant mass was attained. The percentage moisture was calculated using the equation (5)

$$\%\text{ Moisture Content} = \frac{W_i - W_f}{W_i} \times 100 \ldots\ldots\ldots\ldots\ldots (5)$$

Where $W_i$ is the initial mass of the sample and $W_f$ is the final constant mass of the sample

**Percentage Volatile Matter**

Two grams (2 g) of sample was introduced into a closed crucible and heated in a GallenKamp muffle furnace set at 600˚C for 6 (six) minutes and then heated again for another 6 (six) minutes at 900˚C. The amount of volatile matter is equal to the loss in weight which is calculated using the formula in equation (6).

$$\%\text{ Volatile Matter} = \frac{W_i - W_f}{W_i} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots (6)$$

Where, $W_i$ is the initial mass of the sample; and $W_f$ is the final constant mass of the sample

**Percentage Fixed Carbon**

The amount of fixed carbon is calculated using the formula in equation (7).

$$\%\text{Fixed Carbon} = 100 - (\%\text{Moisture} + \%\text{ Ash} + \%\text{ Volatile Matter} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (7)$$

**Heating Value**

The gross energy determination of the oil and the char was done using GallenKamp Ballistic Bomb Calorimeter in accordance with AOAC (2003) official method.

## RESULTS AND DISCUSSIONS

**Analysis of Tyre Samples**

The chemical analyses of the raw tyre samples before their pyrolysis are shown in Table 1. This was necessary in order to compare with the elemental analysis resulting from the chemical decomposition that occurred during the pyrolysis process.

**Table 1: The ultimate analysis of raw scrap tyre samples**

| Element | %C | %H | %N | %S | %O |
|---|---|---|---|---|---|
| Raw Scrap Tyre | 79.86 | 7.35 | 0.39 | 1.57 | 6.50 |

The result in Table 1 revealed that tyres were made primarily of carbon (79.86%). This possibly accounts for large amount of carbon dioxides produced during burning of tyres as well as the large amount of heat associated with the burning process. These values fall in the range of values provided by [25]. The range for carbon, C is 74-86%, hydrogen, H is 6-8%, nitrogen, N is 0.3-1.0%, sulphur, S is 1.4-2.0% and oxygen, O is 1.0-15%.

The proximate analysis of the raw scrap tyre is as shown in Table 2.

**Table 2: Proximate analysis of raw scrap tyre samples**

| Sample | %moisture Content | %Volatile matter | %Ash | %Fixed carbon | Heating value (kJ/kg) | Specific gravity |
|---|---|---|---|---|---|---|
| Raw Scrap Tyres | 1.25 | 62.10 | 8.67 | 27.98 | 36.10 | 243.00 |

The ultimate analysis of the scrap tyres revealed that tyres were made up primarily of volatile matters. This as well has high amount of fixed carbon which increases the fuel properties of scrap tyres and may account for the high amount of heat associated with burning tyres.

**Pyrolysis of Scrap tyres (Char)**

The weight of char produced at each pyrolysing temperature is presented in Table 3.

**Table 3: Pyrolysis of scrap tyre samples at constant retention time of 30 mins between the temperature ranges of 250˚C to 600˚C**

| Temperature (˚C) | Initial Weight (g) | Char Weight (g) |
|---|---|---|
| 250 | 1000 | 936.00 |
| 350 | 1000 | 637.00 |
| 450 | 1000 | 581.50 |
| 600 | 1000 | 519.00 |

The result showed a very small change in weight of tyre at 250 ˚C. The weight reduced rapidly at 350˚C after which there was a relatively small reduction in weight. As pyrolysing temperature increased, there was a corresponding decrease in the weight of the char produced. The rapid reduction in weight at 350˚C unlike at 250˚C may be a precursor to the advent of devolatization in the samples. The amount of gases recorded after each experiment may further make a case for the advent of devolatization in the whole process. This agreed with the assertion that at 450 to 650 $^{o}$C, the volatile constituents of the chars decreased with increasing temperature especially in a rotary kiln reactor as reported by [5, 26].

The result in percentages of the char yield was plotted against pyrolysing temperatures as shown in Figure 2. This showed that as the pyrolysing temperature increased, the char yield decreased. The decline in char yield was rapid between 250˚C and 350˚C and continues till around 400˚C where the decrease in char yield occurs slowly. The rapid change in value between these temperatures may signify the onset of cracking of carbon particles.

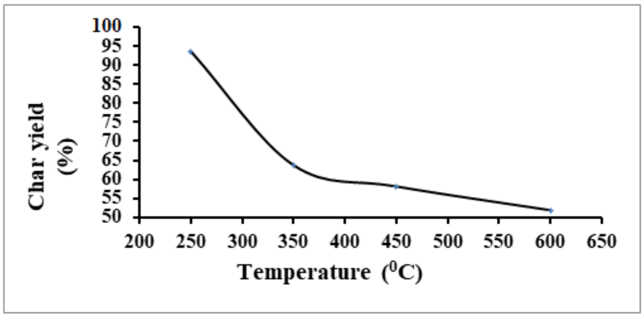


**Fig-2: Effect of Temperature on Char Yield from the Pyrolysis of Scrap Tyres**

**Analysis of Pyrolysed Products**

The ultimate and proximate analyses of the products of tyre pyrolysis are shown in Figs. 3 (a) and (b) to Figs. 6 (a) and (b) for 350 $^{o}$C, 450 $^{o}$C and 600 $^{o}$C.

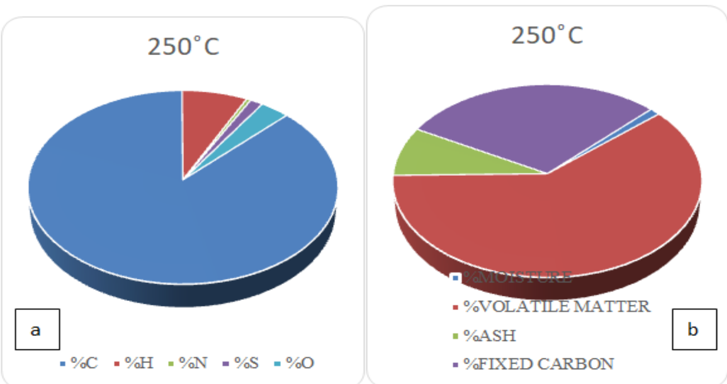


**Fig-3: a) Ultimate analysis, b) Proximate analysis of product of pyrolysis reaction at 250˚C pyrolysing temperature**

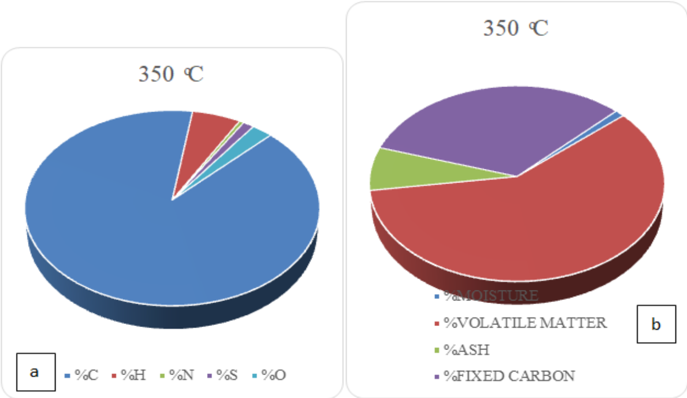


**Fig-4: a) Ultimate analysis, b) Proximate analysis of product of pyrolysis reaction at 350˚C pyrolysing temperature**

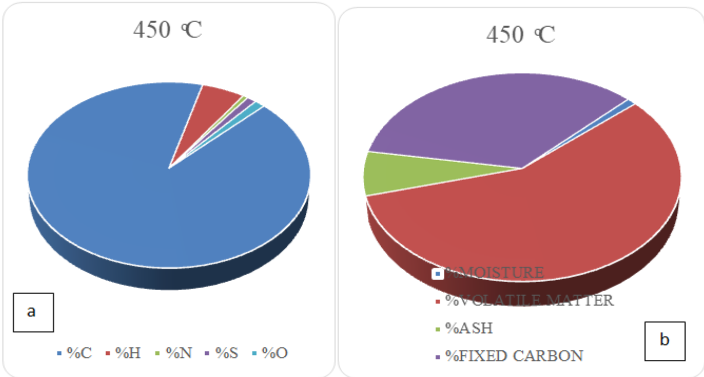


**Fig-5: a) Ultimate analysis, b) Proximate analysis of product of pyrolysis reaction at 450˚C pyrolysing temperature**

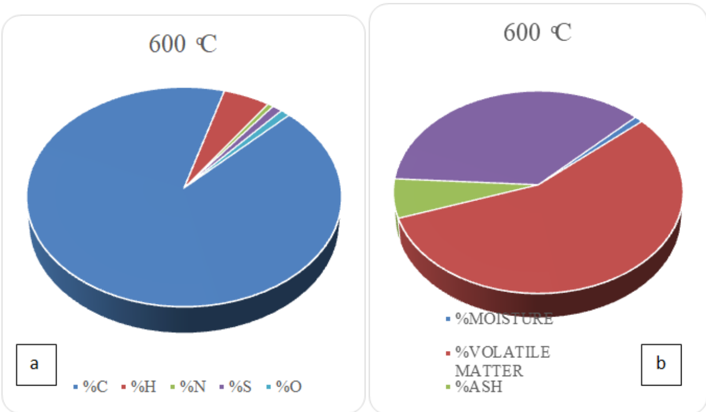


**Fig-6: a) Ultimate analysis, b) Proximate analysis of product of pyrolysis reaction at 600˚C pyrolysing temperature**

Figures 3 to 6 show the ultimate and proximate analyses of each product at each pyrolysing temperature. The physical and chemical analysis of pyrolysed products helped to evaluate the changes in the composition of the pyrolysed product as temperature increased or decreased. The pyrolysis of scrap tyre samples showed that devolatization of the scrap tyres did not start at temperature of 250 $^{o}$C. This was first made obvious by the amount of weight change in the samples after the first experiment. There was however no characteristic change in the physical properties of the pyrolysed products at 250 ˚C compared to the pyrolysed samples at the higher temperatures of 250 $^{o}$C, 450 $^{o}$C and 600 $^{o}$C. The pyrolysis at 250 ˚C produced a char that had close resemblance to the original tyre samples left in the heating/combustion chamber. Some portion of the samples had not even been affected by the heat in the pyrolysis chamber at all. An investigation of the chemical properties of the pyrolysed products at different temperatures further justified the assertions on devolatization at 250 ˚C.

The elemental composition by weight and the ultimate analysis by percentage of the pyrolysed products at different pyrolysing temperatures are presented in Figure 7 and Table 5 respectively.

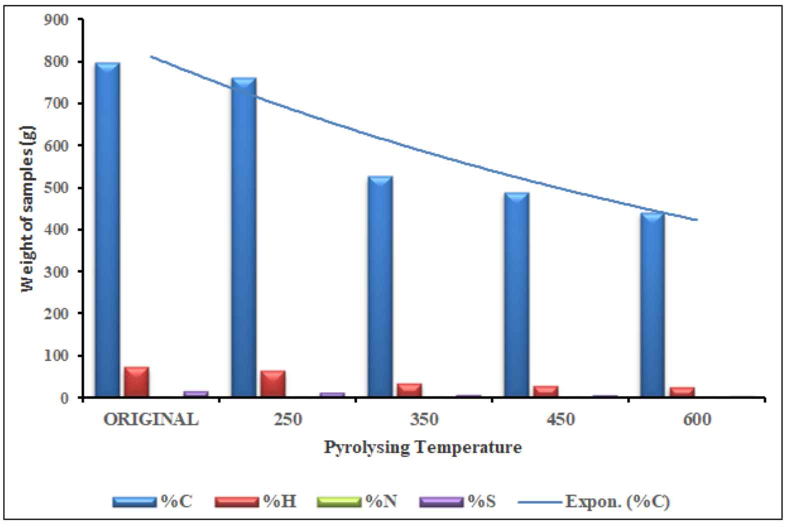


**Fig-7: The elemental composition of the pyrolysed products by weight**

Considering the results detailed by Table 5 and Figure 7, there is an exponential reduction in weight of carbon present in the solid product of the pyrolysis experiment as pyrolysing temperature increases. The rapid change in the weight of carbon between temperatures 250˚C and 350˚C can further corroborate the assertion that devolatization of the tyre starts at temperatures between these pyrolysing temperatures. Comparing the results for the raw tyre samples with those of the average obtained, it can be deduced that hydrogen, oxygen and sulphur are being spent as the process progresses. However, carbon and nitrogen were not decomposed in large quantities throughout the process. This may be due to the absence of oxygen which will readily combine with carbon or nitrogen to form oxides.

In Table 5, the weight of each element was presented as a percentage of the total weight of char produced. It was discovered that the percentage of carbon in the char increases with increased pyrolysing temperature. The percentage of nitrogen took an upward direction like carbon but other elements reduced with increasing pyrolysing temperature. The ash constituent also increased with increase in the pyrolysis temperature.

**Table 5: Results of the ultimate analysis of pyrolysed products by percentage at different pyrolysing temperatures**

| Sample | %C | %H | %N | %S | %O | %Ash |
|---|---|---|---|---|---|---|
| **250$^{o}$C** | 81.37 | 6.84 | 0.45 | 1.34 | 3.10 | 6.90 |
| **350$^{o}$C** | 82.69 | 5.29 | 0.48 | 1.22 | 2.30 | 8.02 |
| **450$^{o}$C** | 84.11 | 4.86 | 0.56 | 1.08 | 1.20 | 8.19 |
| **600$^{o}$C** | 85.01 | 4.81 | 0.58 | 1.04 | 1.00 | 7.56 |
| **Average** | 83.30 | 5.45 | 0.52 | 1.17 | 1.90 | 7.66 |
| **Raw tyre samples** | 79.86 | 7.35 | 0.45 | 1.57 | 6.50 | 4.27 |

The Proximate analysis of pyrolysed products by percentage at different pyrolysing temperatures and the effect of temperature on the heating values of the produced char are presented in Table 6 and Figure 8 respectively.

**Table 6: Proximate analysis of pyrolysed products by percentage at different pyrolysing temperatures**

| SAMPLE | %MOISTURE | %VOLATILE MATTER | %ASH | %FIXED CARBON | HEATING VALUE(kJ/kg) | SPECIFIC GRAVITY |
|---|---|---|---|---|---|---|
| **Raw scrap tyre sample** | 1.25 | 62.10 | 8.67 | 27.98 | 36.10 | 243.00 |
| **250$^{o}$C** | 1.18 | 61.05 | 8.25 | 29.52 | 37.60 | 389.00 |
| **350$^{o}$C** | 1.11 | 59.11 | 7.18 | 32.60 | 38.40 | 437.00 |
| **450$^{o}$C** | 1.04 | 57.24 | 6.88 | 34.84 | 39.30 | 514.00 |
| **600$^{o}$C** | 1.01 | 56.21 | 6.24 | 36.54 | 40.00 | 540.00 |
| **Average** | 1.09 | 58.40 | 7.14 | 33.38 | 38.83 | 470.00 |

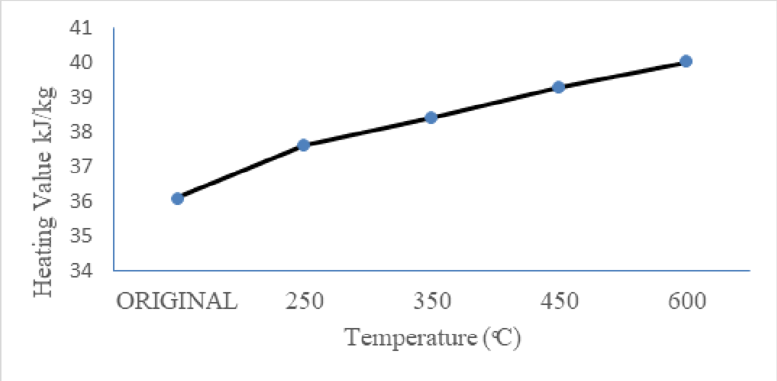


**Fig-8: Plot of Heating Value (kJ/kg) at different temperatures for pyrolysed products**

The experimental results demonstrated an increase in the heating value (HV) concurrently with the increasing pyrolysis process temperature. The individual and average HV of the chars for the three temperatures of 250 °C, 350 °C, 450 °C and 600 °C were higher than the 30 MJ/kg as gross calorific value (GCV) reported by [16] for char obtained from tyre pyrolysis. It can therefore be asserted that pyrolysis at high temperatures can help obtain a more energy content product. Figure 8 shows the measured values of the char HV resulting from the scrap tyre pyrolysis processes realised at different temperatures. The HV of the char increases from 37.6 kJ/kg (pyrolysis at 250°C) to 40 kJ/kg (pyrolysis at 600°C).

## CONCLUSIONS

A mass of 1kg of scrap tyres was pyrolysed in each batch of pyrolysis experiment to obtain char at different pyrolysis temperatures. Proximate and ultimate analyses of the char resulting from the pyrolysis were carried out. The pyrolysis process produced char with an average of 83.30% carbon composition 5.45% hydrogen, 0.52% nitrogen, 1.17% sulphur and 1.90% oxygen composition. The analysis of percentage composition of the raw scrap tyre samples gave 79.86% for carbon, 7.35% hydrogen, 0.39% nitrogen, 1.57% for sulphur and 6.50% for oxygen. There was an increase in carbon content as compared to that of the raw tyre samples. All the remaining compositions reduced with pyrolysis except nitrogen which also experienced an increased percentage composition. The major content of the solid product is however carbon as it has a very high percentage composition in the product after analysis.

The ultimate analysis of the char resulting from pyrolysis gave an average value of 38.83 kJ/kg for the heating value and 470.00 for specific gravity. The average fixed carbon content was 33.38% while the volatile matters gave an average of 58.40%. The ultimate analysis of the raw tyre samples gave values of 36.10 kJ/kg for the heating value and 343.00 for specific gravity. Comparing the heating value of char to the raw tyre sample showed that char has a higher heating value and thus is considered a more viable choice for energy production.

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