

Coconut Shell Pyrolysis for Optimum Charcoal Production

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Abstract. Local charcoal makers take little consideration on the quality of their yield, and often only on the quantity produced. A fixed bed reactor was built and experiments were carried out at final bed temperature ranging from 350°C – 600°C at 50°C intervals at a heating rate of 4°C/min. Maximum charcoal yield was at 39.2% at 500°C. Charcoal yield at every bed temperature were analyzed for proximate analysis. The highest yield had a fixed carbon content, volatile matter and fixed carbon yield of 74.35%, 13.24%, and 28.75% respectively. The lowest yield was 36% at a temperature of 350°C. Its fixed carbon content, volatile matter, and fixed carbon yield were at 29,070.78 kJ/kg and 15, 892 kJ/kg. The 500°C yield is ideal for metallurgy since it had a lower volatile matter content. The lower yield at 300°C is applicable to domestic cooking since ideal volatile matter content for this application is on the range of 20% - 30%.

1. Introduction

Despite the ongoing debate on whether the threat of global warming is real or not, it is quite evident through scientific papers that we are contributing more of these harmful emissions in a rate that would be detrimental to the planet [1]. As the world's fossil fuel dependency have enabled these greenhouse gases to accumulate in the atmosphere at high levels, our present society has realized the actual need for the reduction of these emissions via the use of other forms of energy.

Charcoal have long been an energy source predating back to ancient times. The biomass energy source lists a multitude of applications from domestic cooking to industrial smelting of metals [1]. Researches have listed charcoal as preferred adsorbent for air and water treatment; electrode production [2]; and soil amendments to improve plant growth. Investigations on the potential of charcoal are still on going and its full potential could well be harnessed in the years to come.

Charcoal has been used by a wide range of economic groups in the Philippines. Wood has been, and is still the major source for charcoal production because it is traditionally a free resource. Due to the lack of policy implementation or the lack thereof [3], this energy source is a contributor to the deforestation rate of the country [3]. On the other hand, coconut is an abundant resource in the Philippines. Industries have been built that capitalize on the many functions of this resource from edible oils, copra meat, to lumber. Coconut charcoal is a derivative of its waste – the coconut shell. The local charcoal maker, despite the lack of a systematic procedure where variables such as temperature, heating rate, feedstock properties, etc., which are not accounted for, is still able to produce charcoal of a certain quality which is acceptable as far as domestic cooking or industry activated carbon standards go. Unbeknownst to the charcoal maker, these parameters are of significant importance to the overall quality of charcoal.

The study investigated the characteristics of charcoal from coconut shells via proximate analysis and heating value determination. A fixed bed pyrolysis reactor was built to thermally crack dried

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coconut shells at a slow heating rate of 4°C/min. The shells were heated at final bed temperatures ranging from 350°C – 600°C at 50°C intervals.

2. Literature Review

2.1 Carbonization Efficiency

According to ASTM D 1762-84 [1], proximate analysis of charcoal is heated in a covered crucible to 950 °C and held at this temperature for 6 min. The measured weight loss is defined to be the VM (Volatile matter of the charcoal) and the residual solid is carbonized charcoal i.e.,

$$\%VM = 100 \times (M_{char} - M_{cc})/M_{char} \quad (2.1)$$

M_{char} is the initial dry mass of charcoal and M_{cc} is the dry mass of carbonized charcoal. Ash is determined by heating the carbonized charcoal residue of the VM determination in an open crucible to 750 °C with a soak at this temperature for 6 hrs. The material that remains is ash i.e.,

$$\%Ash = 100 \times m_{ash}/m_{char} \quad (2.2)$$

where m_{ash} is the dry mass of ash that remains following the combustion of carbonized charcoal. Fixed carbon content is then defined as

$$\%FC = 100 - \%VM - \%ash \quad (2.3)$$

The volatile matter of good quality charcoal for cooking is typically in the 20-30% while for the metallurgical industry, the volatile matter usually contains 10-15% or less [1]. The ash content of good quality charcoal typically lies between 0.5 to 5 % resulting in a range of calorific values from 28 to 33 MJ/kg from the 1986 technical report by Gerald Foley entitled Charcoal Making in Developing Countries [1].

Carbonization efficiency [1] is properly given by the fixed carbon content yield:

$$y_{fc} = y_{char} \times [\%FC/(100 - \%ash)] \quad (2.4)$$

Where $y_{char} = m_{char}/m_{bio}$.

Due to the fact that the metallurgical industry employs the fixed carbon content of charcoal to determine its price; and the fixed carbon yield y_{fc} can be compared to the theoretical thermo-chemical equilibrium yield of carbon from feed-stock, there is enough justification in using y_{fc} as the parameter for carbonization efficiency [1] as described in Eq. (2.1).

Energy efficiency conversion [1] of the kiln is defined as

$$\eta_{char} = y_{char} \times (HHV_{char}/HHV_{bio}) \quad (2.5)$$

where HHV_{char} is the higher heating value of charcoal and HHV_{bio} is the higher heating value of the feed-stock.

2.2 Effect of Peak Temperature

Albeit the conditions of pyrolysis in other experiments are not the same (e.g. reactor dimensions, heating rate, feed particle size), the results exhibit a similar trend in terms of the products yielded during the pyrolysis process. A study [4] showed that for a temperature range of 400-600 °C and a heating rate of 20° C/min, the products for coconut shell pyrolysis were as follows:

A maximum liquid yield was obtained at about 575° C. Whereas the char yield decreased from 32% at 400°C to 25.4% at 600°C. The gas yield decreased from 29% to 24% at 575°C and increased from thereon after. Similar results were obtained by Ganapathy and Natarajan [5] wherein at a similar temperature range, liquid yield increased to a point and started decreasing at 550°C; char yield decreased all throughout; and the gas yield increased. The differences in the graphs of the gas yield could be attributed to the minor differences on the controlled conditions of the experiments.

Joardder and Islam [6] hypothesized that the decrease in liquid yield after reaching an optimum temperature could be due to the decomposition of oil vapors into permanent gases and the secondary char reactions. At higher temperatures, the increased gas yield could also result from char loss reactions.

2.3 Effect of Heating Rate

Heating rate of the feed stock influenced the product yields. From 20°C/min to 60°C/min, char yield decreased from 29% to 25% as shown by Sundaram and Natarajan [5]. The results also showed an increase in liquid yield and gas yield.

2.4 Vapor residence time

The residence time of the pyrolysis gases inside the reactor affected the yield of the products. It was found out that the effects of holding the vapors longer promoted secondary char formation. This was investigated by Sundaram and Natarajan [5] by varying the length of the reactor. Pyrolysis of the coconut shells were done at a reactor having lengths of 200mm and 300mm at varying feedstock particle sizes. The results show that a higher liquid yield was produced by the 200mm reactor. The 300mm reactor produced more gas and solid char.

Mok et al [7] demonstrated the effects of gas flow on the yield of char cellulose pyrolysis and showed that lower gas flows provide reactive volatile matter more time to thermally charck thereby producing more charcoal as a result. This was further corroborated by Vahegyi et al [8] which showed more char yield when an open sample pan was covered.

2.5 Running/holding time

The amount of time which a certain peak temperature is being held is defined to be the running/holding time. Several tests have been conducted to illustrate its effects on the products of pyrolysis. The methods conducted by these tests involved the testing of these pyrolysis runs at certain holding times [6]. Increasing the holding time from 50 min to 100 min marked an increase in charcoal yield. The bio-oil and non-condensable gases however, increased and stagnated when it reached the 100 min mark. Joardder et.al. [6] concluded that pyrolysis ends at approximately 100 min for coconut shells. However, for industrial slow pyrolysis units, a 100 min running time is not economically feasible. Crombie et al. [9] optimized their experimental set-up and had the running times minimized to 10, 20 and 40 min. In a related study [5], the pyrolysis run was only terminated whenever the non-condensable gases venting out of the condenser were no longer significantly visible.

3 Materials and methods

3.1 Raw feed stock collection and analysis

Coconut shells, already removed of its meat and kernel, were collected from a wet market stall located in Mandaue City, Cebu, Philippines. The shells were sun dried for 3 days to remove adequate moisture. The dried coconut shells were subjected to a proximate analysis test to determine its gross components such as moisture, volatile matter, ash, and fixed carbon according to ASTM D3172-13. The heating value was determined through the use of an Oxygen Bomb calorimeter conducted in the Department of Mechanical and Manufacturing Engineering ME Laboratory of the University of San Carlos as per ASTM D4809-95. The dried coconut shells were broken into pieces by a ball peen hammer with an approximate area of 2 cm x 2 cm before being loaded inside the reactor. The broken pieces were measured by a Vernier caliper.

3.2 Experimental setup

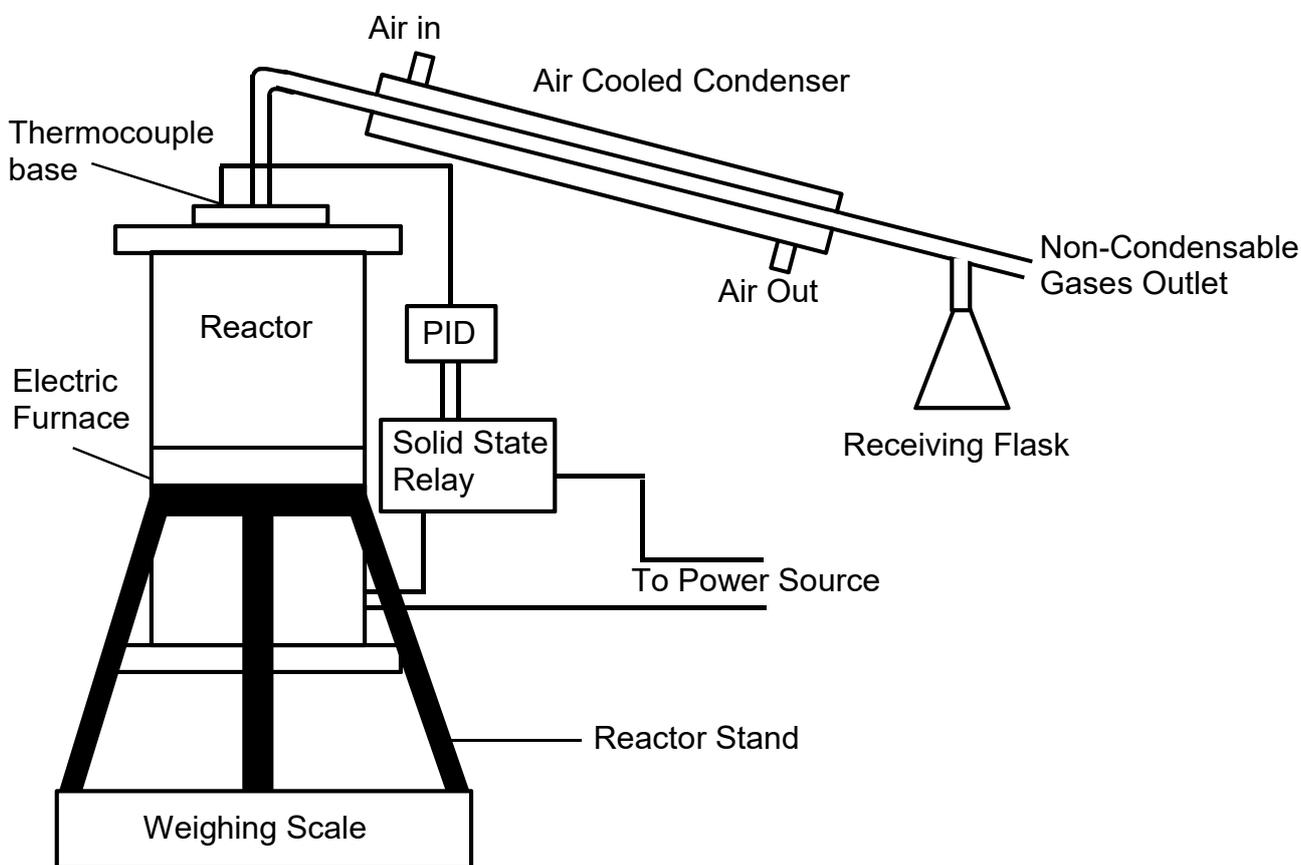


Fig. 1 Experimental Setup: A thermocouple is securely locked on top of the reactor that is connected to the PID controller. The gases evolved out of the reactor were condensed in an air-cooled condenser. Condensable matter is collected onto a receiving flask while non-condensable gases are vented out. Temperature and weight were recorded at 1-minute intervals until the pyrolysis process was completed.

A fixed bed pyrolysis reactor, seen in Fig 1, was built by insulating a 141.30 mm diameter stainless steel pipe by cylindrical layer made from refractory cement having a thickness of 77.50 mm. Nichrome wires were wound on the bottom part of the reactor that provided heating power. The reactor was

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designed with a 4-kW heating capacity. The whole assembly was placed on top of a digital weighing scale through a three-legged stand intended for the experiment.

The weighing scale recorded the changes in weight as the pyrolysis process commenced. 500 g of broken dried coconut shells were loaded inside the reactor and heated to final bed temperatures of 350°C - 600°C at 50°C intervals at a heating rate of 4°C/min. A PID controller, connected to a multipoint thermocouple, was used to control the heating rate.

At each final bed temperature (350°C, 400°C, 450°C, 500°C, 550°, 600°C), temperature and weight were recorded at 1-minute intervals until the pyrolysis process was completed. Completion of the pyrolysis process was identified by a constant weight reading at 10 intervals (10 minutes). This indicated that there is no more significant amount of volatile matter coming out of the coconut shell feed stock. The gases coming out from the reactor were condensed by an air-cooled condenser. The non-condensable gases were vented out from the condenser while the condensable matter was collected onto a receiving flask. Charcoal produced from the experiment was collected at the bottom of the reactor through a threaded steel cap at the bottom.

3.3 Charcoal Analysis

Charcoal yield at different final bed temperature were calculated using the equation:

$$y_{char} = \text{Charcoal yield}\% = \frac{\text{Charcoal product weight}}{\text{feedstock weight}} \times 100 \quad (3.3)$$

A proximate analysis on every charcoal yielded was conducted. The method used corresponded to the ASTM D 1762-84 procedure for the analysis of wood charcoal. Charcoal samples were first crushed in a hammer mill and screen filtered for a powdered output. The milled charcoal was then dried in an oven at a temperature of 110°C for 1 hour to obtain the percent content of moisture. Drying involved the use of a drying oven. The dried charcoal powder was then heated in a petri dish. A flame at the top end of the test tube was produced due to the excretion of volatile gases. After the flame was extinguished, calculations for the percent volatile matter was then calculated by subtracting the mass before and after all the volatile gases were excreted.

The resulting mass obtained is composed of the fixed carbon and ash content of the yielded charcoal. In obtaining the ash content, the sample was further heated until its embers dissipated and disappeared. The remaining material is the ash content of the charcoal.

3.4 Heating value determination

A Parr 1341 plain jacket bomb calorimeter was used in the determination of the heating value. 1 gram of crushed charcoal was used as the sample specimen. At 30 psi, the bomb was detonated inside the jacket and its temperature rise at specific time points were recorded. From the data obtained. The heating value was determined based on the equations provided by the Parr 1341 Plain Jacket Bomb Calorimeter Manual.

3.5 Carbonization efficiency

As discussed in section 2.1, the fixed carbon yield y_{fC} is a better measure of carbonization efficiency than the charcoal yield y_{char} . This is due to the fact that y_{char} does not take into account the fixed carbon content and moisture of the wood as expressed by Eq. (3.3). The fixed carbon yield in equation (3.5) represents the efficiency realized by the pyrolysis conversion of the ash-free organic matter in the feed-stock into a relatively free ash-carbon [1].

$$y_{fC} = y_{char} \times [\% fC / (100 - \% feedash)] \quad (3.5)$$

4. Data and Results

4.1 Charcoal Yield

From Fig. 2, it is shown in the graph that the highest yield (black line) with respect to its weight is at the 500°C final bed temperature. From 350°C to a 500°C, the yield increased and reached its maximum value at 500°C. Starting from the 500°C point, the yield decreased having its minimum value on the 600°C bed temperature setting. Results from [5] presented a different trend. Although the heating rate used was 60°C/min, coconut shells pyrolyzed at increasing bed temperatures had a decreasing trend with the maximum yield at 350°C and the minimum yield at the highest temperature of 600°C. Graphs from a similar study [6] illustrated an identical trend to that of Sundaram and Natarajan [5]: charcoal yield decreased with increasing bed temperature.

Although the charcoal yield from Joardder et al [6] are higher than the other graphs from Fig. 2, the trend is similar to that of the other related studies. Charcoal yields are inversely proportional to that of bed temperature. This is due to the extended cracking of the charcoal inside the reactor giving rise to an increase in bio-oil and gas yield [1]. It was only that of the data obtained in the study that an increase in yield was experienced from 350 °C to 500 °C.

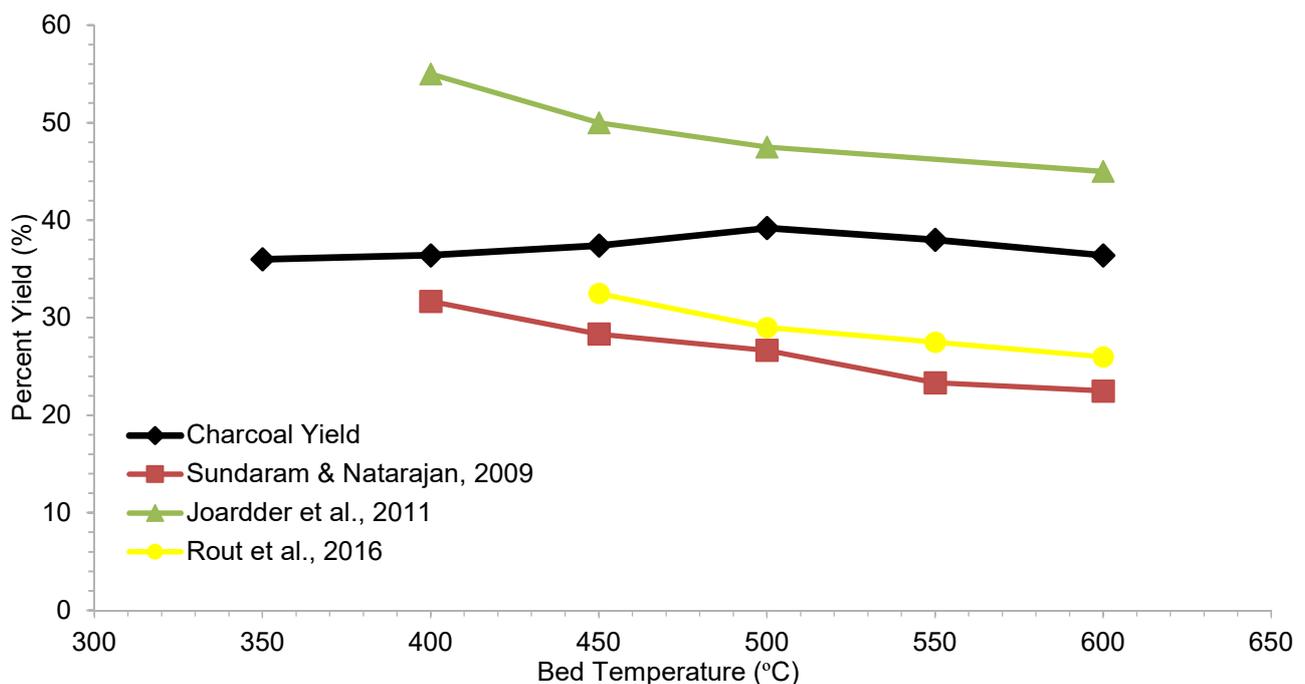


Fig. 2 Charcoal yield and related studies

4.2 Mass Loss Curves

The graph in Fig. 3 illustrates the behavior of the coconut shells subjected to different final bed temperatures (600°C, 550°C, 500°C, 450°C, 400°C, 350°C). The steep decline in slope within the 400°C to 500°C range indicated a hastened reduction in weight. This reduction was caused by the rapid evolution of gases from pyrolysis. At this stage, volatile and non-condensable gases are being exhausted out from the raw coconut shells. The curve for the 550°C temperature explicitly demonstrated the pyrolysis phenomenon. A steady decline in weight can be clearly seen. The weight decreased because moisture leaves the coconut shells once heating commences. At around 425°C, approximately all of the moisture was removed. Starting at this point, the thermal cracking of the coconut shells produced volatile and non-condensable gases at a rapid rate with regards to the rate of moisture removal. Except for the 350°C curve, it can be seen visually that the graphs behaved in the same manner with the steep drop in weight beginning approximately at the 400°C mark and maintained a constant value at around 430°C to 500°C. It can be observed from the graph in Figure 3 that a decrease in temperature occurred during the 350°C run. The explicit decrease in temperature is caused by the endothermic reaction occurring at this stage. Pyrolysis becomes endothermic at this point where heat is needed for the thermal cracking of the volatile gases into charcoal [1].

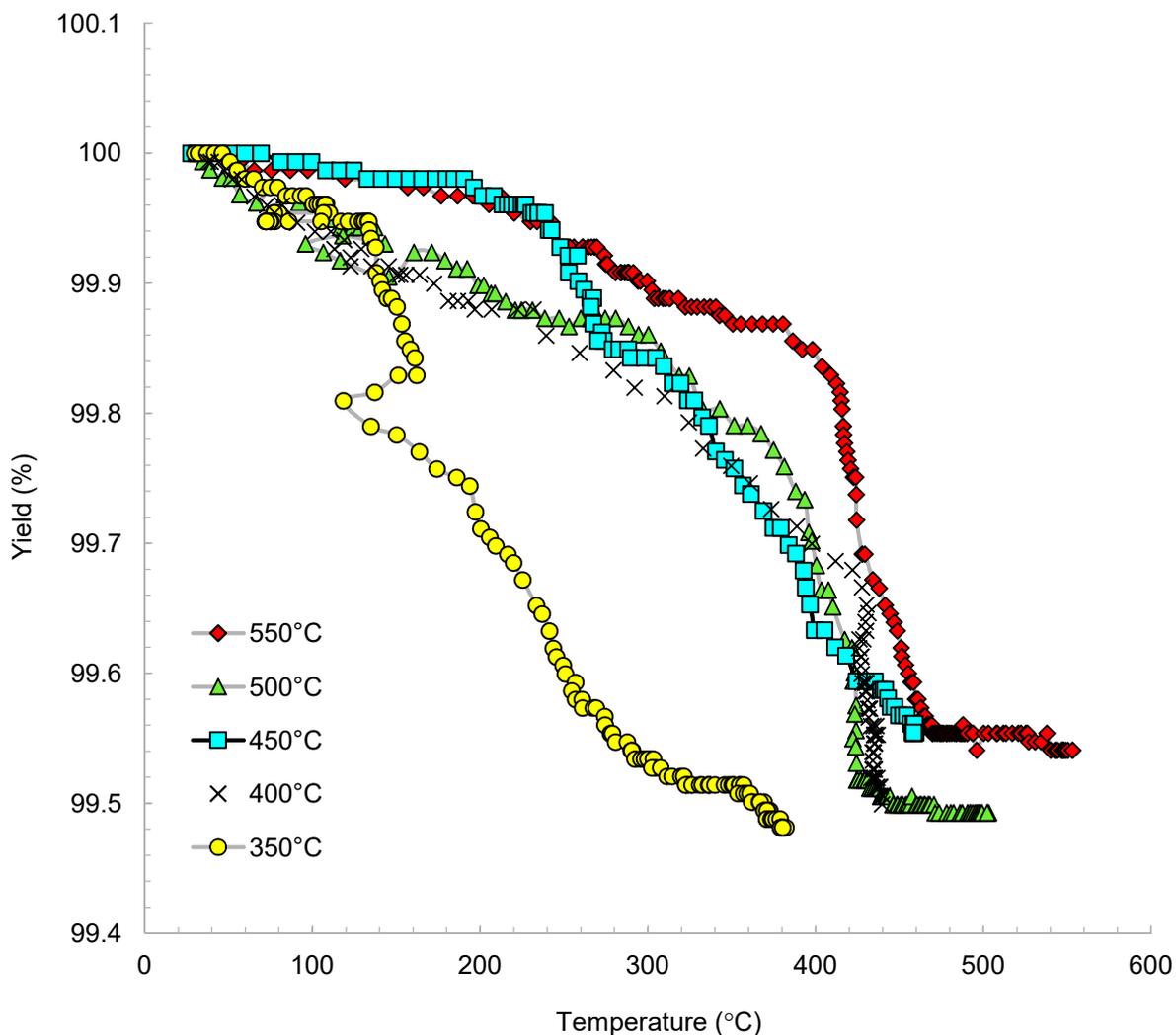


Fig. 3 Mass loss curve at different final bed temperatures

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4.3 Proximate Analysis, Heating Value, and Fixed Carbon Yield

The heating values obtained in Table 1 have increased in value when the bed temperature was increased. This is similar to the study [10] cited when grape bagasse was pyrolyzed from 300 °C to 900 °C. The heating value increased together with its fixed carbon content while the volatile matter decreased. Moisture on the other hand had no direct relationship with increasing bed temperature.

Table 1 Charcoal Yield, Heating value, and Composition

Bed Temperature (°C)	Yield (kg)	Heating Value (kJ/kg)	Proximate analysis			
			Moisture (db)	Volatile Matter (%)	Fixed Carbon (%)	Ash (%)
350	0.180	15892.34	5.65	36.47	53.60	4.28
400	0.182	26947.90	2.73	18.93	71.30	7.04
450	0.187	28067.85	2.95	16.21	78.60	2.24
500	0.196	29070.78	8.04	13.24	74.35	4.37
550	0.190	29273.34	10.55	10.85	78.32	3.22
600	0.182	31082.12	2.22	7.358	88.92	1.50
Coconut Shell	-	23416.00	11.26	80.98	18.36	2.42

The randomness of the obtained moisture values could be credited to the storage and handling of the sun-dried coconut shells. The sun-dried shells were stored in a container inside the laboratory with no special precautions on how to maintain the moisture the whole time the study was conducted. Environmental moisture is speculated to be a strong culprit on why the shells never achieved uniform moisture content.

Table 2 Fixed carbon yield and carbonization efficiency

Bed Temperature (°C)	Charcoal Yield (kg)	Charcoal Yield (%)	Fixed Carbon (%)	Fixed Carbon Yield (%)
350	0.180	36.00	53.60	19.30
400	0.182	36.40	71.30	22.95
450	0.187	37.40	78.60	29.40
500	0.196	39.20	73.35	28.75
550	0.190	38.00	78.32	29.76
600	0.182	36.40	88.92	32.37

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Table 2 tabulates the fixed carbon yield on the last column along with the charcoal yield and fixed carbon determined from the proximate analysis procedure. The fixed carbon yield describes the carbonization efficiency of the whole process. This efficiency, represented by Eq. (3.5), states the conversion of the ash free organic matter in the feed stock into a relatively pure ash-free carbon. Since the metallurgical industry uses the fixed carbon yield to determine the price, and its value could be well compared to the theoretical thermochemical equilibrium yield of carbon from feed stock [1], the fixed carbon yield is considered by the researcher to be a good indicator on the quality of charcoal. At increasing bed temperatures (350°C, 400°C, 450°C, 500°C, 550°C, 600°C), Fixed Carbon Yield increased attaining a maximum value of 32.37% at 600°C. Both Fixed Carbon and Fixed Carbon Yield exhibited an increasing trend with increasing temperature despite the decrease Fixed Carbon from 450°C to 500°C.

5. Conclusion

Despite the difference in results from related studies, the highest charcoal yield was attained at the 500 °C experimental run. Contrary to the phenomenon that charcoal yield decreases with increasing temperature, the study exhibited that coconut shell charcoal increased its yield at 500 °C and subsequently decreased at 600 °C. The final bed temperature of 600 °C had the highest heating value obtained at 31,082.12 kJ/kg. At the highest yield of 196 g at the 500 °C final bed temperature, 13.4% of the total charcoal mass was found out to be volatile matter. From an industrial standpoint, charcoal having 13.4% volatile matter is ideal for metallurgical smelting while 20 – 30% is good for domestic cooking [1]. Depending on the user's preferences, charcoal from a final bed temperature of 350 °C is ideal for home cooking while that of the 600 C°, which has the lowest volatile matter percentage of 7.358%, is good for industrial smelting. For carbon sequestration, the charcoal yield with the lowest volatile matter, and therefore the highest fixed carbon, is highly essential for the minimization of CO₂ in the environment [11]. Except for the 400 °C experimental run, all of the charcoal yields have ash percentages of less than or equal to 5%, which is an indication of good quality charcoal. Although the carbonization efficiency was increasing as represented by the fixed carbon yield tabulated on Table 2, efficiencies are still below 35% and are relatively low. A low efficiency indicates a need to redesign a better reactor for the process. Further studies are recommended that investigates other parameters that affect the yield of charcoal from pyrolysis.

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