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Effect of Temperature and Residence Time on Product Yields during Fixed-Bed Pyrolysis of Siam Weed (*Chromolaena Odorata*)

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Abstract

Thermochemical conversion of biomass into biofuels is a promising approach to renewable energy. The widespread proliferation of Siam weed in Nigeria, coupled with its limited utilization as an energy or chemical resource, has resulted in significant environmental concerns. This research investigated how selected operating parameters influence the distribution of products obtained from the pyrolysis of Siam weed using a fixed-bed reactor system. The biomass feedstock was sourced from the National Centre for Agricultural Mechanization, Ilorin, Kwara State, Nigeria. Prior to experimentation, 100 g of oven-dried Siam weed was charged into an airtight metallic retort for thermal conversion. The retort was then transferred to a furnace, wherein pyrolysis was performed. Initial trials happened at 300 °C, with residence time starting from 10 to 30 minutes in 5-minute intervals. This procedure was systematically repeated at increased temperatures of 350, 400, 450, and 500 °C. Following every run, the product yields of char, tar, and gas were quantified. The experimental outcomes established an extensive range in product yields depending on the pyrolysis conditions. The minimal char yield recorded was 35.85 wt% (at 500 °C), while the maximum reached 73.13 wt% (at 300 °C). Tar yields varied from a low of 7.07 wt% (at 300 °C) to a high of 16.55 wt% (at 450 °C). Gas production ranged from 19.81 wt% (at 300 °C) to 52.94 wt% (at 500 °C). This study highlights the tremendous capability of siam weed pyrolysis as a viable source for biofuels, contributing to sustainable development..

Keywords: Pyrolysis, Fixed bed reactor, Siam weed, Biofuels, Char, Tar and Gas.

Introduction

The world's energy demand is on the increase because of rapid population growth (He et al., 2024). Economic development, among other factors, has accelerated the consumption of the limited petroleum derivatives, which are the most important and widely consumed in society, providing fuel for energy, transportation, and industries (Akinrinade et al., 2023). Oil-based commodities are crude materials for some chemical products, including drugs, solvents, manures, pesticides, and plastics; nonetheless, petroleum reserves are limited and non-renewable. About 90% of the world's energy consumption is derived from petroleum (Okekunle et al., 2018). High levels of impurities like carbon dioxide (CO2), sulphur oxides (SO2, SO3), and nitrogen oxides (NOx) have resulted from the development and use of non-renewable energy sources. As a result, the environment has suffered greatly, causing contamination of water sources, natural resources, and food sources, as well as global warming and severe harm to humanity (Cinthia et al., 2015).

The main reasons driving the search for alternative and renewable energy sources are worries about these problems. A few different types of research have been done to look for clean, abundant, and aesthetically pleasing fuel options (Moudene et al., 2023). The production of biofuel from biomass has drawn a lot of

attention because it has the potential to minimize environmental pollution, halt global warming, and lessen reliance on finite fossil fuels. Nigeria, despite its status as a petroleum-exporting nation, continues to experience persistent challenges related to energy supply, including inadequate availability of refined fuels and escalating fuel costs. These conditions underscore the urgent need for the country to diversify its energy portfolio by developing and adopting sustainable alternative energy sources (Rabbani et al., 2024).

Despite being an oil-producing country, Nigeria faces extreme power crises, petroleum product shortages, and relentlessly growing fuel prices, necessitating the pressing pursuit of alternative power sources (Takahashi et al., 2020). Nigeria needs to prioritize and motivate researchers into biofuel production, using available and economically viable feedstocks, to mitigate its dependence on fossil fuels. Siam weed (*Chromolaena Odorata*) is an excellent example of such feedstock because they have comparatively few competing foods uses, which encouraged research into the potential for biofuel production (Tiamiyu and Okunlade, 2020).

The primary sustainable source of carbon is biomass, which makes it an acceptable feedstock for chemical production as well as for the generation of fuel (Adewumi et al., 2023). Biomass resources embody a wide range of organic substances, along with natural sources like woody and herbaceous plant species, grasses, aquatic plants, and algae. They also encompass a broad range of by-products and discarded materials such as wood offcuts, sugarcane residue, farm and industrial by-products, discarded paper materials, municipal refuse, wood shavings, treated organic sludge, and residues generated from food-processing activities (Giakoumatos and Kopsidas, 2021).

Siam weed (*Chromolaena Odorata*) has been dubbed one of the most dreadful tropical weeds in the world due to its rapid invasion, easy establishment, and ability to cover existing tropical organic product crops, fruit crops, immature forestry plantations, and pastures (Djitie et al., 2025). It poses a substantial fire risk during the dry season, invades nearby woods and ecologically sensitive areas and has been reported to have an extremely high nitrate content, causing cattle to die and give birth early when fed with polluted fodder (Ogunjobi et al. 2016).

Utilizing a variety of technologies, such as thermochemical processes and biochemical/biological processes like fermentation and aerobic digestion, it is possible to convert biomass into a useful source of energy (Itabiyi et al., 2019). In contrast to fermentation and aerobic digestion, which have specific requirements for the biomass feedstock they can use, the thermochemical conversion processes, which include pyrolysis, gasification, and combustion, represent one of the most promising future energy alternative pathways (Ogunsuyi and Olawale, 2021).

Pyrolysis has attracted more interest among these conversion techniques for the production of liquid fuel products because of its potential advantages in storage and transportation as well as its usefulness in applications like combustion engines, boilers, turbines, etc. (Osung and Alabi, 2022). In the pyrolysis method, the solid, liquid, and gaseous product yield generally depends on various operating parameters like pyrolysis temperature, residence time, heating rate, and type of reactor used (Itabiyi et al. 2019). Similarly, biomass properties consisting of composition, particle size, and density significantly have an effect on each the quality and quantity of pyrolysis products. Fast pyrolysis, leveraging excessive heating rates and shorter vapour residence times, constantly yields more pyrolytic oil than gradual pyrolysis, which employs decreased heating rates and prolonged residence times (Khraisha, 2022).

Prior studies on biomass and coal pyrolysis show that increased temperatures cause decreased char yields because of the expanded incidence of gasification reactions at higher temperatures (Itabiyi et al. 2019). Specifically, for agricultural residues, a lower char yield has been found with higher temperatures, smaller particle sizes, and increased heating rates (Itabiyi and Lucas, 2016). Furthermore, Itabiyi et al. (2018) discovered that increased temperatures and smaller particle sizes promoted the cracking of hydrocarbons, resulting in a higher hydrogen content. Consequently, this study aimed to examine the results of temperature and residence time on product yield at some stage in the pyrolysis of siam weed in a fixed-bed reactor.

Materials and Methods

Feedstock procurement and preparation

The Siam weed employed for the pyrolysis investigations was obtained from the National Centre for Agricultural Mechanization located in Ilorin, Kwara State, South-Western Nigeria with coordinates approximately 8.41° N, 4.78° E. Prior to analysis, the biomass was carefully sorted and cleaned to remove unwanted materials, including soil particles, stones, and other impurities. The initial mass of each sample (W_1) was measured using an Ohaus digital top-loading balance (Model PA4102; capacity 0-4100 g; readability ±0.001 g). Thereafter, the samples were dried in a laboratory oven at 105 °C until constant mass (W_2) was achieved, in accordance with the standard procedure outlined in ASTM D5373-02 (2005).

The proximate and ultimate analysis

The physicochemical characterization of the Siam weed biomass was performed following established standard test methods. Proximate and elemental evaluations were conducted in accordance with relevant ASTM and AOAC protocols. The proximate assessment quantified moisture, ash, volatile constituents, and fixed carbon fractions, while the elemental analysis determined the proportions of carbon, hydrogen, nitrogen, sulphur, and oxygen. In addition, the material's chemical composition and calorific value were evaluated to further describe its fuel characteristics.

The moisture content determination

The moisture level of the Siam weed samples was evaluated using a gravimetric approach in line with the AOAC (2012) standard. About 2 g of each prepared sample was placed in a crucible and dried in an oven maintained at 103 °C until a constant mass was attained, typically after a 24-hour period. The moisture content percentage was subsequently computed using Equation 1.

$$\%MC = \frac{(W_i - W_f)}{W_i} X\ 100 \quad (1)$$

Where: W_i is the initial weight of the sample (g), and W_f is the final weight of the sample (g)

The ash content determination

The Percentage Ash Content ($\%AC$) of siam weed was examined gravimetrically in line with ASTM D5373-02 (2005). The technique comprises oven-drying 2 g of the sample in an electric-powered oven for 24 hours. The dried sample was then powdered, and a specific 2 g aliquot was weighed right into a pre-weighed crucible. Following this step, the samples were combusted in a muffle furnace maintained at 550 °C for a duration of 8 hours. Upon completion of the heating process, the crucibles containing the residual ash were carefully withdrawn, allowed to cool in a desiccator to prevent moisture uptake, and then weighed. The ash content was subsequently expressed as a percentage using Equation 2.

$$\%AC = \frac{W_a}{W_s} \times 100 \quad (2)$$

Where: W_s is the weight of the sample (g), and W_a is the weight of ash (g)

The volatile matter determination

The Percentage Volatile Matter ($\%VM$) was examined in line with ASTM D5373-02 (2005). The technique concerned pulverizing 2 g of every sample and putting it in a porcelain crucible, which was then heated in an oven till a steady weight was achieved. The prepared sample was then subjected to thermal treatment in a furnace operated at 550 °C for a period of 10 minutes. After removal from the furnace, the material was transferred to a desiccator to cool under controlled conditions before its mass was recorded. The volatile matter content was subsequently determined as a percentage using Equation 3.

$$\%VM = \frac{(W_x - W_y)}{W_s} \times 100 \quad (3)$$

Where: W_s is the weight of the sample (g), W_x is the weight of the dry sample (g), and W_y is the weight of residue (g)

The fixed carbon content determination

The fixed carbon content (%FCC) of the feedstock was determined following the ASTM D5373-02 (2005) standard. This value was calculated by subtracting the combined percentages of moisture (MC), volatile matter (VM), and ash (AC) from 100, as expressed in Equation 4.

$$\%FCC = 100 - (MC + AC + VC) \quad (4)$$

The heating values determination

The Higher and Lower Heating Values in (HHV) and (LHV), respectively, of the siam weed sample were examined with the aid of a bomb calorimeter, following ASTM E1019 (2011). This involved initiating the stoichiometric combustion of moles of hydrogen and one mole of oxygen in a metallic box at 25 °C, permitting the reaction to finish and produce water vapour. For HHV, the heat released was measured after cooling the vessel and its contents returned to 25 °C, ensuring all product water condensed to liquid. For LHV, cooling was stopped at 150 °C, which means the water remained in its vapour phase, and only partial heat was recovered. This 150 °C threshold is frequently precise because of its relation to the acid gas dew point. Ultimately, HHV calculations consider product water in liquid form, at the same time as LHV calculations assume it remains in vapour form, with specific formulation supplied in Equations 5 and 6.

$$HHV = 4.18 \times (78 \times C + 241.3 \times \left(\frac{HxO}{8}\right) + 22.1 \times S) \quad (5)$$

$$LHV = 4.18 \times (94.19 \times C - 0.550 - 52.14 \times H) \quad (6)$$

Where: C is the percentage of carbon content, H is the percentage of hydrogen content, O is the percentage of oxygen content, and S is the percentage of sulphur content

The carbon content determination

The carbon content (%C) of the sample was determined following the procedure outlined in ASTM D5373 (2005). In this method, the carbon present in the sample is first oxidized to carbon dioxide by combustion in a continuous stream of oxygen. The generated carbon dioxide is subsequently absorbed onto a zeolite material, then released through controlled heating and carried by a carrier gas—commonly helium or oxygen—into a chromatographic column for analysis. The concentration of carbon dioxide is measured using a thermistor-based conductivity detector, which provides precise quantification. Finally, the percentage of carbon in the original sample is calculated based on these measurements, as described in Equation 7, ensuring accurate and reproducible results.

$$\%C = \frac{(A-B) \times C}{D} \quad (7)$$

Where: A is a digital volumetric reading for the sample, B is a digital volumetric reading for blank, C is the mass compensator setting, and D is the Sample setting

Nitrogen content determination

Nitrogen content (%N) was examined with the aid of the Kjeldahl method (Ibitoye, 2005). Approximately 0.5-1.0 g of dried sample was weighed right into a digestion flask, to which 20 mL of concentrated H_2SO_4 and approximately 0.2 g of selenium catalyst were added. The flask was swirled for thorough mixing, then heated till the combination became clear. After cooling, the digest was quantitatively transferred to a 100 mL volumetric flask and diluted to the mark with distilled water.

Distillation of the digested sample was performed using a Markam Still Distillation Apparatus to determine the crude protein content. In this procedure, 10 mL of the digested sample was carefully transferred into the distillation tube, followed by the gradual addition of 10 mL of 40% sodium hydroxide (NaOH) solution to facilitate the release of ammonia. The distillation process was maintained for a minimum of 10 minutes, during which the evolved ammonia (NH_3) was absorbed as ammonium borate in a conical flask containing 5 mL of 2% boric acid solution along with a few drops of a modified combined indicator to enable endpoint detection. The collected distillate was then titrated against a standard 0.002 M hydrochloric acid (HCl) solution until a stable pink colour appeared. A reagent blank was processed alongside the samples to ensure accuracy, and the crude protein content was subsequently calculated using Equation 8.

$$\%N = \frac{(S-B) \times N \times 0.014 \times V1 \times D}{Ws \times V2} \times 100 \quad (8)$$

Where: S represents the titration volume of the sample, B denotes the titration volume of the blank, N is the normality of the HCl solution, D indicates the dilution factor of the sample after digestion, V_1 is the total volume of the digested sample, V_2 is the portion of the digested sample used for distillation, 0.014 corresponds to the milliequivalent weight of nitrogen, and Ws is the mass of the sample in grams.

Sulphur content determination

The sulphur content (%S) of the sample was determined according to ASTM E1019 (2011). For the analysis, 1.0 g of dried sample was combined with approximately 1.5 g of an accelerator in a crucible and combusted in a stream of oxygen, converting the sulphur present into sulphur dioxide. The resulting sulphur dioxide was quantified using infrared absorption spectrometry, and the percentage of sulphur was calculated as shown in Equation 9.

$$\%S = \frac{(A-B) \times Y}{Z} \quad (9)$$

Where: A is the Digital Volt Meter (DVM) reading for samples, B is the DVM reading for blank, Y is the mass compensator setting, and Z is the sample mass (g).

Hydrogen content determination

The hydrogen content (%H) of the sample was determined in accordance with ASTM D5373 (2005). In this procedure, a 0.5 g portion of the sample was combusted in a stream of oxygen at around 1350 °C. Any products of incomplete combustion were passed over copper oxide to ensure complete conversion of all carbon and hydrogen into carbon dioxide and water, respectively. The hydrogen percentage was then calculated using Equation 10.

$$\%H = \frac{W_w \times 0.119 \times 100}{W_s} \quad (10)$$

Where:W_w is the weight of water (g), and W_s is the weight of the sample (g).

Oxygen content determination

The Percentage of Oxygen (%O) was determined by ASTM D5373 (2005) by using Equation 11

$$\%O = 100 - (C + H + N + S + \%ASH) \quad (11)$$

The effect on the selected parameter

Pyrolysis experiments were performed to analyze the effect of diverse parameters on the product yields from siam weed. In every run, 100g of dried siam weed was loaded right into a retort. The retort was finally positioned in a furnace and pyrolyzed at temperatures of 300, 350, 400, 450, and 500 °C. Volatile products were channelled via a pipe to an ice-cooled condensate receiver, taking into consideration the fast collection of liquid tar. The non-condensable gases were directed through an insulated pipe into a gas collection system. The residual char in the retort and the collected tar were weighed using an Ohaus top-loading digital balance, while the gas yield was determined by difference in mass. The percentages of the various product yields were then calculated according to Equation 12.

$$\text{Percentage product yield Y} = \frac{M_y}{M_x} X\ 100 \quad (12)$$

Where M_y is the mass of the product, and M_x is the Mass of the sample

Results and Discussion

Proximate and ultimate analysis of the siam weed

The proximate and ultimate analysis outcomes for siam weed is presented in Table 1. The moisture content of the siam weed was 6.38%. The moisture level in biomass feedstock is understood to noticeably affect the stable phase temperature through exothermic evaporation, in addition to the overall power input essential for pyrolysis. Siam weed`s ash content was determined at 9.22%. The quantity and chemical composition of ash are essential elements influencing biomass fuel properties and, consequently, the choice of conversion technology (Adewumi *et al*., 2023).

A decrease in ash content is indicative of decreased slagging and fouling outcomes and is normally related to higher heating values. Although ash components no longer make a contribution to the general heating value, certain factors and alkali metals within ash can act as catalysts for thermal decomposition. Moreover, low ash content has additionally been proven to optimize bio-oil yields (Bhattacharjee and Biswas, 2019). The volatile content for siam weed was 25.55%. This combustible volatile matter is instrumental in beginning pyrolysis with the aid of using releasing flammable gases at higher temperatures. The last evaluation of siam weed, additionally provided in Table 1, discovered its major elemental constituents: 52.80% carbon (C), 19.22% hydrogen (H), and 31.78% oxygen (O_2).

The relatively decreased heating values of those samples, when contrasted with traditional fuels, are normally due to their increased oxygen content. Nevertheless, this excessive oxygen content positions them as a promising chemical feedstock. For instance, Siam weed`s nitrogen content is 0.65%, aligning with the range discovered in other agricultural residues, which includes broomweed (0.52%) (Adewumi *et al.*, 2023). Its sulphur content is 3.82%. Crucially, the commonly low percentages of nitrogen and sulphur in those samples suggest a significant environmental benefit: fuels derived from them could be, in large part, free of the harmful nitrogen and sulphur oxides feature of petroleum-based fuels. The cellulose content for siam weed is 63.99%.

The value is comparable with the findings of other agricultural residues, such as oil palm husk (51.3%) and broom weed (72.83%). The hemicellulose content for siam weed is 6.96%. The concentration of cellulose and hemicellulose contents of biomass feedstock is higher than lignin, so it will produce higher pyrolytic-oil yield during pyrolysis (Itabiyi *et al.,* 2019). The value for lignin content for siam weed is 2.28%. Lignin contributes significantly to the formation of char. Lignin is a rich source of phenolic compounds, which end up as tar upon thermal degradation (Kumar *et al.,* 2018). The higher heating value of siam weed is 20.85 MJ/kg. The value was compared to other agricultural residues such as broom weed and palm kernel shell (Adewumi *et al,* 2023; Itabiyi *et al,* 2019).

Table 1: Results of proximate and ultimate analysis of siam weed

Percentage Composition (%)	Siam weed
Moisture content	6.38
Ash content	9.22
Volatile matter	25.55
Fixed carbon content	64.65
Carbon	52.80
Hydrogen	19.22
Oxygen	31.78
Sulphur	3.82
Nitrogen	0.65
Cellulose	63.99
Hemicellulose	6.96
Lignin	2.28
Higher Heating Value (MJ/Kg)	20.85

Temperature's impact on pyrolysis product yields of siam weed (10-30 minutes)

Figures 1-5 graphically demonstrate the product yields acquired from the pyrolysis of broom weed, carried out for 10 to 30 minutes at 5-minute intervals. Analysis discovered that as the pyrolysis temperature increased from 300°C to 450°C, char yield reduced at the same time as tar and gas yields increased. However, at 500°C, char and tar yields each reduced, with gas yield continuing to rise. This observed decrease in tar yield between 450°C and 500°C is generally attributed to the secondary cracking and response of the liquid fraction volatiles, which similarly increased gas yield and decreased char yield, indicating the completion of the pyrolysis process. This trend of reducing char production with increasing pyrolysis temperature is consistent with findings in the reviewed literature (Okekunle *et al*, 2018; Itabiyi *et al*, 2019; Adewumi *et al*, 2023)

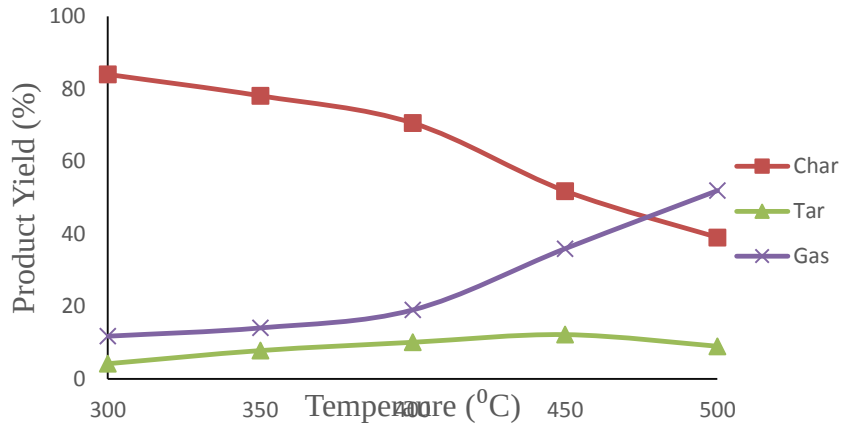

Figure 1: Influence of temperature (°C) on the percentage yields of products obtained from the 10-minute pyrolysis of Siam weed.

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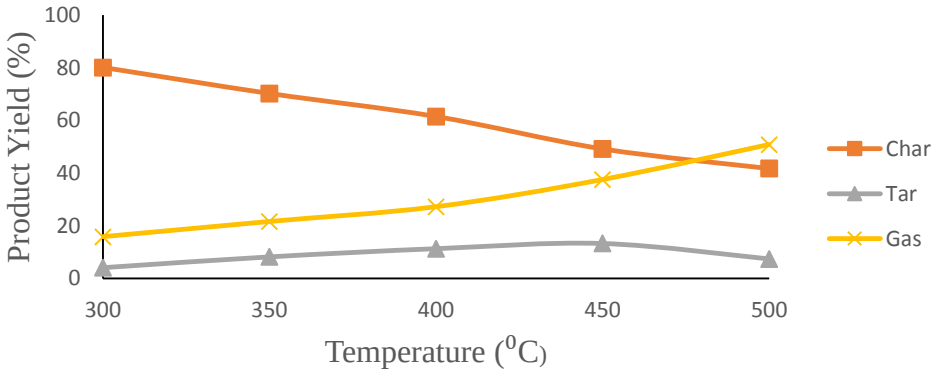

Figure 2: The effect of temperature (⁰ C) on the product yields (%) during the pyrolysis of siam weed for 15 minutes

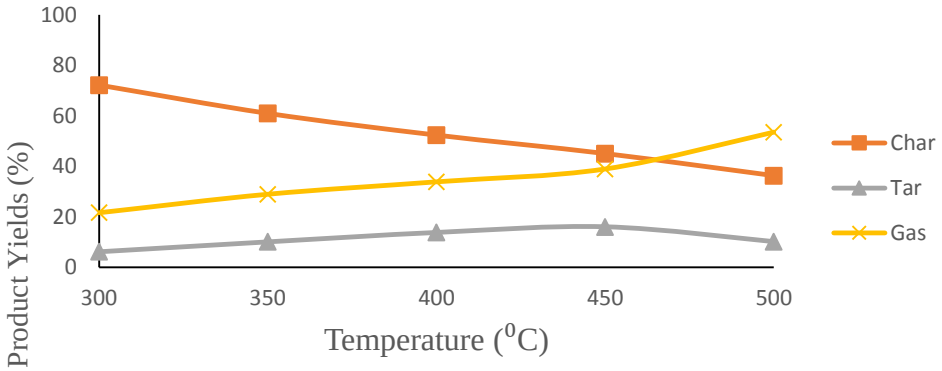

Figure 3: The Effect of temperature (⁰ C) on the product yields (%) during the pyrolysis of siam weed for 20 minutes

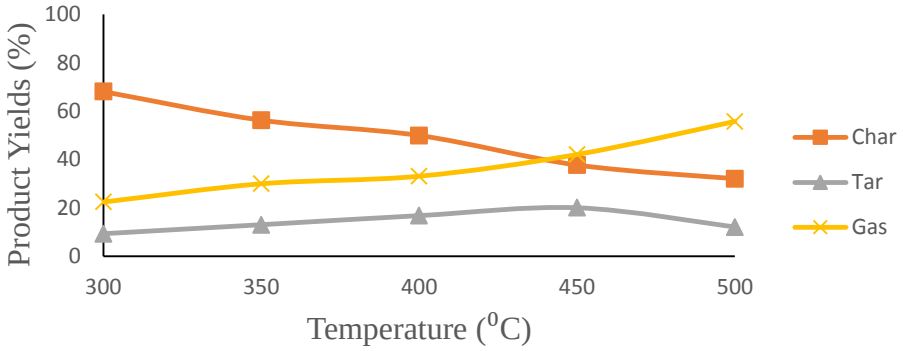

Figure 4: The effect of temperature (⁰ C) on the product yields (%) during the pyrolysis of siam weed for 25 minutes.

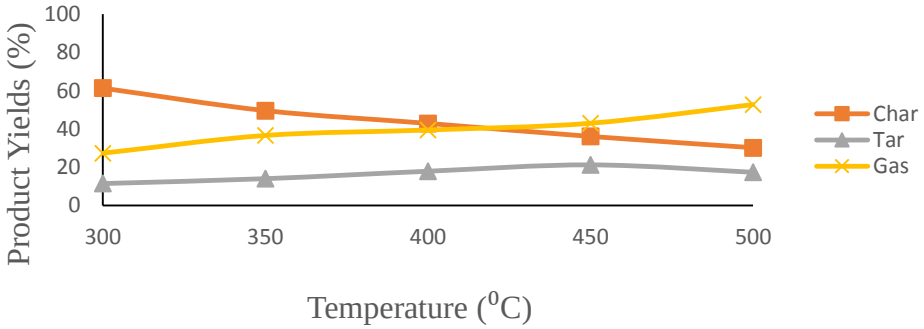

Figure 5: The effect of temperature (⁰ C) on the product yields (%) during the pyrolysis of siam weed for 30 minutes.

Conclusions

The production of biofuel through the pyrolysis of Siam weed has been thoroughly investigated, highlighting its potential as an alternative energy source. Experimental results indicate that the yield of pyrolysis products is strongly dependent on the operating temperature, with variations in temperature significantly affecting the distribution and quantity of the resulting bio-oil, char, and gas fractions. These findings suggest that careful control of pyrolysis conditions is essential to optimize fuel production. Furthermore, the study demonstrates that Siam weed, traditionally considered an invasive plant and environmental nuisance, can serve as a valuable feedstock for generating combustible gases. This gas can be utilized for both residential and commercial energy applications, providing a sustainable alternative to

conventional fossil fuels while mitigating ecological hazards associated with its uncontrolled growth.

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