Application of Torrefaction for Production of Quality Briquette from Sugar cane leaves and Top Composite

Eliyas Manaye

A Thesis Submitted to the Department of Applied Chemistry

School of Applied Natural Science

Presented in Partial Fulfillment of the Requirements for the Degree of Master of Science in Applied Chemistry (Industrial Chemistry)

Office of Graduate Studies

Adama Science and Technology University

March, 2023

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DECLARATION

I, hereby declare that this Master Thesis entitled **"Application of Torrefaction for Production of Quality Briquette from Sugar cane leaves and Top Composite"** is my original work. That is, it has not been submitted for the award of any academic degree, diploma or certificate in any other university. All sources of materials that are used for this thesis have been duly acknowledged through citation.

_____	_____	_____
Name of Student	Signature	Date

RECOMMENDATION

I, the advisor of this thesis, hereby certify that I have read the revised version of the thesis entitled **"Application of Torrefaction for Production of Quality Briquette from Sugar cane leaves and Top Composite"** prepared under my guidance by **Eliyas Manaye** submitted in partial fulfillment of the requirements for the degree of Master of Science in Applied Chemistry (Industrial Chemistry).

Therefore, I recommend the submission of revised version of the thesis to the department following the applicable procedures.

_____ _____

Major Advisor Signature Date

APPROVAL SHEET

I, the advisor of the thesis entitled "**Application of Torrefaction for Production of Quality Briquette from Sugar cane leaves and Top Composite**" and developed by **Eliyas Manaye**; here by certify that the recommendation and suggestions made by the board of examiners are appropriately incorporated into the final version of the thesis.

Eshetu Bekele (PhD)

Major Supervisor	Signature	Date

We, the undersigned, members of the Board of Examiners of the thesis by **Eliyas Manaye Mekonnen** have read and evaluated the thesis entitled **"Application of Torrefaction for Production of Quality Briquette from Sugar cane leaves and Top Composite"** and examined the candidate during the open defense. This is, therefore, to certify that the thesis is accepted for partial fulfillment of the requirement of the degree of Master of Science in Applied Chemistry (Specialization in Industrial Chemistry).

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Finally, approval and acceptance of the thesis are contingent upon submission of its final copy to the Office of Postgraduate Studies (OPGS) through the candidate's Department Graduate Council (DGC) and School Graduate Committee (SGC).

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School Dean	Signature	Date
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ACKNOWLEDGEMENTS

Above all, I thank Almighty God for always being with me in all my endeavors and giving me the endurance to complete my study. Second would to like to express my sincere and heartfelt gratitude to my advisor, Dr. Eshetu Bekele (PHD) for his very useful comments, guidance, willingness to supervise my research, support, and professional advice from the inception and completion of the thesis. It is my greatest pleasure to thank: ASTU for providing this chance; Department of Applied chemistry and members of the staff for their cooperation in facilitating class study. Also, I want to express my deepest thanks to Ethiopian sugar corporation research center laboratory service workers Mr. Tilahun Zeleke laboratory service head, Mr. ,Fiseha Tarekegn Laboratory chemist II ,Mr. Dula Debela Mr Tesfaye Nemera for there technical support. Finally, I would like to forward my heartfelt appreciation and respect for my friends Mr. Bezunhe seifu who face all the challenges up and downs with me directly to accomplish this thesis work.

I went to express my special thanks and appreciation to Mr. Amenu Assefa (Director of sugar Academy director), Mr. Wondemu W/giorgise there encouragement and moral support, patience have made this work possible. Lastly but not least , I would like thank My wife W/ro Sadiya Haji for her understanding , love, patience and shouldering through this long trip.

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LIST OF ABREVIATIONS

CV	Calorific value
C/O	Carbon to oxygen ratio
ρ br	Density of Briquette
FAO	Food and Agricultural organization
g/ cm3	Gram per centimeter
GHG	Greenhouse gas
GCV	Gross Calorific Value
GWH	Giga watt hour
HHV	Higher heating value
H/O	Hydrogen to oxygen ratio
Kg/cm2	Kilogram per centimeter square
Kg/h	Kilogram per hour
KJ/ kg	Kilo joule per kilogram
L: D	Length to diameter ratio
LHV	Lower heating value
ML	Mass loss
MJ/kg	Mega joule per kilogram
MPa	Mega Pascal
Mol/liter	Mole per liter
PI	Porosity Index
W/W	Weight by Weight

ABSTRACT

Every year millions of tons of agricultural residual biomass are generated worldwide which are either destroyed or burnt inefficiently in loose form causing air pollution. Among those residues sugar cane biomass, especially the dry leaves and tops of the sugarcane produced during harvesting of sugar cane is the major one. On other hand, there is high consumption of fire wood and charcoal that is causing the degradation of the limited forest resource. Therefore, the objectives of this study was to recover sugar cane dry leaves and tops composite as alternative source of energy by improving its characteristics through proper pretreatment technique like torrefaction and converting biomass waste into high density fuel briquettes. Sugarcane dry leaves and top composite with the ratio(60:40) in dry weight bases were Torrefied at a temperature (230^{o}C,260 ^{o}C, and 290^{o}C),curing time (30 & 60 min) and particle size (2 mm,4 mm ,and 6 mm). Briquetting of selected torrefied sample were done at briquetting pressure of (8, 10, and 12Mpa), die length (40, 50, and 60mm) and holding time (3, 5, and 7min) to elevate its character as a solid fuel. The produced briquette was then checked for bulk density, shattering resistance and HHV values to assess its quality. The optimum pretreatment value that yield maximum calorific value of 20.03 MJ/Kg compared to the raw composite (17.07MJ/kg) was found to be particle size 2mm and torrefaction temperature of 290 ^{o}C and dwelling time of 60 minute. The optimum briquetting process variables were 50 mm die length, a pressure 12 Mpa and a dwelling time of 7 min. The produced briquettes had bulk density (0.89g/cm^{3}), scattering resistance (99.83%), and HHV (22.04 MJ/Kg). Therefore, from the obtained result, thermal pretreatment /torrefaction/ of sugarcane dry leaves and top composite with composition (60:40 in dry weight bases) significantly improve the energy values and properties of leaves and tops that lead to the formation of quality briquette that can be used as alternatives energy resource.

Key words Sugarcane biomass, Sugarcane dry leaves, Sugarcane top, Briquette, Torrefaction

1. INTRODUCTION

1.1. Background of the Study

In the past decades, fossil fuels appear as the main energy source in the world. However, due to depletion of stocks of fossil fuels, the increase in oil prices, and growing concerns over global climate change have forced policy makers and researchers to investigate comparably greener (renewable) alternatives of energy and technology for the production of alternative energy sources (Rencoret et al., 2017, Höök & Tang, 2013). Increasing the use of renewable energy sources for energy generation purposes is of particular interest nowadays because they allow mitigation of greenhouse gases, provide means of energy independence and may even offer new employment possibilities (Nag, 2007). Moreover, renewable energy is now capturing a good share of the worldwide headlines because of concerns about declining supplies of fossil fuels, escalating population and industrialization triggering ever-increasing demand of fuel (Tursi, 2019).

Among renewable energy sources , biomass is the most abundant and renewable material in the world for the production of bio-fuels, which can be used as a fuel resource alternative to fossil resources (Cai et al., 2017). It is developed from agricultural residues, crop residue, animal manure, agro industrial and food processing residue, municipal solid wastes and other biological resources. It is the fourth highest primary energy source in the world after oil, coal and gas contributing 14% of the total global primary energy supply (Gebrehiwot et al., 2019).

In developing countries, it is the major energy source for cooking and heating. For instance, the contribution of biomass to total energy consumption is about 70% in sub-Saharan Africa (Hailu, A. D., & Kumsa, D. K. (2021). Ethiopia has also huge renewable energy resource potential which includes biomass, hydro power, wind, solar, and geothermal energy. A total biomass fuel supply in Ethiopia is about 1.2 billion tons per year which includes 1.14 million tons per year of woody biomass, 22.8 million tons per year agricultural residues and dung 33 million tons per year (Geissler et al., 2013). Agricultural and agro-industrial residues constitute averagely 15-20% of the total biomass energy consumed (Aragaw, 2016). However, except current energy coverage from woody biomass (50%), agricultural residue (30%), hydropower (5%), wind (3%) and geothermal (1%) which are exploited, the available potential

is not developed (Asresu, 2017). This is primarily due to technological constraints, which require prior attention for further development.

Energy in biomass can be converted by a number of routes: Biochemical and thermo chemical conversion route (Tursi, 2019). The biochemical transformation relies on the processes of anaerobic digestion, fermentation, distillation and hydrolysis, in which the feed stock biomass molecules are broken into smaller molecules through the action of bacteria or enzymes under controlled process parameters like concentration, temperature, pH .Where as in thermo chemical conversion heat is used to promote chemical transformations of biomass for higher-quality pyrolysis, combustion and gasification products. In which the combustion product is widely used because of ease of change is simple and low cost (C. L. Mendoza Martinez et al., 2021).

Agricultural residues for energy production have a very insignificant threat to food security; hence, they could be one of the most reliable bio-energy resources (Mohammed et al., 2014). The residues are classified into crop residues (materials left on the farm after harvest) and process residues (materials left on industrial sites after processing). The crop residues include straw, leaves and stalk of cereals such as rice, maize/corn, sorghum, and millet, cassava stalk/peelings and cocoa pods. The process residues include corn cob, cocoa husk, coconut shell and husk, rice husk, oil seed cakes, sugar cane bagasse, and empty fruit bunch of oil palm (Yusuf Kpalo & Faiz Zainuddin, 2020). Residues of the sugarcane industry are particularly important considering that sugarcane crop is extensively cultivated in several tropical area of the world. It generate huge amount of residual biomass such as green leaves (fresh leaves), dry leaves (brown leaves) and tops. The average sugarcane trash comprised of dry leaves or straws (~68 %w/w), which either naturally falls off as it withers or is removed from the stalk together with the green leaves(~18 %w/w) during harvest and the sugarcane tops (20 %w/w) (Go & Conag, 2019). Sugarcane trash is the field residue remaining after harvesting green leaves (fresh leaves), dry leaves (brown leaves) and tops, worldwide bio-energy potential of sugarcane trash is around 9,475GW/year (Solangi et al., 2018).The sugarcane trash is frequently burned in the open fields during harvest and emitting greenhouse gases and creating the environmental issue (Patil & Bavda, 2017). In Ethiopia Since the inception of the first sugar estate in 1954, pre-harvest cane burning has been one of the

adopted practices. It was intended to get rid of cane trashes and thereby increase harvest efficiency but burning pollutes the surrounding village with smoke, ash and cause chronic respiratory problem , in addition it deteriorate soil quality and decline sugar recovery. The emissions contribute to the greenhouse effect and global warming (Dengia & Lantinga, 2018). On the other hand the residual biomass can provide opportunity for the production of alternative dry fuel such as briquettes through densification process. Briquetting is the physical transformation of loose raw organic materials into high density fuel briquettes through a compacting process which increases the calorific value and combustion efficiency of the product (Asresu, 2017).

However, biomass has its drawback to be utilized particularly as a direct feed stock for power generation. These include low combustion efficiency attributable to its high moisture content, low energy density, hydrophilic behavior, high oxygen content which makes it susceptible to biological attack and biodegradation (Toscano et al., 2015). To overcome these undesirable properties different pre-treatment processes have been introduced in order to improve biomass properties. One of the pre-treatment processes in the conversion of biomass to energy is torrefaction. Torrefaction is a thermo-chemical conversion method where biomass is subjected to thermal heating in the absence of air; typically in the temperature range of 200 to 300^{o}C at atmospheric pressure to improve its properties and reduce the emission of gas during combustion (Matali et al., 2016). But to achieve those properties the torrefaction process parameters of the biomass such as particle size ,temperature ,and residence time should be optimum to get good product with low cost. According to Mamvura the torrefaction conditions are reaction temperature 200 to 300^{0}C, heating rate <50^{0}C/min , residence time ≤60 min (Mamvura & Danha, 2020), particle size ≤ 2mm (Jaya Shankar Tumuluru, 2016 , Trubetskaya et al., 2020).The principle of this process rests on the reduction of oxygen (O/C) to carbon and hydrogen to carbon (H/C) ratio, results increases elemental carbon in torrefied biomass leading to an increase in calorific value (Basu, 2013) and make final solid product hydrophobic, which facilitates storage conditions, improved grindability and homogeneity, make torrefied biomass more attractive as a fuel when compared to non-heat-treated biomass (Nunes et al., 2020).

Using biomass as its original form has been known for century. However handling, stability and transportation of an enormous quantity of biomass is energy and labor-intensive, which is

one of the major financial factors impeding the use of biomass for sustainable energy and heat generation (Ibitoye et al., 2021). Densification of biomass into solid fuels makes the biomass uniform in size and shape for stress free handling (OYELARAN et al., 2018, Jiang et al., 2016). This makes it fit for use in thermal conversion processes, for example, gasification, co-firing with coal, combustion, and pyrolysis (Bazargan et al., 2014). It involves the mixing of feed stock particles and the application of pressure. The common densification processes are briquetting, pelletizing, bailing, and cubing (Akogun & Waheed, 2019). Briquetting is the process of compacting homogenous or non-homogenous loose combustible materials into a product of higher density for fuel-making purposes (Supatata et al., 2013). The operating parameters considered during briquetting include pressure, residence time, and temperature, while the feed stock parameters include moisture content, particle shape size, and external additives these parameters can be optimized so that briquettes of good quality can be produced (Ibitoye et al., 2021).

The optimum briquetting temperature and pressure range from 100 to 250°C and 50–250 Mpa, respectively, while the optimum residence time is between 4 and 25 min (Ahiduzzaman & Islam, 2013). An optimum proportion of binder/adhesive range of 5–25% is recommended to produce high-quality briquettes (Espuelas et al., 2020, Ajimotokan et al., 2019).Successful and effective briquetting required feed stock with moisture content ranges of 5–15% and particle size ranges of 1–10 mm (Mopoung & Udeye, 2017).

In this study, dry sugarcane leaves and tops as one of agricultural residue were subjected to torrefaction for better grind ability, less moisture uptake, higher C/O ratio for the production of quality briquette at optimized condition as alternative energy source for domestic and industrial usage.

1.2. Statement of the Problem

In Ethiopia the amount of land covered by sugar cane crop is around 78,238 hectare both in private and government. From this amount Wonji sugar estate owns 11,000 hectare. The total amount of cane crushed in 2021 is 522,590.6 ton. The quantity of sugar cane trash burnt in open field estimated around 7,838.94 ton by assuming that 15% trash is obtained per ton of cane (Zafar, 2015). Utilizing this residual biomass for compact energy is an opportunity to minimize the consumption and cost of fire wood and charcoal.

On the other hand, the existing practice of harvesting sugar cane in Ethiopia, which is made by burning valuable dry leaves and top cane biomass to openly to separate the stalk from the residual biomass causing respiratory problem to people live in the sugar estate, increase emission of GHG globally. Moreover, the conventional practice reduce the amount of sucrose in the stalk in line with the time which the cane reach to the factory greater than eight hour, make the amount of trash reach to the factory cane table large, and decrease water percolation to the soil because of accumulation of ash at the bottom of the soil structure, this attributed for a decline in yield (Carvalho et al., 2017). Therefore, utilizing the excess leave as well as top cane as feed stalk for domestic energy application by making briquettes alleviate the problem of continuous destruction of forest cover and health risk resulting from exposure to emissions. Beside to decreasing the amount of GHG released to the atmosphere. However direct use as solid bio-fuel hindered by its high moisture, heterogeneous shape, and size. Hence, pre-treatment like torrefaction followed by briquetting to convert biomass into compatible energy fuel would be a solution to the observed problem. Hence, in this study, the agricultural residue of dry sugarcane leaves and tops will be subjected to torrefaction for better grind ability, less moisture uptake, higher C/O ratio followed by briquetting to investigate the energy value for its potential application for domestic and industrial usage.

1.3. Objectives

1.3.1. General objective

The main objective of this study is process optimization of Torrefaction for the production of quality Briquetting from sugar cane leaves and top composite at optimized condition.

1.3.2. Specific objectives

- To analyze the chemical composition of the dry leaves and tops of sugar cane biomass.
- To optimize the torrefaction temperature, particle size and residence time for better energy and physicochemical values of sugar cane leave & top composite.
- Produce briquette of the required properties from torrefied sugar cane leave & top composite by varying densification pressure, holding time, and length of the die.
- Characterize the briquette produced at optimum operation conditions with respect to bulk density, scattering resistance, and HHV of briquette.

1.3.3. Significance of the research

Finding of this research offers knowledge and understanding of the performance briquette obtained from torrefied sugar cane leave & top cane. This study should be significant in the sense of:

- Reduce environmental and socioeconomic problems.
- Show how to use sugar cane leave & top cane to produce briquette in small scale which will reduce our dependence on wood fire based fuels and charcoal.
- Be used as base-line information for further studies particularly for small scale production of biomass that will meet international standards.
- It is become additional source of income for the factory.
- Provides an alternative energy as a substitute for dry wood and wood charcoal in house hold cooking.
- Job creation and improve income of the people.

1.4. Scope of the Study

In this research the sugarcane residual biomass dry sugar cane dry leaves and tops sundry. Grinding of sugar cane dry leaves and top is made by mill .The particle size of the dry sugar cane leaves and top going to optimize limited to 2, 4 and 6mm.The treatment for the torrefaction is limited to a temperature of 230, 260 and 290^0C. The mould that used to produce briquette will be a inner diameter of 50 mm with a height 40, 50 and 60mm and the compaction of torrefied biomass is done by hydraulic press using 8,10 and 12 MPA with holding time of 3 ,5 and 7 minute. This work is not intended to produce briquettes in large amount for sell and use, rather it is to produce sample briquettes characterize their thermal, fuel and physical properties. Finally it intends to recommend industries and enterprises how to produce briquettes.

2. LITERATURE REVIEW

2.1. Biomass as Alternative Energy Source

Any organic materials, living or that has been dead for a short period of time, derived from plants or animals are classified as biomass (Mamvura & Danha, 2020). Search for new alternatives for power generation has been intensified in the scientific community (Zanella et al., 2016) In plants, biomass is formed through conversion of carbon dioxide in the atmosphere into carbohydrates in the presence of the sun's energy. Biological species will then grow by consuming these botanical or other biological species adding to the biomass chain (Basu, 2013). In the presence of light from the visible spectrum particularly blue range (425–450 nm) and red range (600–700 nm), green plants breakdown water to obtain electrons and protons and use them to turn carbon dioxide into glucose and release oxygen as a waste product through a process called photosynthesis (Pessarakli, 2005).

$$6CO_2\,(g) + 6H_2O\,(g) \xrightarrow{\text{Sunlight}} C_6H_{12}O_6\,(g) + 6O_2\,(g)$$

Biomass can be determined from different sources such as wood, energy crops, forest and agricultural residue, industrial and municipal wastes. Plant biomass has a major part known as lignocellulose, consisting of three main polymeric components: hemicellulose, cellulose, and lignin and they are usually termed lignocellulosic based biomass. In general, lignocellulosic-based biomass is widely used for renewable energy applications (Mamvura & Danha, 2020).

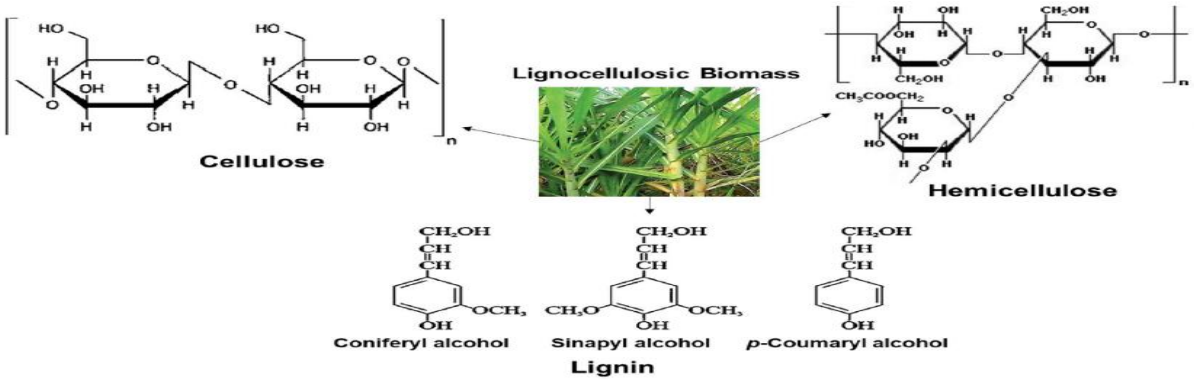

Figure 1: Chemical structure of lignin, cellulose and hemicelluloses (Khaire et al., 2021).

Lignocellulosic biomass can also be derived as a by-product from food crops e.g. agricultural residue, and grasses. At present, these residues are combusted directly without optimization of energy efficiency or control of air emissions, or they are left on farm land/processing sites (Engineering, 2019)**.** The residues generated from industrial production chains not only represent unused potential, but land-filling or burning can have significant negative environmental impacts (e.g., emission of large quantities of volatile organic compounds in the case of combustion, and contamination of ground-water in the case of landfill). The unused residues treatments often generates high costs that producers want to avoid (C. L. Mendoza Martinez et al., 2021).

2.2. Sugar Cane Biomass

Sugarcane (Saccharum officinarum) is cultivated in tropical and subtropical regions and it is the most produced agricultural commodity worldwide. It is primarily used for the production of sugar and ethanol, with the latter being mostly used to yield alcoholic beverages and low carbon bio-fuels (Formann et al., 2020). Sugarcane produces mainly two types of biomasses, namely sugarcane trash and bagasse. Sugarcane trash is green leaves (fresh leaves), dry leaves (brown leaves) and tops is an excellent biomass resource, as it is being used as a raw material for energy production (Zafar, 2015). The sugarcane trash quantity can be evaluated by measuring the sugarcane trash produced per unit tone of the sugarcane crop. The production of sugarcane trash depends on the plant variety, crop age; type of soil and weather conditions of the area. It is reported that the average sugarcane trash (green leaves, brown leaves and tops) per yield (ton) of sugarcane crop is around 13% and 15% (Solangi et al., 2018).

In Ethiopia the amount of sugar cane biomass before and after burning of the sugar cane crop is estimated 33% and 2.5% from 120 ton of cane respectively. This fraction is lower than reported in 37-44% for Mauritian, Hawaiian and South African plantations. However, it is in agreement with Stewart who found 13-33% for an Australian plantation (Dengia & Lantinga, 2018).

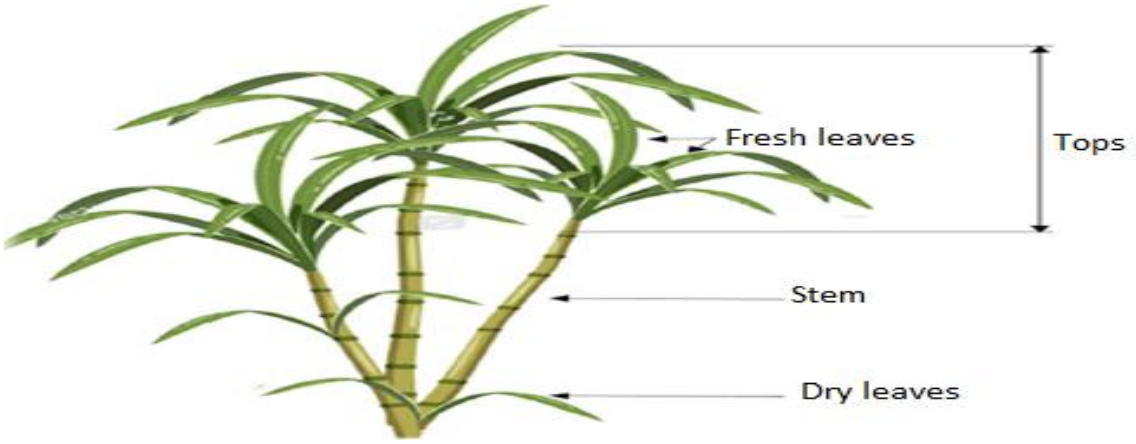

Figure 2: sugar plant morphology (Khaire et al., 2021).

2.2.1. Sugarcane harvesting

The production systems for sugarcane include either green cane or burnt cane harvesting operations. In burnt cane harvesting, sugarcane fields are set on fire in order to burn off leafy material before harvesting. In green cane harvesting, sugarcane is harvested without burning, and a thick leafy residue (commonly called "trash blanket" or trash) remains on the soil surface (Sandhu et al., 2015). Increasing concern with environmental issues has led to changes in legislation and harvesting practices, as the burning of sugarcane causes significant emissions of particulate matter and polyaromatic hydrocarbons. A further problem associated with pre-harvest burning is that the sugar content of the harvested stalks is reduced by the high temperatures reached when the sugar cane is burnt (Carvalho et al., 2017). Many sugar cane producing countries have decided to stop burning at harvest and implemented green harvesting. This has also been pushed by the growing demand of cane trashes to use for field blanketing or today for energy use and bio-plastics. However in some countries in Africa, Asia and South America, cane burning is still employed and has a devastating effect on biodiversity (Goebel & Nikpay, 2017).

Figure 3: Burning of sugar cane trash(Goebel & Nikpay, 2017).

In some countries, hand cutting of sugarcane is still widely practiced, although this has been completely replaced by mechanical harvesting in many countries. Traditional sugarcane-harvesting processes cut the stalk around ground level and discard tops and leaf materials. Only the clean stalk (either as a whole stalk or cut into billets) is transported into the factory for the extraction of the juice and production of sugar (Hara, 2016).

Figure 4: Mechanized harvesting system (Mokhena et al., 2016)

Mechanized harvesting has increased gradually in recent years and currently accounts for around 89% of sugarcane harvested in the state of Sao Paulo (Carvalho et al., 2017). In Ethiopia since the inception of the first sugar estate in 1954, pre-harvest cane burning has been one of the adopted practices. It reduces injury to workers from sharp foliage, insects and snakes, and improves economic returns. However burning pollutes the surrounding village with smoke and ash particulate matter emissions and severe respiratory diseases in the area

(Dengia & Lantinga, 2018). The gases (CO_2, NO, NO_2 and N_2O) emitted to the atmosphere during burning contribute to the greenhouse effect and global warming (Formann et al., 2020). Woody biomass from natural forests supplies the highest proportion of biomass energy in Ethiopia. Total national consumption of wood (including charcoal equivalent of wood) is estimated to be 105.2 million tons per year with 5.7 million tons of charcoal which covers more than 95% of biomass consumption in the country, this impose pressure on the limited biomass and forest stock of the country (Asresu, 2017). Therefore, converting the residual biomass from sugar industries to solid fuel like briquette rather than burning could contribute towards clean energy supply, waste management and reducing deforestation.

2.2.2. Composition of sugarcane residues (Top, Dry leaves and Straw)

The approximate composition of the sugarcane in nature is the following: Stem and green leaves: 8%, Sheath and dry leaves: 20% and Clean Stalk: 72%. Sugarcane straw refers to the dry leaves and sheaths removed from stems or stalks during the cleaning process. The amount of silica contained in the straw is two or three times as much as the amount found in the green leaves. Consequently, the amount of lignin is approximately 60% smaller than those found in the green leaves (Gómez et al., 2014).The trash composed by dry leaves (60%) and green tops (40%) (Aguiar et al., 2021).

Table 1: Chemical composition of sugar cane residue

Feed Stock Material	Cellulose %	Hemi Cellulose%	Lignin %	Ash %	Extractives	Reference
Sugarcane Dry Leaves	27.64	19.15	11.95	-	-	(Patil & Bavda, 2017)
Sugarcane Top (w/w	35.2	37.7	8.1	-	-	(Aguiar et al., 2021)
Sheath + Dry Leaves(straw)	44.5	-	12.3	7.5	3.7	(Gómez et al., 2014)
Green leaves	40.5	-	22.8	2.1	2.5	(Sandhu et al., 2015)

Lignocellulosic biomass are different based on their compositions and thus will behave differently in thermal conversion and energy generation. The relative proportions of these

constituents depend on the nature of biomass (Kambo & Dutta, 2015). The relative contents of three constituents in biomass are roughly ranked as cellulose > hemicelluloses > lignin (Bui et al., 2015). Lignin in lignocellulosic biomass acts as a binding substance for hemicelluloses sand cellulose structures (Acharya et al., 2015).

2.2.3. Major properties of sugar cane dry leaves and tops

The breakdown of the compositional analysis into elemental composition is achieved via ultimate analysis and proximate analysis, which is the summary of the moisture, ash, volatile matters and fixed carbon (FC) contents of the biomass. Carbon, hydrogen, and oxygen have more influence in determining the properties and classification of biomass fuel.

Table 2: Proximate analysis result of sugar cane residue

Material	**Parameter**					**Reference**
	Fixed carbon %	Volatile matter %	Moisture %	Ash %	Heating value$Kgx10^3$	
Dry leaves w/w	11.6	84.5	13.5	3.9	-	(Solangi et al., 2018)
	-	-	13	3.7	17.4	(Vergara et al., 2020)
	16	78.3	12.9	5.7	18.4	(Carvalho et al., 2017)
Tops w/w	16.4	79.3	82.3	4.3	-	(Solangi et al., 2018)

Table 3: Ultimate analysis result of sugar cane residue

Material	**Parameter**					**Reference**
	Carbon	Hydrogen	Nitrogen	Oxygen	Sulfur	
Dry leaves w/w%	46.2	6.2	0.5	43	0.1	(Solangi et al., 2018)
	43.1	6.2	0.35	50.2	0.05	(Carvalho et al., 2017)
Tops w/w%	43.9	6.1	0.8	44	0.1	(Solangi et al., 2018)

According to Axelsson, for green top regardless of sit, cycle or variety, the HHV ranged between 16.3 and 17.9 MJ/Kg while for dry leaves ranged between 15.4 and 18.1 MJ/kg (Menandro et al., 2017). Tops green leaves have high moisture content 66-82% while dry leaves moisture content is around 10-14%. Ten to fifteen days after the harvest the moisture of the green leaves and top in the soil is usually reduced to 15-30% (Carvalho et al., 2017).

2.3. Biomass to Energy Conversion Process

The conversion of biomass to energy can involve different routes according to the biomass characteristics. The type of transformation used, to convert biomass are biochemical, physiochemical and thermo-chemical (C. L. Mendoza Martinez et al., 2021). Biochemical conversion relies on the processes of anaerobic digestion, fermentation, distillation and hydrolysis, in which the feed stock biomass molecules are broken into smaller molecules through the action of bacteria or enzymes (Naik et al., 2010). This type of conversion requires a longer time than the thermo chemical routes, but it can be achieved with a smaller amount of external energy. Physicochemical conversion encompasses the densification of biomass into pellets or briquettes as well as physical extraction of oils by compression (C. L. Mendoza Martinez et al., 2019). The thermo-chemical conversion route involves the use of heat to promote the chemical transformation of biomass into energy and chemical products (C. L. Mendoza Martinez et al., 2021).

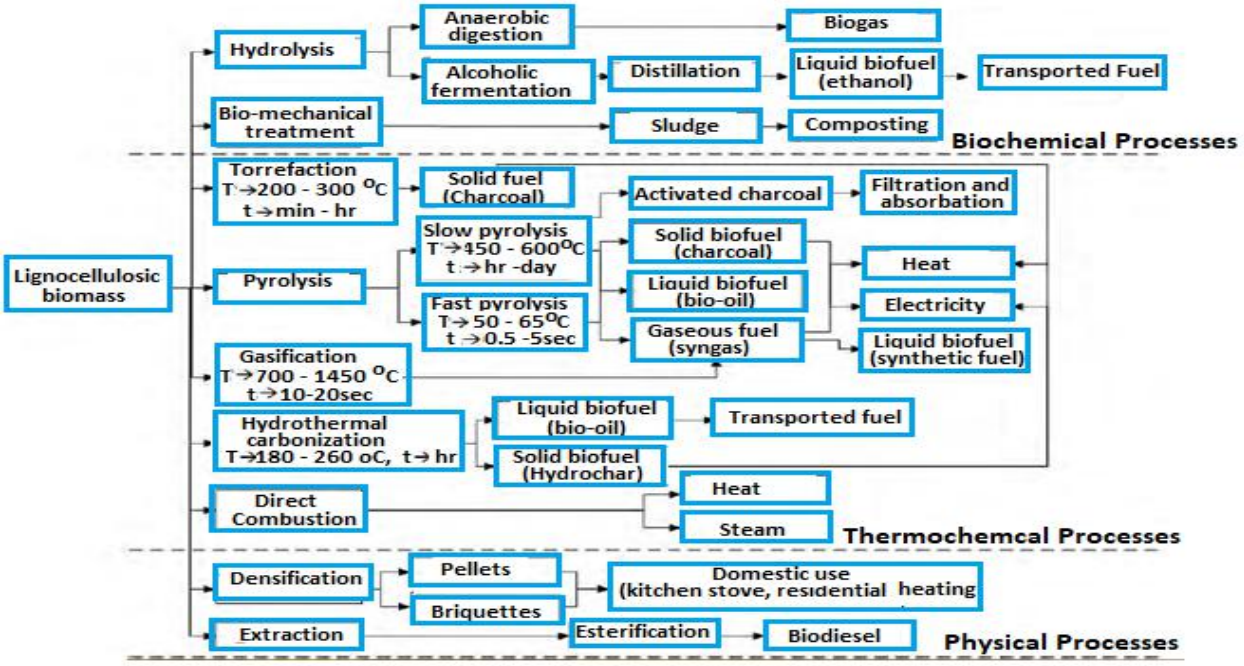

Figure 5: Conversion routes of biomass to energy (C. L. Mendoza Martinez et al., 2021).

2.4. Pre-treatment of Biomass

Pre-treatment helps reduce the specific energy consumption and produce different high-quality densified products for different end user applications. In general pre-treatment improves the quality attributes (higher durability and bulk and energy densities), storage and handling

characteristics, and transportation (J. Tumuluru et al., 2010). Some promising pre-treatment methods for bio-energy applications include drying, grinding, and torrefaction.

2.4.1. Drying

Moisture content of biomass remains one of the major factors that affect the performance of densification processes and that of energy conversion systems because the quality of a densefied material as well as successful operation of densification systems are highly moisture sensitive which preferably should not exceed 15% (Iyer et al., 2022). Successful and effective briquetting required feed stock with moisture content ranges of 5–15% and particle size ranges of 1–10 mm (Mopoung & Udeye, 2017, De Oliveira Maia et al., 2014).

2.4.2. Milling/ Grinding

The total surface area and the pore size of the material including the number of contact points for inter particle bonding increases with particle size reduction during compression. According to many studies, the recommended particle size of biomass used for producing both charcoal based and non-carbonized briquettes ranged below 6 mm (Bazargan et al., 2014, Adeleke et al., 2021). The process of size reduction is energy intensive, and for this reason, it cannot be met through combustion of the material. However, the energy demand for size reduction can be reduced when the material is first torrefied, and the reduction in energy can be as high as 80% (Anukam et al., 2016).

2.4.3. Torrefaction

Torrefaction, is a thermal pretreatment process conducted between the temperatures of 200–300^{O}C in inert (absence of oxygen)atmosphere with the aim of modifying chemical properties of biomass (Matali et al., 2016). This process is described as a mild form of pyrolysis since volatiles are removed resulting in a product with about 80–90% of the original calorific value of the material but with only 70% of the initial weight (Mamvura & Danha, 2020). It makes the material hydrophobic mainly due to the elimination of hydroxyl groups (OH). Non-polar unsaturated compounds that result from the rearrangement reaction process of torrefaction help preserve the biomass by reducing biological degradation which may render it less useful for energy production purposes. More O_2 and H_2 are driven off as compared to C, increasing the calorific value of the material in the process (Anukam et al., 2016).

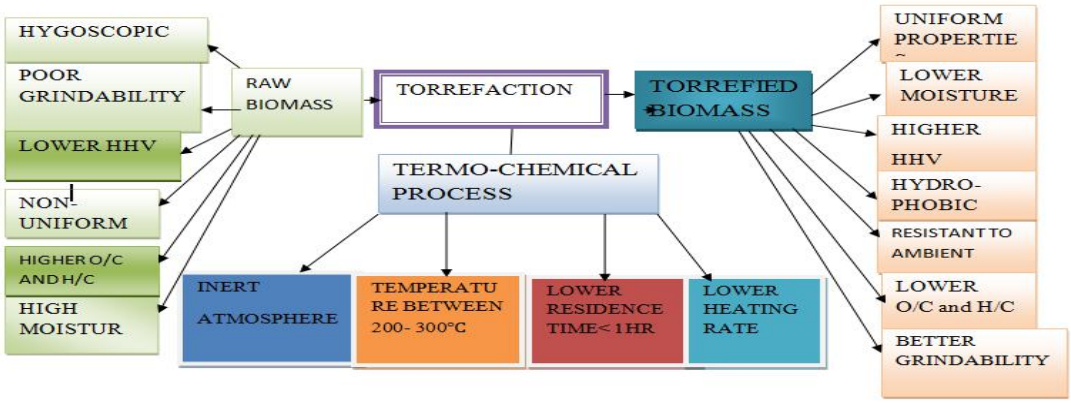

Figure 6: Property variation of biomass undergoing torrefaction(Morales, 2017)

2.4.4. Torrefaction process

Non-oxidative torrefaction is the major method used to pre-treat solid biomass and has a high potential for its application in the industry. For non-oxidative torrefaction, the entire biomass torrefaction process is split into five stages: (1) initial heating; (2) drying; (3) post-drying and intermediate heating; (4) torrefaction; and (5) cooling (Nunes et al., 2020), as illustrated in the figure 8. The five stages are described as follow.

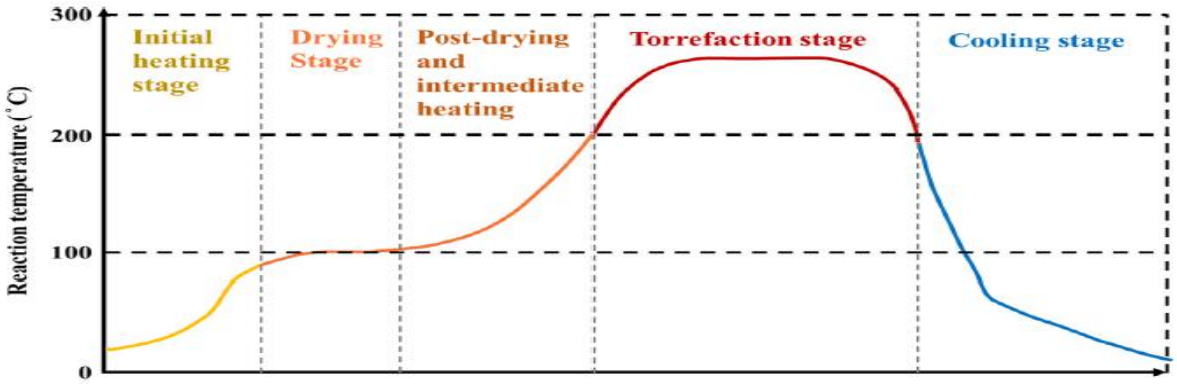

Figure 7: A schematic of different stages during dry torrefaction (Nunes et al., 2020).

2.4.5. Torrefaction process parameters

The performance of torrefaction is affected by some factors such as temperature, duration, and carrier gas flow rate and its composition, particle size, the presence of the catalyst, etc. The influences of these parameters on torrefaction performance are described below (Chen et al., 2021).

2.4.5.1. Temperature

Torrefaction temperature is the most important parameter among the operating parameters. The thermal decomposition temperatures of hemicelluloses and cellulose are in the ranges of 220-315^{o}C and 315- 400^{o}C, respectively, while torrefaction is operated at temperatures of 200-300^{0}C, it is thus known that the operating temperature has a significant influence on the results of torrefaction. Torrefaction can be categorized into light torrefaction (200^{o}C to 235^{o}C), mild torrefaction (235 to 275^{o}C), and severe torrefaction (275 to 300^{o}C), according to torrefaction temperature (Gan et al., 2020, Chen & Kuo, 2011). In slight torrefaction, only hemicellulose is affected, while lignin and cellulose are poorly affected. In the mild torrefaction, the hemicellulose decomposition and volatile release is intensified. The cellulose is also decomposed in some part and the properties of the final biomass begin to improve in comparison with initial properties. In severe torrefaction the hemicellulose is completely decomposed while the cellulose is mostly degraded. Lignin is difficult to thermally degrade so it hardly decomposes a small proportion, achieving a final material with high lignin content which improves its properties as a solid fuel (Chen & Kuo, 2011).

2.4.5.2. Reaction time

Apart from temperature, torrefaction time or duration is an- other important factor in evaluating torrefaction performance and severity. Torrefaction is normally conducted from several minutes to several hours (Gan et al., 2020). The calorific value of resultant solid fuel or biochar is enhanced by torrefaction, and an increase in duration raises the carbon content and heating value in the biomass. For instance, the torrefaction of wood briquettes operated under 250^{0}C for 0.5, 1 and 1.5 hours showed that the calorific values of the biomass increased from 20.0 to 22.7 kJ/kg, while the longer torrefaction duration would take more energy for the thermal pretreatment (Chen et al., 2021).

2.4.5. 3. Particle size

The particle size is one of the important parameters for biomass torrefaction. Biomass has poor heat conductivity, while the temperature gradient across the particle will influence the biomass pyrolysis mechanism (Peng et al., 2012). In general, smaller particles can promote the heat and mass transfer to keep the relatively constant temperature within them during

pyrolysis, thereby enhancing bio-oil production by restraining the char formation and secondary cracking of vapors (Kan et al., 2016).

2.5. Briquetting

The densification/briquetting process is the physical transformation of loose raw organic materials into high density fuel briquettes through a compacting process which increases the calorific value and combustion efficiency of the product (Asresu, 2017). They are made of different qualities and dimensions depending on the raw materials, mold and technologies applied during production (Oladeji, 2015). They are typically cylindrical in shape with a diameter of between 25 and 100 mm and lengths ranging from 10 to 400 mm. Other shapes of briquettes include square, rectangle and polygon and also in different sizes (Yusuf Kpalo & Faiz Zainuddin, 2020). Currently, the high demand for briquettes and pellets is driven, significantly by governmental policies and incentives. The EU as the main consumer, accounted for 80% the global biomass briquette market and reported around 20 million tons of wood pellet production, since the solid fuel is used also in non-industrial environments, such as domestic heating and commercial boilers, on a small scale (C. Mendoza Martinez, 2021).

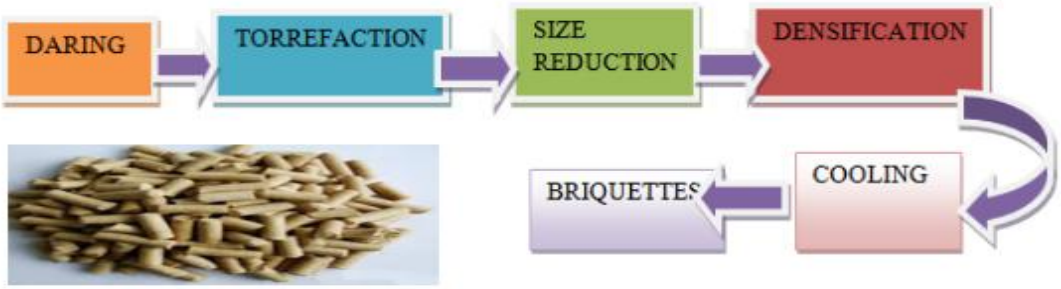

Figure 8: Briquetting process of torrefied biomass (Anukam et al., 2016)

2.5.1. Briquetting technology

The technologies for briquetting are classified according to the method used to compress the material. These include the piston press, the screw extruder and the manual Presses. The piston press densification technologies include the hydraulic piston press, the mechanical piston press and the roller press (Yusuf Kpalo & Faiz Zainuddin, 2020). The following section elucidates the types of briquetting technologies that can be used for the densification of biomass. Their merits and demerits are also described.

Table 4: Basic densification equipment (Adekunle A. Adeleke et al., 2021).

1	Hydraulic piston	The pressure required by the hydraulic press is generated through a designed hydraulic cylinder which when the pressure require is reached, it releases the briquettes. The capacity of this machine is between 50 and press 400 kg/h. It can tolerate high moisture content than the accepted 15%.
2	Screw extrusion	A screw is used in forcing a feed stock into a die under high pressure, which forms large cylinders between 2.5 and 10 cm in diameter. The conical screw press, screw press with a heated die, and the twin screw press are the three types of screw presses.
3	Mechanical piston	The equipment is designed to use a compression force of approximately 2000 kg/cm2 to acquire briquettes with high quality and energy density. It does not use binder and can be on large-scale production of 200-2500 kg/h.
4	Tabletizer	Using hydraulic motor and ram, the machine firmly compact biomass in 4–6-inch diameter cylindrical mold. This reduces the material from 10 to 2 inches, which is smaller than most biomass briquettes.
5	Briquetting roll press	Compaction of crushed materials into lumps by pre-compressing feed stock to fall between two opposite direction rotating rollers. The feed stock is compacted into pillow-shaped briquettes, which is formed after Feed stock compaction.
6	Punch and die	Two opposing pistons are used within a cylinder to compact the biomass in the mold. The end product is pushed out of the mold by a piston in the form of a slug, log or tablet. Relatively wet biomass can be briquetted at high pressure in this machine.
7	Manual presses	Different types of manual presses used for briquetting biomass feed stocks. They are specifically designed for the purpose or adapted from existing implements used for other purposes. Manual clay brick making presses are a good example. They are used both for raw biomass feed stock or charcoal. The main advantages of low-pressure briquetting are low capital costs, low operating costs and low levels of skill required to operate the technology. Low-pressure techniques are particularly suitable for briquetting green plant waste such as coir or bagasse (sugar-cane residue)(Yusuf Kpalo & Faiz Zainuddin, 2020).

2.5.2. Briquetting process variables

Process variables such as pressure, die temperature and die geometry play a major role in densification of biomass. Other process variables that also play major roles include material variables such as moisture content, particle size and shape; and material composition such as cellulose, hemicelluloses and lignin (Shaw, 2008). System variables for densification are important if the desired densities, durability and quality of the densified material are to be achieved, with proper process conditions ensuring improved briquette quality (J. Tumuluru et al., 2010).

2.5.2.1. Pressure

Diffusion of molecules from one particle to another at contact points may form solid bridges due to the application of high temperatures and pressure during briquetting, leading to arise in briquettes density. The optimum pressures that have been used for producing non-carbonized briquettes ranged from 50 Mpa to 250 Mpa for different feed stock characteristics. The optimum compression time ranged between 4 and 25 minutes (Bazargan et al., 2014). However, studies have shown that low compaction pressure can produce low-cost briquettes that are durable (Yank et al., 2016,Lubwama & Yiga, 2017).

2.5.2.2. Die geometry

The geometry (shape and size)of the die greatly affect both the amount of material that can be densified and the energy required for densification, and influences material properties such as bulk density and durability as well as moisture content ,with degree of compression determined by die length to diameter (L:D)ratio during densification (Anukam et al., 2016). An increase in pellet/briquette die length increases the required pressure for densification, whereas die diameter increases in lower compacting pressure. However, larger die diameter of 7.2 mm produced less durable product compared to smaller die of 6.4 mm for drilled distiller grains pellet (Adekunle A. Adeleke et al., 2021).

2.5.2.3. Temperature

Control of temperature during briquette production enhances production efficiency and improves the durability and strength of the final briquette (Asamoah et al., 2016). According to many studies on the use of different feed stock, optimum temperatures during the densification of non-carbonized briquettes ranged between 100 to 250^{O}C (Ahiduzzaman & Islam, 2013). Low temperature will result in higher pressure and power consumption. It also leads to lower production rate, but higher-quality briquettes (Yusuf Kpalo & Faiz Zainuddin, 2020).

2.5.2.4. Holding time

Briquette quality is also greatly affected by the time interval between the point of feeding in to the densification system and the time required for compaction. The effect on density of biomass briquettes could significantly be reduced at retention times longer than 20 seconds (Anukam et al., 2016).

3. MATERIAL AND METHOD

3.1. Description of Study Area

The samples used in this experiment were collected from wonji/shoa sugarcane estate, which was located 8.38^{o}N 39.30^{o}E, in East shoa zone of Oromia National regional state at, 10 Km away from Adama , and 110 Km East of Addis Ababa, Ethiopia. Its altitude, lies between 1223 and 1553 m above sea level. The area has a semi-arid climate with a ten year mean monthly rainfall of 70mm, and mean monthly minimum and maximum temperature is 27^{o}C, and 12^{o}C respectively. The area is dominantly used for sugarcane cultivation and the total area is covered by sugarcane is 10,000 hectare. The soil is predominantly Andsols, Fluvisols, Laptosols and Phaezemes, according to the FAO soil classification.

3.2. Instruments used in the study

The instruments used includes, Mill grinder (Sr.N. 129070704D, Germany) size reduction of the sugarcane tops and dry leave samples; Furnace (model: 683, Great Britain) for analysis of ash, volatile matter and torrefaction process; memmert oven (models 30-1060, Germany) for drying of samples and subsequent determination of moisture content; Hydraulic press (model HAN TECH,COREA) for the production of briquettes; FTIR (model: SDTQ600 PerkinElmer) to identify the functional groups of raw and torrefied samples, TGA/DTA (C30575100456TK series) for analyzing the mass change, thermal stability and Composition of biomass; Bomb calorimeter (IKA- WERKE C5000 control, Germany) to determine the heating value of the biomass; Thermo deny Horizontal shaker (se.N.321,AS 200,Germany) to separate under and over size crushed biomass; Condenser and Water bath (Sr.N.106544116,Germany) to reflux sample and heat treatments during extractive, hemicellulose, cellulose material determination; HANNA PH meter (model PH 211,Romania) ; Electronic analytical balance (model:WB1160078,kern & sohn Gmbny, Germany) to check the neutrality of the filtrate during cellulose and hemicellulose determination Electronic digital caliper (model 3510106820) to measure the height and diameter of briquette).

3.3. Chemicals Used

Analytical grade Alcohol (AR, Assay 96%) and Sulfuric acid (AR, Assay 98%), the two chemicals used for this experiment.

3.4. Characterization of Sugar cane leaves and Top

The Samples of crop residues (Sugarcane dry leaves and tops) used for this experiment were collected by polyethylene bag. A portion of samples were taken for moisture analysis, and the rest were sun dried grounded by mill and sieves to obtain 2, 4 and 6 mm particle size for torrefaction and briquetting.

Figure 9: Sample of dry and top sugar cane collected

3.4.1. Compositional analysis of sugarcane dry leafs and top

The percentage of lignocellulosic fractions (cellulose, hemicelluloses, lignin, extractive and ash) of the sugar cane residue were determined gravimetrically by the method proposed by (Chesson, 2013). Based on this method, one gram of previously dried dry and top sugar cane leafs sample (a) was added with 150 ml alcohol for each (1 gm dry and l gm top) and refluxed at 100^{o}C in water bath for 1hr. After 1hr the solution was filtered, and the residue washed with 300 ml hot distilled water. Then it was dried until constant weight in an oven and weighted (b). To the weighted residue 150 ml 1N H_2SO_4 was added and refluxed in a water bath at a temperature of 100^{o}C for 1hr. The mixture will be filter and the solid residue is washed by using deionized water until neutral pH and then the sample was dried using an oven at 105^{0}C and weighted (C).The dried residue was soaked by 10 ml of 72% H_2SO_4 for 4hr. After 4hr the solution was refluxed by adding 150 ml 1NH_2SO_4 on a water bath for 1hr.Then the residue was filtered and washed with distilled water until the pH become neutral and it was dried at 105^{o}C, and weighted (d). Finally the weighted mass was ashed in a furnace (e). The compositions were computed using the following formula:

$$\%\ lignin\ content = \left[\frac{d-e}{a}\right] * 100 \qquad (1)$$

$$\% \, cellulose \; content = \left[\frac{c-d}{a}\right] * 100 \qquad (2)$$

$$\% \, Hemicellulose \; content = \left[\frac{b-c}{a}\right] * 100 \qquad (3)$$

3.4.2. Proximate analysis

The proximate analysis of a sample provides the content in mass percentage of moisture, ash, volatile materials, and fixed carbon, contributing to the understanding of the material behavior when subjected to a thermal conversion process. The proximate analyses were carried out according to the procedure mentioned in Silveira (Silveira et al., 2020).

3.4.2.1 Moisture content

To determine the moisture content (% MC) of raw dry and top, 5g samples was dried at a temperature around 105°C, until constant mass. The moisture content (% MC) represents the amount of water present in the biomass and determined according to Equation 4. However, analysis of moisture percent of different particle size (2, 4 and 6mm) composite raw and torrefied sugar cane dry and top leaves was obtained by putting 5 gram sample on moisture analyzer (Sartorius MA35, Germany).

$$\%MC = \frac{(\mathrm{A}-\mathrm{B})}{\mathrm{A}} \; \mathrm{X} \; 100 \qquad (4)$$

Where A = initial mass of the sample; B = final mass of the sample after heating.

Figure 10: Oven and moisture analyzer used for analysis

3.4.2.2 Ash content

The ash content (% AC) represents the mass of biomass that does not undergo combustion, that is, it represents the inorganic residue that remains after the burning of organic matter. The ash content of different particle size (2,4 and 6mm) sugar cane dry and top leaves composite

raw and torrefied sample were determined in triplicate by firing 2g sample in a muffle furnace at 750°C for two hours with semi-capped crucibles. The ash content was calculated according to Equation (5).

$$\%AC = \frac{(C-D)}{A} X100 \quad (5)$$

Where, C = mass of the crucible with lid and ash residue; D = mass of empty crucible with lid;

A = initial mass of the sample.

3.4.2.3 Volatile matter

The volatile matter (% VM) represents the fraction of the biomass that is released in the form of gases formed from the exposure of the sample to high temperatures. The volatile fractions of different particle size sugar cane dry and top composite raw and torrefied sample were determined in triplicate by heating 1g sample in a crucible with a lid for 6 minutes at 950°C under muffle furnace. To know the percentage of total mass loss (% ML) in the thermal process, first calculated it was used Equation (6).

$$\%ML = \frac{(F-G)}{(F-D)} x100 \quad (6)$$

Where D = mass of the empty crucible with a lid; F = mass of crucible with lid and sample before heating; G = mass crucible with lid and sample after heating.

The percentage of volatile material (% VM) was obtained by Equation (7), where the moisture content initially present in the sample is discounted.

$$\% VM = \% ML - \% MC \quad (7)$$

Where % ML = mass loss; % MC = moisture content of the sample.

3.4.2.4 Fixed carbon

The fixed carbon (%FC) of different particle size sugar cane dry and top raw and torrefied composite sample were determined in triplicate by difference using Equation (8) and corresponds to the amount of carbon remaining after discounting moisture, ash content, and volatile matter.

$$\% FC = 100\% - (\% MC + \% AC + \%VM) \quad (8)$$

3.4.3. Calorific value determination

The characterization of the high heating value (HHV) allows the knowledge of the energy efficiency of the material, that is, the amount of energy released in the form of heat during the complete combustion per unit mass of the material, which can be measured in kJ/kg or MJ/kg. Gross heating values (HHV) of raw dry leaves and tops, different particle size sugar cane dry and top composite torrefied samples, and briquettes produced at optimized condition were determined in triplicate using an oxygen bomb calorimeter (Wondemagegnehu et al., 2022). For each analysis, 0.78 gram of dried and milled sample was taken in a clean glass crucible. The crucible was placed in the bomb by letting the nickel wire tied with cotton to be immersed in fuel sample. Then it filled with oxygen at 30 bars and placed in the calorimeter, which was filled previously by a known mass of water. Then after the bomb automatically ignite sample and after a few minute it gives the value.

Figure 11: Bomb calorimeter used for raw and torrefied sample analysis

3.4.4. Ultimate analysis

The elemental composition of biomass is an important property that defines the energy content and determines the applicability of a material. It is an assay used to determine the chemical composition of different materials, providing the mass percentages of the elements carbon (C), hydrogen (H), oxygen (O), nitrogen (N) and sulfur (S) contained in the sample. In addition to the elements mentioned above, the ultimate analysis also provides the ratio between the atomic percentages of hydrogen/carbon (H/C) and oxygen/carbon (O/C). In this work, the elemental composition was determined through the use of empirical correlations based on a large number of data and covering all categories of solid lignocellulosic materials. Such correlations

use the results previously obtained in the proximate analysis, being a simple, rapid, economic, and efficient method. The values of the ultimate analysis were obtained through Equation 9 (wt% carbon), Equation 10 (wt% hydrogen), and Equation 11 (wt% oxygen), (Silveira et al., 2020).

$$C(\%) = 0.637 = (FC) + 0.455(M)Wt\% \quad (9)$$

$$H(\%) = 0.052(FC) + 0.062(VM)Wt\% \quad (10)$$

$$O(\%) = 0.304(FC) + 0.476(VM)Wt\% \quad (11)$$

Where CF = fixed carbon; VM = volatile material.

3.4.5. Fourier transmission infrared spectroscopy analysis (FTIR)

With the help of Fourier transform infrared spectrometer (FT-IR), surface functional groups and atomic bonding on the surface of the selected particle size (2mm) torrefied at different temperature and the raw composite were characterized (spectrum 65 FT-IR, PerkinElmer), using the infrared spectra range of 4000 to 400 cm^{-1} at 32 scans for a resolution of 8 cm^{-1}. First, the sample was mixed with KBr tablet particles to make it suitable for infrared analysis. The mixture then pressed to small thickness, slightly below l mm, which is required for FTIR analysis.

3.4.6. Thermo-gravimetric analysis

Thermo-gravimetric analysis is a high- precision analytical technique that can be used to support the study of torrefaction at low heating rate, being able to provide relevant information on the kinetics of the reaction processes. It also applied to evaluate the gradual rate of mass loss of the biomass as a function of temperature. In this work tests were performed to investigate change in chemical structure of the selected particle size (2mm) torrefied at different temperature and raw composite by TGA-DTA analyzer (C30575100456TK series), by heating a typical mass of 11mg in a purge of nitrogen (50 ml min^{-1}), at a heating rate of 10 ^0C min^{-1} with final temperature of 1000^oC.

3.5. Design of Experiments

The set of torrefaction and briquetting were based on a central composite design. It is useful for statistical modeling and optimization of a response variable of interest. Moreover, central composite design allows estimating coefficients in a second degree polynomial regression and modeling of a quadratic response surface. The response surface can be further used for process

optimization, identification of maximum or minimum responses, and significance of each involved factors, or their combination. In this work ,to establish the optimum conditions in terms of particle size, residence time and torrefaction temperature, 3 Level factorial methodology for experiment were used (Table 5).

Table 5: Coded and actual levels of the factors for three factors design of torrefaction

Independent variables	**symbol**	**Treatment combination for coded and actual levels**		
		-1	0	1
Particle size(mm)	X1	2	4	6
Torrefied temperature(°C)	X2	230	260	290
Residence time(min)	X3	0	30	60

Where x_1, x_2 and x_3 are independent variables and -1, 0 and 1 are levels

Similarly, in the case of briquetting, to establish the optimum conditions in terms of compression force, holding time, and pressure also, 3 Level factorial methodology for experiment were used as the torrefaction (Table 6).

Table 6: Coded and actual levels of the factors for three factors design of briquetting

Independent variables	**symbol**	**Treatment combination for coded and actual levels**		
		-1	0	1
Pressure (Mpa)	Y1	8	10	12
Dwell time (min)	Y2	3	5	7
Length of the die (mm)	Y3	40	50	60

Where, Y_1 and Y_2 and Y_3 is independent variable and -1, 0 and 1 are levels.

In three-level factorial, the number of experiments is according to the equation: $N = 2^n + 2xn + nc$, where n*c* is the number of central points, *n* is the number of factors and *N* is the number of experiments. As required by design, in both cases all factors will be adjusted at three equally spaced levels (− 1, 0, + 1) (Portilho et al., 2020).

3.5.1 Torrefaction of sugarcane dry leaf and top composite

Medium walled porcelain crucibles (50 ml) was pre-fired in a furnace at 290 °C for an hour and was allowed cooling in desiccators for an hour before weighing to determine their empty

dry weight. Pre-fired and pre-weighed porcelain a crucible is filled up to 90% of their capacity with 13 g of sugarcane dry leaves and tops composite (60:40 ratio) with 2, 4 and 6mm size. The covered crucibles containing the samples were loaded into the muffle furnace at a maximum of 6 crucibles pretreatment for each particle size. The furnace is heated from room temperature to the desired furnace temperatures (230, 360 and 290°C) at a heating rate of 10 °C/min. After reaching the desired temperature, the composite sugar cane sample was held for a torrefaction holding time of 30 and 60 minutes. After torrefaction experiment were completed, furnace was turn off, left to cool down to ambient temperature. Cooled samples were stored in 250 ml screw-capped glass sample bottles for later characterization and analysis. Duplicate trials were carried out for each torrefaction condition (Conag et al., 2018).

3.5.2 Mass yield of the torrefied sample

The mass and energy yields determine the transition of mass and chemical energy from biomass to bio-char. Solid Yield (Mass Yield) is defined as the fraction of the original organic component of biomass that is converted into solid char. It is defined on dry basis for biomass with high inorganic content or on dry ash free basis for biomass with low inorganic content. Responses solid and energy yields to the torrefaction treatment were tabulated as equation (12 and 13) (Mamvura & Danha, 2020).

$$Solids\ yield = \frac{Mtorr}{Mraw} \quad (12)$$

Where M_{raw} is the mass of raw biomass input after drying,

M_{torr} is the mass of product output (bio-coal or torrefied biomass).

3.5.3 Energy yield of torrefied sugar cane dry leafs and tops

On the other hand, energy yield gives the fraction of the original energy in the biomass retained after torrefaction. It was calculated by the following equation (Eq 13).

$$\text{Energy yield} = \text{Mass yield} * \text{Energy density, Energy density} = \frac{HHVtorr}{HHVraw} \quad (13)$$

Where, HHV_{torr}, HHV_{raw} are higher heating values of torrefied biomass and raw biomass respectively. The higher heating values of torrefied biomass were calculated according to (Wondemagegnehu et al., 2022).

3.6. Briquette Production

The main post – treatments of torrefied product is densification by briquetting, aiming to promote it as a promising bio-fuel in terms of transportation, storage and handling. The sugarcane dry and top composite torrefied briquettes were produced by the selected 2mm particle size torrefied at 290^{o}C for 60 minute product with cylindrical mould of 50mm diameter and a height of 40, 50 and 60 mm, a pressure of 8, 10 and 12 Map (Espuelas et al., 2020) pressing period of 3, 5 and 7 minute with a constant temperature of 150^{o}C throughout the production respectively. For heating purpose the die part was covered with coil heater. The densification process was performed at material science department in Adama science and Technology University using laboratory hydraulic briquette machine. Based on the design expert software, a combination of pressure dwell time and die length 30 briquettes were produced. After the briquettes removed from the mould, the length, height and weight was measured using vainer caliper and analytical balance.

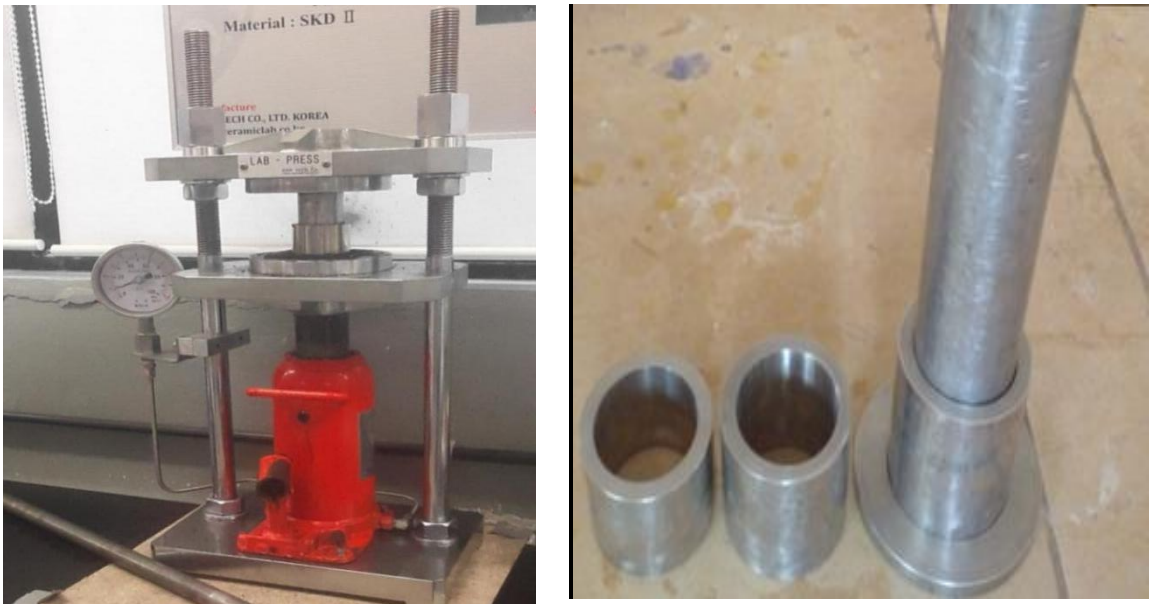

Figure 12: Mechanical press and cylindrical die for briquette production

3.6.1 Bulk density

Bulk density is a major physical properties in designing the logistics system for biomass handling .The weights of Briquettes were determined using a digital balance and the volumes were calculated based on the direct measurement of height and diameter of the Briquettes

since the Briquettes shape is cylindrical. The bulk density (dry basis) was calculated from the mass and volume of briquettes as given below (Yusuf Kpalo & Faiz Zainuddin, 2020).

$$\rho_{br} = \frac{M_{br}}{V_{br}} \quad , \quad V_{br} = Base\ area\ \boldsymbol{x}\ Height\ = \pi r^2 h \tag{14}$$

Where; V_{br} = volume of Briquette (cm^3)

Π = mathematical constant (3.14)

H = height of the Briquette (cm),

r= radius Briquette

ρ_{br} = density of Briquette (g/cm3) & M_{br} = mass of Briquette (gm).

3.6.2 Shatter resistance

The shatters resistance of the briquette was determined using a pellet durability tester. Each briquette sample was allowed dropping from a height of 2 m onto a concrete floor five times. The shatter resistance calculated as the ratio of the final weight of the briquette retained after five drops to the initial weight of the briquette. The fraction of the briquette that remained un-shattered was used as an index of briquette durability. The percent weight loss of briquettes is expressed as a percent of the initial mass of the material remaining on the solid base; while the shatter resistance is obtained by subtracting the percent weight loss from 100 as shown below (Yusuf Kpalo & Faiz Zainuddin, 2020).

$$\text{WL (\%)} = \frac{(W1-W2)}{W1} x100 \tag{15}$$

$$\text{SR (\%)} = 100 - WL \tag{16}$$

Where; WL (%) = weight loss,

W1 = weight of Briquette before shattering

W2 = weight of Briquette after shattering

SR (%) = shatter resistance

4. RESULTS AND DISCUSSIONS

4.1. Raw materials composition

The chemical composition analysis result and heating values of raw sugarcane dry leaves and sugarcane top were shown in Table 7. The analysis result showed that, the percentages of extractive were found to be lower in sugarcane dry leaves (11.7%) than top (18.29%). The lower proportion of extractives indicates lower coloring matter in the dry leaves than top. however, the amount in both dry leaves and tops are much lower than the amount of extractive reported by (Menandro et al., 2017). The composition of the material concerning lignin is very high for the dry leaves (28.31%) compared to the amount in tops (21.48%) (Table7). This is associated with silica ,which the tops leaves does not need to metabolize a lot of lignin in the tops since silica does (Gómez et al., 2014). The composition with respect to hemicelluloses and cellulose in the dry leaves were 27.62% and 27.39% respectively and for sugarcane tops the corresponding values were 25.12%, and 30.97%. The ash content of dry leaves (4.98%) and tops (4.14 %) is within the range acceptable to make solid fuel (2-16%), according to (Go & Conag, 2019). The heating value obtained from bomb calorimeter was 16.88 MJ/kg and 16.82 MJ/kg for sugar cane dry and top leaves, respectively. The result obtained in agreement with the HHV, result reported by (Menandro et al., 2017). The amount of sugarcane dry and top in the sugar estate was assessed by taking sample using X-shape design, were found to be 10,512.84 kg/ha (105.1284Kuntal) dry and 5,679.6795 kg/ha (56.797kuntal) top leaves respectively. This means 64.92% dry and 35.08% top leaves of the residual biomass can be used to generate energy for cooking and heating. Nevertheless, much greater amounts of surplus electrical energy could be generated if this straw is incorporated as fuel in the co generation systems of the cane sugar industry.

Table 7: compositional analysis result of raw sugarcane leaf and top

Sample	Extractive %	Hemicelluloses %	Lignin %	Cellulose %	Ash %	HHV MJ/kg
Dry	11.7±2.12	27.62±1.7	28.31±0.66	27.39±2.68	4.98±0.12	16.88
Top	18.29±1.13	25.12±2.08	21.48±1.34	30.97±0.37	4.15±0.02	16.82

Data were means ± SD (standard deviation)

4.2. Proximate Analysis Result of Torrefied Sugarcane Dry and Top Composite

Table 8 shows the proximate analysis result (moisture, volatile matter, fixed carbon and ash content) of raw and torrefied sugarcane dry and top composite obtained by varying particle size, temperature, and retention time. Moreover, the 30 experimental run and its responses are tabulated based on equation (4, 5, 6, 7, and 8) below using designs expert (Table 8).

Table 8: Proximate analysis result of sugarcane dry and top composite based on the design

Run	Factor			Response			
	PS(mm)	**TT(^{O}C)**	**RT(min)**	**M (%)**	**V M (%)**	**FC (%)**	**Ash (%)**
1	6.00	260.00	60.00	2.4	75.98	11.75	9.87
2	4.00	260.00	30.00	3.21	76.57	10.89	9.33
3	4.00	230.00	30.00	3.75	80.1	7.24	8.91
4	2.00	290.00	60.00	1.32	63.12	23.36	12.2
5	4.00	260.00	30.00	3.21	76.57	10.89	9.33
6	4.00	260.00	60.00	2.35	73.06	14.62	9.97
7	2.00	260.00	60.00	2.2	68.17	19.43	10.2
8	4.00	290.00	60.00	1.61	67.7	18.59	12.1
9	4.00	290.00	0.00	6.93	82.18	5	5.89
10	4.00	230.00	60.00	2.97	78.6	9.3	9.13
11	2.00	290.00	0.00	5.75	82.43	5.72	6.1
12	4.00	290.00	30.00	2.15	68.23	18.72	10.9
13	2.00	230.00	60.00	2.87	76.33	11.65	9.15
14	6.00	230.00	0.00	6.45	82.01	4.58	6.96
15	2.00	290.00	30.00	2.05	66.42	21.13	10.4
16	2.00	260.00	0.00	5.75	82.43	5.72	6.1
17	6.00	290.00	30.00	2	71.13	15.37	11.5
18	6.00	230.00	60.00	3.65	80.44	7.3	8.61
19	2.00	260.00	30.00	2.95	74.39	13.21	9.45
20	6.00	260.00	30.00	3.71	77.96	9.74	8.59
21	4.00	260.00	0.00	6.93	82.18	5	5.89
22	6.00	290.00	0.00	6.45	82.01	4.58	6.96
23	6.00	290.00	60.00	1.76	69.96	12.48	15.8
24	4.00	260.00	30.00	3.21	76.57	10.89	9.33
25	4.00	230.00	0.00	6.93	82.18	5	5.75
26	6.00	260.00	0.00	6.45	82.01	4.58	6.96
27	6.00	230.00	30.00	3.92	82.37	6.21	7.5
28	2.00	230.00	30.00	3.55	79.68	7.83	8.94
29	2.00	230.00	0.00	5.75	82.43	5.72	6.1
30	4.00	260.00	30.00	3.21	76.57	10.89	9.33

Where PS = Particle Size, TT = Torrefaction Temperature, RT =Retention Time, M = Moisture, VM =Volatile Matter, FC = Fixed Carbon

By applying multiple regressions analysis on the experimental data, the following second degree polynomial was found to present the relationship between the response variables

(moisture , VM, FC, and Ash content) of the torrefied composite sample and the factors (particle size, temperature and retention time) adequately.

$$Moisture = +3.25 + 0.26 * A - 0.55 * B - 2.01 * C - 0.063 * A * B - 0.057 * A * C - 0.40 * B * C - 0.25 * A^2 - 0.100 * B^2 + 1.34 * C^2 \quad (17)$$

$$\text{Volatile matte} = +76.02 + 1.58 * A - 3.94 * B - 4.81 * C + 0.40 * A * B + 1.67 * A * C - 2.88 * B * C - 0.31 * A^2 - 0.64 * B^2 + 2.01 * C \quad (18)$$

$$Fixedcarbon = +11.63 - 2.07 * A + 3.34 * B + 4.59 * C - 0.89 * A * B1.62 - * A * C + 2.18 * B * C - 0.34 * A^2 - 0.24 * B^2 + 2.33 * C^2 \quad (19)$$

$$Ash = +9.10 + 0.23 * A + 1.16 * B + 2.24 * C + 0.56 * A * B + 0.013 * A * C + 1.09 * B * C + 0.24 * A^2 - 0.49 * B^2 - 1.04 * C^2 \quad (20)$$

The significance of the fit of the second-order polynomial for the torrefied moisture, volatile matter, fixed carbon and ash was assessed by carrying out analysis of variance (ANOVA) with results shown in Table 9.

Table 9: Analysis of variance for moisture, volatile matter, fixed carbon and ash of the composite

Source	**df**	**Mean Squares**			
		moisture	**volatile matter**	**Fixed carbon**	**Ash**
Model	9	94.03*	100.46*	87.99*	141.60*
A-Particle size	1	1.18*	45.03*	*76.80**	0.94 ns
B-Temperature	1	5.36*	279.74*	*200.80**	*24.04**
C-Residence Time	1	73.04*	415.68*	*378.86**	*90.32**
AB	1	0.048ns	1.88 ns	*9.49*ns	*3.72**
AC	1	0.039ns	33.40*	*31.66**	1.875E-003 ns
BC	1	1.92*	99.71*	*57.12**	*14.24**
A^2	1	0.42ns	0.67ns	*0.78*ns	0.38 ns
B^2	1	0.069ns	2.78ns	*0.40*ns	*1.63*ns
C^2	1	12.37*	27.78*	*37.10**	*7.40**
CV %		8.82	2.11	17.65	7.97
R-Squared		0.9761	0.9455	0.9216	0.9336
Adj R-Squared		0.9653	0.9209	0.8863	0.9037
Pred R-Squared		0.9422	0.8654	0.7939	0.8037
Standard deviations		0.34	1.61	1.84	0.71

Remark:∗ indicates the effect of the specific factor has a significant effect on the specified response variable at alpha = 5% while ns indicates insignificant terms at some alpha value's = coefficient of variation, R^2 = the coefficient of determination, df = desirability function

As observed from Table 8, after torrefaction, the MC, FC, VM, and ash of torrefied samples were in the ranges of 1.32–3.92, 6.21–23.36, 63.12–82.37, and 7.50–15.80%, respectively. It can be clearly concluded that regardless of the processes, the increments of reaction temperature reduced the volatile matter content and increasing the content of fixed carbon (Table 8). For example, the maximum fixed carbon and volatile matter content obtained was 23.36 and 63.12%, respectively. Both parameters significantly changed compared to raw materials (5.72% fixed carbon, 82.43% volatiles) at 2mm particle size. This is in line with the result reported by (Gan et al., 2019) which as the particle size decreases from 2mm to 1mm the, FC, VM, and ash content 20.94, 65.13, and 11.5%. Thus, the combinations that gave high energy density were found to be at PS (2mm), TT (290^{o}C), and RT (60 min). During torrefaction, a part of the raw biomass is released as volatiles due to the thermal decomposition of hemicellulose and a bit of cellulose, thus a significant reduction in volatile content of the torrefied solid product was observed, while the relative amounts of fixed carbon and ash increased (Khempila et al., 2022).

As shown in Table 9, the R^2, coefficient of determination of the model was 0.9761, 0.9455, 0.9216, and 0.9336 for moisture, volatile matter, fixed carbon and ash, respectively. The obtained values were close to 1 and it reveals that the model adequately represented the real relationship between the variables under consideration (Helwani et al., 2018).The "Pred-R Squared" of 0.9422, 0.8654, and 0.8037 were in reasonable agreement with the "Adj R-Squared" of 0.9653, 0.9209, 08863, and 0.903. With pre-R^2 of 94.22%, 86.54%, 79.39% and 80.37% variability, the suggested model equations can predict the responses (moisture, volatile matter, fixed carbon and ash respectively) (Tadesse, 2018). The observed low value result of coefficient of variation (C.V) was 8.8, 2.11, 17.65, and 7.97 for moisture, volatile matter, fixed carbon and ash, respectively suggests a high reliability of the experiment (Belay, 2014).

Analysis of variance for MC, VM, FC and Ash revealed that, one way interaction of PS, TT, and RT has significant effect on volatile matter, moisture, and fixed carbon except ash content (Table 9). Moreover, the two way interaction effect of PS and, TT had a significant impact on ash content. And also a two way interaction of PS and RT has significant effect on VC and FC. The result also proved that the two way interaction effect of TT and, RT has a significant

effect on all of the responses tested and hence the mean separations of these interactions were considered to determine the optimum treatments combination. The interaction of particle size and residence time with temperature and residence time has a significant effect on the volatile matter of the torrefied sample. In the case of fixed carbon, the interaction of AC, BC, and C^2 had a significant contribution to the increase in fixed carbon content during the process. But the interaction of AB, A^2, and B^2 was not significant, due to the large p-value. The analysis of variance of the Model for Ash as shown in Table 9: B, C, AB, BC, and C^2 are significant model terms.

4.3. Optimization Result of Torrefaction process

4.3.1 Effect of temperature and particle size on moisture content of composite

As torrefaction temperature increases from 230°C to 290°C the sample moisture percent of the torrefied material decrease from 6.93 to 1.32 %, in which the composite with the lowest particle size (2mm) had the lowest MC (1.32%) (Figure13). This is due to the distribution of heat increase within the particles. The improved hydrophobicity of the sugar cane composite as the temperature increased may be attributed to the removal of components having functional group (-COOH and –OH) having strong affinity with water. These components are often released as organic acids, ketones and aldehydes up on decomposition and volatilization, that makes the torrefied material more easily combustible (Chen et al., 2015).

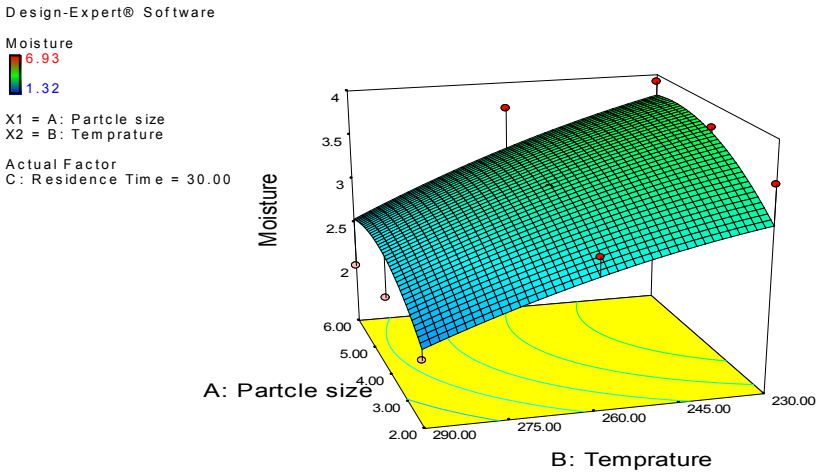

Figure 13:3D response surface plot of temperature and particle size on moisture content

4.3.2 Effect of temperature and particle size on fixed carbon content of the composite

As shown in Figure14, the increase in torrefaction temperature to 290 ºC , reduces the VM content (79.68 to 63.12%) and increase the FC content (7.83 to 23.36%). The highest FC content and lowest VM content of dry torrefied sample, 23.36% and 4.58%, respectively were obtained in the composite with particle size 2 mm than 6 mm (Figure 14). This is comparable with the result reported by (Khempila et al.,2022) and (Go & Conag, 2019), in which the FC content of sugar cane dry and top composite after torrefaction were 22.72 at 275ºC and 24.40 % at 300ºC, respectively. This implies as the particle size decreases from 6mm to 2mm the formation of carbonized materials occurred in carbonization, and thermal cross-linking reaction increased the fixed carbon of the torrefied biomass (Gan et al., 2019).

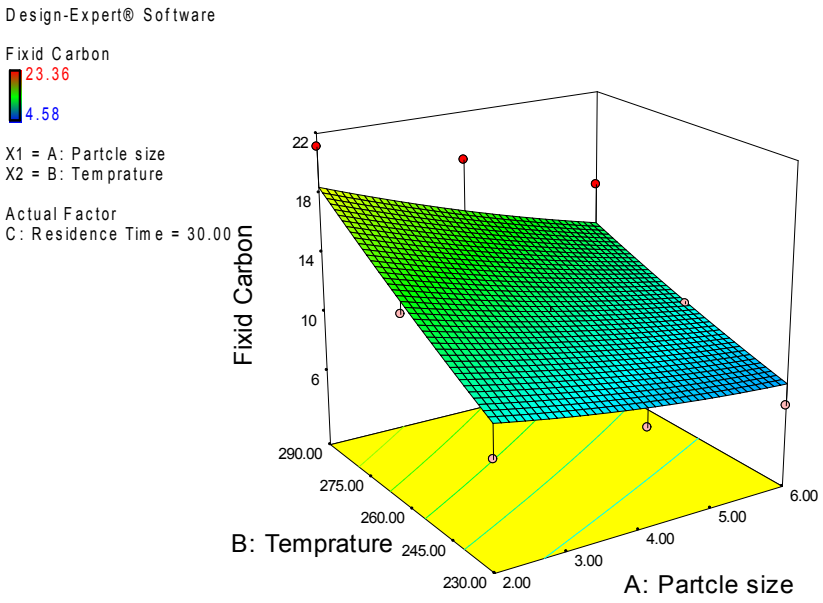

Figure 14: 3D response surface plot of temperature and particle size on fixed carbon content

4.3.3 Effect of temperature and particle size on volatile matter content of composite

During torrefaction , a part of the raw biomass is released as volatiles due to the thermal decomposition hemicelluloses and a bit of cellulose, thus a significant reduction in volatile content of the torrefied solid product was observed, which is about 30% of the mass are lost as volatile and permanent gases, mainly CO_2 and condensable species. From the Table 8 the

volatile matter of the raw composite was 82.43, 82.18 and 82.01% with particle size of 2,4 and 6mm respectively, however after the sample undergo torrefaction, the volatile matter of the torrefied product decrease as the torrefaction temperature increase from 230-290^{0}C and with decrease in particle size. Similarly the literature mad by (Conag et al., 2018) on sugar cane leaves shows the decrease in volatile matter as temperature and torrefaction time increases 65.67,39.86 and 32.17% with a time of 30, 45 and 60 minute. From the Figure 15 below the minimum volatile matter of the sample obtained were 63.12% with 2mm particle size. This result strongly correlates with torrefied sugar cane leaves, which is 63.64±0.53by (Khempila et al., 2022).

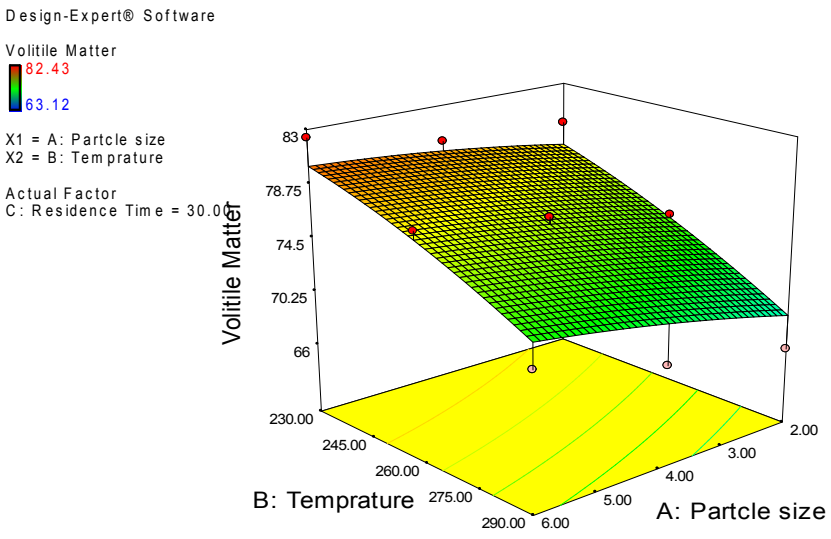

Figure 15: 3D response surface plot of temperature and particle size on volatile matter content

4.3.4 Effect of temperature and particle size on ash content of composite

The increased ash content was observed in torrefied sample as shown in Table 8.Thus,ash content increased with raising reaction temperature .The decrease in mass yield during torrefaction, leading to the buildup a high concentration of metallic elements (Cellatoğlu &İlkan, 2015). Figure 16 revels that, as torrefaction temperature increase from 230 to 290^{o}C with decrease in particle size 2mm the ash amount increases too. High ash concentrations are not favorable for thermal conversion, since they decrease the burning yield, in addition to causing problems in the reactor structure, such as scale, corrosion and slag formation (Silveira et al., 2020,Portilho et al., 2020).

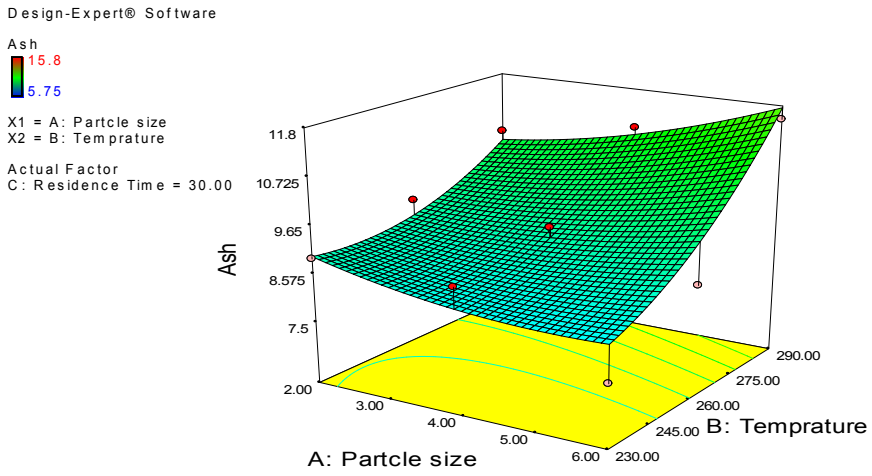

Figure 16: 3D response surface plot of temperature and particle size on ash content.

4.4. Analysis Result of Optimization of Torrefied Composite

From 30 experimental run of Table 8, the optimum torrefaction condition that yield maximum FC, minimum VM, and ash content were TT of 290, RT of 60min and PS of 2mm. Similarly, from the responses optimizer in Table 10 after torrefaction at TT 285.03, RT 59.36 min and PS of 2.00 mm the responses result obtained was MC of 1.32%, FC of 23.36%, VM of 63.30%, and AC of 12.01%, which is strongly agree with (Khempila et al., 2022). The observed result indicated that, the torrefaction process could be preferably made around a temperature of greater or equal to 290^{o}C at a particle size of 2mm with a holding time of less than equal to 60 min. Hence, the experimental run carried out at TT of 290, RT 60min and PS of 2mm were selected for further investigation.

Table 10: Optimized result of torrefaction

PS	TT(^{o}C)	RT(min)	MC (%)	VM (%)	FC (%)	AC (%)	Desirability
2.00	285.03	59.36	1.32	63.30	23.36	12.01	0.781(selected)
2.00	284.85	59.7	1.33	63.29	23.35	12.01	0.781
2.00	286.06	59.47	1.26	63.34	23.36	12.02	0.78
2.00	286.34	56.99	1.25	63.35	23.35	12.03	0.78

Where, PS referees = Particle Size, TT= Torrefaction Temperature RT= Residence Time, MC= Moisture Content, volatile Matter, FC= Fixed carbon, AC= Ash Content

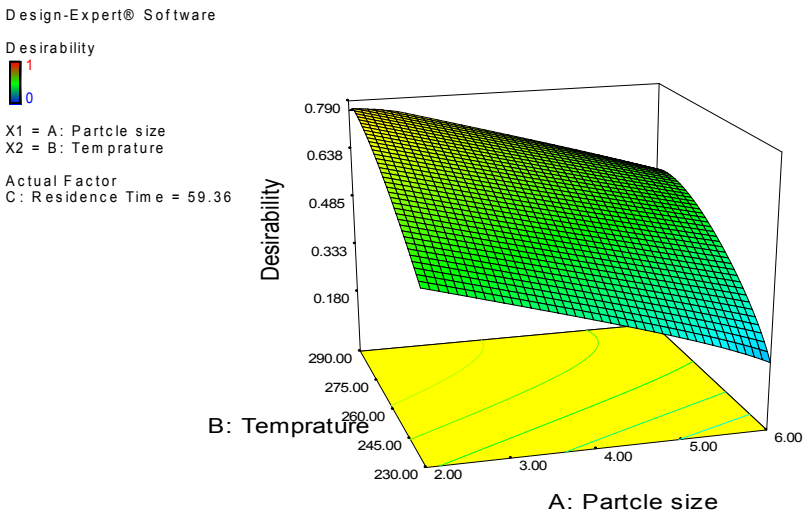

Figure 17: 3D response surface plot for optimum torrefaction yield

4.5. Ultimate Analyses Result

The ultimate analysis provides the proportion of C, H, O, N, and S that plays a major role in biomass energy properties. In general, biomass with a high content of carbon and low content of oxygen are preferred for energy purposes. Table 11 showed the ultimate analysis result of raw and torrefied sugarcane dry and top composite sample with grain size of 2mm. The contents of C, H, and O in raw sugar cane dry and top composite were 41.15, 5.41, and 39.67 %, respectively. After torrefaction the contents of C, H, and O at various torrefaction temperature and dwelling time were in the range of (41.24 to 43.68%), (5.35 to 5.13), and (38.71 to 37.15), respectively (Table 11). The highest C (43.60%) was recorded for samples torrefied at a temperature of 290^{o}C and dwellings time of 60 min., which is accompanied by lowest O (37.15) and H (5.13%) contents. The observed increase in carbon content and respective decrease in the content of O and H with elevating reaction temperature, and holding time in the process of torrefaction is in agreement with the results reported by Matali (Matali et al., 2016). It is mainly due to decarbocylation and dehydration reaction of hydrolyzed products, which release water and oxygen containing compounds like phenol and acids in liquid phase and gaseous products as carbon dioxide and (Khempila et al., 2022). The results with respect to H/C and O/C of the torrefied product revealed that, when the reaction temperature increased to 290^{o}C, the atomic H/C and O/C atomic ratio dropped from 0.13 to 0.12 and 0.96 to 0.85, respectively (Table 11). This indicated the need of torrefaction

pretreatment at higher temperature and holding time to increase hydrophobicity of the raw sugarcane dry and top composite (Cellatoğlu & İlkan, 2015, Khempila et al., 2022), which makes it more suitable for dry biofuel production. Moreover, the higher the proportion of oxygen and hydrogen, compared to carbon, indicated the lower the energetic value of a material, due to the lower energy involved in the C-O and C-H bonds than in the C-C bond (Silveira et al., 2020).

Table 11: Ultimate analysis result of 2mm grain size raw and torrefied at (230ºC, 260 ºC, and 290ºC) on holding time (30 min, 60min) sugarcane dry and top composite.

samples	C	H	O	H/C ratio	O/C ratio
Raw SDL & TC	41.15	5.41	39.67	0.13	0.96
TTt-230/30	41.24	5.35	38.71	0.13	0.94
TTt - 230/60	42.15	5.34	38.95	0.13	0.92
TTt-260/30	42.26	5.30	36.46	0.13	0.86
TTt-260/60	43.39	5.24	37.52	0.12	0.86
TTt-290/30	43.68	5.22	38.04	0.12	0.87
TTt-290/60	43.60	5.13	37.15	0.12	0.85

Where SDL = Sugarcane Dry Leaf, TC =Top and Composite, TTt =Torrefaction Temperature and Time

4.6. Mass yield, Energy yield and Energy density Results

The major decomposition of biomass depends on properties of biomass and the reaction temperature during torrefaction. Thus, the effect of reaction temperature on the change in biomass samples physical properties such as color and solid yield was investigated**.** The change in color is a natural phenomenon during torrefaction or roasting of the biomass material (Khempila et al., 2022). As observed in appendix (VI), when increasing the torrefaction temperature, the color of the biomass changes from brown to black. This is mainly attributed to the chemical compositional changes that occur in the biomass components, such as hemicellulose, lignin, and cellulose (Akanni et al., 2019). The major reactions that change the biomass color are devolatilisation and carbonization of hemicellulose, de-polymerization

and de-volatilization/softening of lignin, and de-polymerization and de-volatilization of cellulose (Jaya Shankar Tumuluru, 2016). The chemical compositional changes are due to the breakage of hydrogen and carbon bonds. The disruption of most inter- and intra-molecular hydrogen bonds, C–C, C-H and C–O bonds, results in the formation of hydrophilic extractives, carboxylic acids, alcohols, aldehydes, ether, and gases (e.g. CO, CO_2, and CH_4) (Jaya Shankar Tumuluru, 2016, Akanni et al., 2019, Khempila et al., 2022).

Table 12 shows the high heating values (HHVs), mass yield and energy yield as well as energy density of raw and torrefied sugar cane composite biomass at various temperature and residence time. The result reveals that the mass yield of terrified sugar cane sample ranges from 82.53% at (230^{o}C, 30 min.) to 55.9% (at 290^{o}C, 60min), which slightly agree with literature work by (Chen et al., 2015). Mass yield of torrefied sample were significantly decreased with increasing reaction temperature and time in torrefaction processes as reported by (Conag et al., 2018) on torrefied sugar cane leaves. The char yield decreased with increasing temperature and residence time due to release of Volatile gases or condensable substances (Trubetskaya et al., 2020). The energy yield can be regarded as a significant indicator to the amount of energy retained after torrefaction (Matali et al., 2016).

The energy yields of sugarcane composite also decrease from 86.86% to 65.40% with increase in torrefaction temperature from 230^{o}C to 290^{o}C. This implied that temperature has a large effect on both the mass and energy yield negatively. On the other hand, the HHV of torrefied biomass increased from 17.35MJ/kg to 20.03MJ/kg with increase in temperature 230^{o}C to 290^{o}C (Table 12). It is in agreement with the result reported by (Conag et al., 2018), in which the maximum HHV found to be 22.04 MJ/kg after torrefied at 300^{o}C but increase in temperature to 350^{o}C and torrefaction time beyond 60 minute did not result further increase in HHV. The increase in heating value with temperature and dwelling time is due to reduction of H and O following torrefaction (Khempila et al., 2022). The combined effect of torrefaction temperature and dwelling time were more pronounced on MY (mass yield) and EY (energy yield) during torrefaction at higher temperature in which as torrefaction temperature progressing towards 290^{o}C and dwellings time towards 60min, both the MY (mass yield) and EY (energy yield) drastically decreased. This implied that, for maximum MY, torrefaction must be carried out at low temperature and residence time. This is due to the increased mass loss as a result of degradation of hemicelluloses and cellulose content of biomass with

temperature and residence time (A. A. Adeleke et al., 2019; Orisaleye et al., 2022). Hence, torrefaction of the sugarcane leaves and tops composite at a temperature of 290^{o}C and residence time of 30 min. were found to be optimum for better mass yield, energy yield and HHV (Table 12). However, the lower moisture content, the higher heating value of 20.03 MJ/kg in the torrefied sample at 290^{o}C and holding time of 60min which make preferable than heating value of 19.07 MJ/kg at a temperature of 290^{o}C and residence time of 30 min. In addition to this, the FC content of the latter was greater (23.36%) than the former one (20.13%). In addition to this, the energy density at 290^{o}C and holding time of 60min is greater than 290 ^{o}C and residence time of 30 min (Table 8 and 12).

Table 12: Mass yield, Energy yield HHV, and Energy density of raw and torrefied at (230 ^{o}C, 260 ^{o}C, and 290^{o}C) composite sample of particle size 2mm and for holding time (30,60min).

TT (^{O}C) / RT (min.)	Mass Yield (%)	HHV(MJ/kg)	Energy yield (W %)	Energy Density
Raw		17.07		
TT /t-230/30	82.53	17.35	83.36	1.01
TT/ t-230/60	82.72	17.89	86.86	1.05
TT/t-260/30	83.34	17.50	85.84	1.03
TT/t-260/60	74.04	18.63	80.70	1.09
TT/t290/30	71.84	19.07	79.74	1.11
TT/t-290/60	55.9	20.03	65.40	1.17

Where**,** TT = Torrefaction Temperature, RT = Retention Time**,** t =Torrefaction time

4.7. FT-IR Analysis Results of Torrefied Composite

The FT-IR spectra of the raw and torrefied sugar cane composite are shown in Figure 20 $_{a\text{-}c}$. The different spectra between the raw and torrefied sugar cane leaves composite occurred during the degradation of bio- polymer components after the torrefaction process. From figure 20, sugar cane leaves composite spectrum, an Overton peak of the O-H bond was found at a wave number of 3350cm^{-1}. This hydroxyl group, which can form ions and attract water molecules, is responsible for the hydrophilic behavior of biomass. As shown from figure 20, the intensity of the band decreased as the result of torrefaction severity rich to 290^{o}C. Again at a wave number of around 2911cm^{-1} indicates aliphatic hydrocarbon vibration of C-H symmetric stretching and dehydration. The strong band in the sugar cane leaves composite

observed at wave number of 1609cm^{-1} was associated with vibration in C=O and C=C from carboxyl groups of bio-polymer components such as cellulose, hemicelluloses (Tadesse, 2018). It's obvious that the intensity significantly become weaker as the temperature increases (Khempila et al., 2022) subsequently, less oxygen compounds (such as acid, alcohols, aldehydes and ethers) and non-condensable gas like CO_2 and CO were produced. The extinction(decrease) of OH group at 290^{o}C was primarily due to the complete dehydration and decarbocylation of hemicelluloses and cellulose which are rich in the OH group (Khempila et al., 2022). The loss of OH functional group from hydrophilic to hydrophobic make the torrefied samples losing the ability to attract water molecules and became more stable against chemical oxidation and microbial degradation. Moreover the structural components of the biomass were changed during torrefaction from hydrophilic to hydrophobic, which was associated with the formation of hydrogen bond (Manatura, 2020). This was consistent with the decreasing trend of oxygen and hydrogen with the elevated reaction temperature as indicted in Table 11.

The narrow band that occurs at a wave number of around 1030cm^{-1} is assigned to asymmetric stretching of C-O and C-C and is related to cellulose and hemicelluloses (Tadesse, 2018). This may be due to vibrations C-O and C-C that occurs in raw and torrefied sugarcane leaves composite. A significant reduction in cellulose and hemicelluloses or water were apparent only in sample torrefied at higher temperature (290^{o}C), showing that the biomass cellulose and hemicelluloses completely degraded under those conditions.

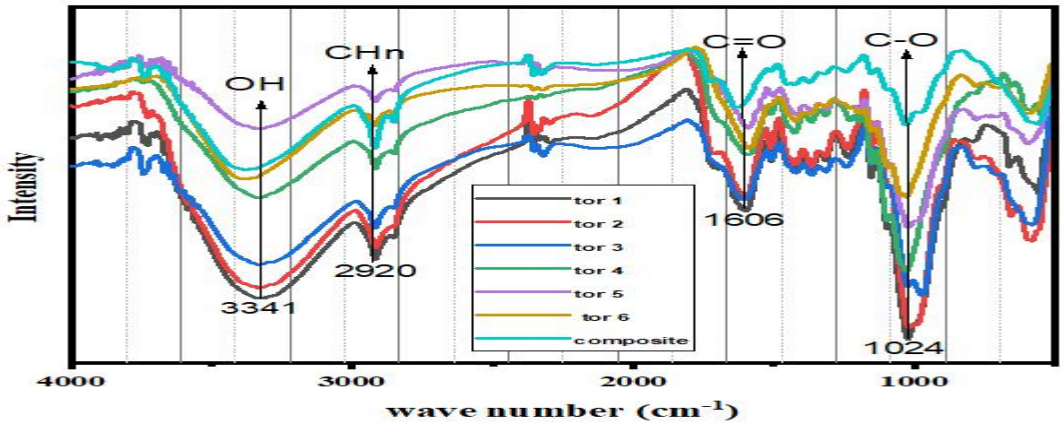

Figure 18: FTIR result of 2mm grain size at tor1 (230^{o}C/30min), tor 2 (230^{o}C/60min), tor 3 (260^{o}C/30min), tor 4 (260^{o}C/60min), tor 5 (290^{o}C/30min), tor 6 (290^{o}C/60min)

4.8. TGA/DTA Analysis Result

Differential Thermal Analysis (DTA) and Thermogravimetric Analysis (TGA) result of torrefied sugarcane composite were shown in Figure 19. As shown in the TGA curve, significant mass loss (4.27%) of torrefied sugar cane composite occurred at temperature beyond 260^{o}C (Figure 19$_{e}$). This implied that exposure of sugar cane composite to a temperature higher than 260^{o}C will bring about a massive volatile lose. This is consistent with the mass yield indicated in Table 12. To have a stable or combustion properties torrefied product curing in the neighborhood of 300^{o}C was needed (A. Adeleke et al., 2021). Thus, curing of sugar cane dry and top leaves blend biomass was carried out at 290^{o}C to avoid massive devolatilazation. Most of the torrefied samples were converted to char at around 600^{o}C. This is in line with torrefaction result of sugar cane bagasse made by Manatura (Manatura, 2020).

Degradation of mass is demarcated by the three different stages: dehydration, devolatilisation and char formation. From the previous studies(Gan et al., 2019), the initial stage of weight loss (4.63 and 6.18%) at Figure 19 $_{a \text{ and } b}$ at a torrefaction temperature less than 230°C occurred due to the moisture loss and thermo condensation (Chen & Kuo, 2011 , Jaya Shankar Tumuluru, 2016). This was then followed by the second stage, which the torrefaction temperature 260^{o}C at Figure 19 $_{c \& d}$, the weight loss, becomes (3.54 and 4.18%) as a result of de-volatilization (Dhaundiyal et al., 2020). During this stage, most of the hemicellulose decomposition and slightly degradation cellulose was observed (Chen & Kuo, 2011). The mass loss in Figure 19 $_{e \& f}$ observed were 4.27 and 1.09 % at severe torrefaction 290 ^{o}C, where large amounts of hemicellulose and cellulose were destroyed because of severe torrefaction and also at this condition lignin degradation, char formation occurs.

Though from the Figure 19 the torrefaction of sugar cane composite the TGA data tells us an effective torrefaction process can be at a temperature of 290^{o}C with holding time of 30min or 60min. But to made decision on the latter parameter other finding should be seen first like moisture, FC, and HHV values.

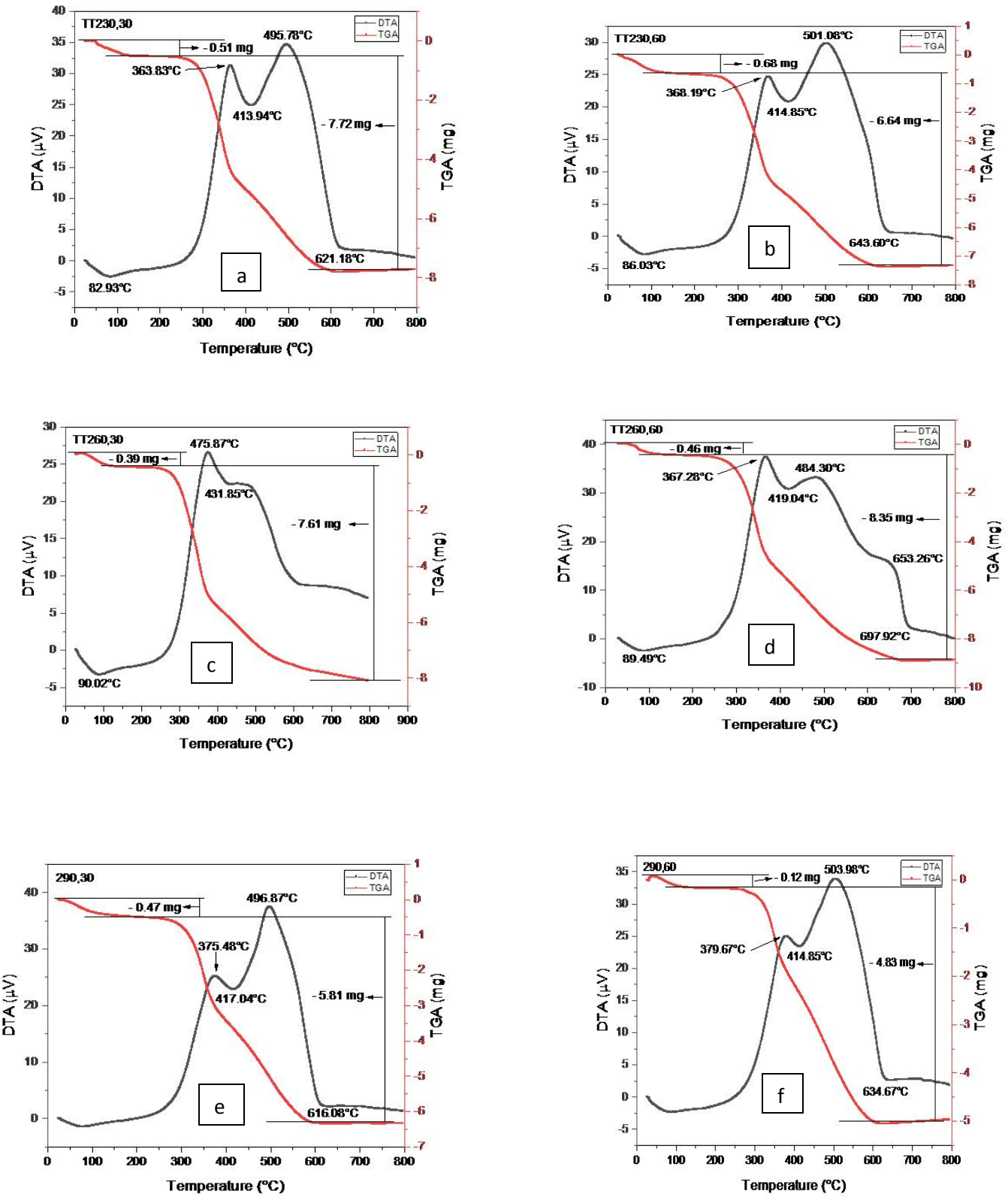

Figure 19: TGA/DTA result of Raw and torrefied at a (230ºC/30 min), b (230ºC/60 min), c (260ºC/30 min), d (260ºC/60 min), e (290ºC/30 min), and f (290ºC/60 min

In Figure 19 above DTA shows upward direction or voltage gain denotes the exothermic reactions; whereas, the voltage drop or the downward trend implies the endothermic nature of the reactions. The DTA curve of torrefied sugar cane composite reveals endothermic peak at a temperature of 82.93, 86.03, 90.02 and 89.49^{o}C when Pre- treated at 230^{o}C/30, 230^{o}C/60, 260^{o}C/30, 260^{o}C/60min., respectively (Figure 19$_{a\text{-}d}$). The endothermic peak implies the dehydration (moisture removal) of pre-treated sugar cane dry and top composite. Whereas at a torrefaction temperature of 290/30 and 290/60 in (Figure 19$_{e\ \&f}$), the peak disappears as the moisture completely removed from the sample. The observed exothermic peak at a temperature of 363.83, 368.19, 475.87, 367.28^{o}C was due to devolatilisation and degradation of hemicelluloses and cellulose (Kumar et al., 2022). The rest exothermic peak at a temperature 495.78, 501.08, 484.30.496.87, 503.98 indicates the heat released during char formation (Dhaundiyal et al., 2020). From (Figure 19$_{a\&\ b}$) the exothermic peak increases as the temperature of the system increases from 363.83 to 501.08 ^{o}C, on the because of the release of heat, and also the temperature in increasing order (475.87 to 484.30 ^{o}C) in the figure 19$_{c\&\ d}$ as that of the peak. This phenomenon was also true for Figure 19$_{e\&f.}$ This is due to the decomposition of the pretreated sample. On the contrary the endothermic peak found on the fire stage (Figure 19$_{a,\ b\ \&\ c}$) with its smaller peak and temperature between (82.93, and 90.02). Finally the temperature drops to 86.03 (Figure 19$_{d}$). As the torrefaction temperature and residence time increases, there was no any peak observed in the Figure 19$_{e\ \&\ f.}$ In the case of the second endothermic peak, there was no large temperature difference seen.

4.9. Experimental Results of Produced Briquette

Based on the design expert software 30 experimental run was made. After that, the actual bulk density and shattering resistance were calculated (Table 13).

4.9.1 Response surface methodology (RSM) analysis

The 30 experimental run shown in Table 13 was carried out according to the design expert. By applying multiple regressions analysis on the experimental data, second degree polynomial equation was found to present the relationship between the response variables (bulk density and shatter resistance) of the briquette with factors pressure, dwell time and die length.

$$Bulk\ density = +0.67 + 0.060 * A + 0.082 * B - 0.044 * C + 0.019 * A * B - 0.023 * AC + 6.475E - 0.03 * B * C - 0.026 * A2 - 0.045 * B2 - 2.75E - 0.03 * C2 \quad (21)$$

$$Shatter\ resistance = + 96.63 + 0.95 * A + 1.86 * B - 0.49 * C - 0.29 * A * B - 0.29 * A * C + 0.50 * B * C + 0.38 * A2 - 0.57 * B2 + 0.012 * C2 \quad (22)$$

Table 13: The 30 experimental run made using the factors pressure, dwell time and die length and response variables (bulk density and shatter) calculated using equation (21 and 22).

Run	Parameters			Responses	
	Pressure (Mpa)	Dwell time (min)	Die length (mm)	Bulk density (g/cm3)	Shattering resistance (%)
1	8	3	40	0.56	95.52
2	8	5	40	0.60	96.54
3	8	7	40	0.61	96.76
4	8	3	50	0.52	94.18
5	8	5	50	0.57	95.57
6	8	7	50	0.62	96.79
7	8	3	60	0.50	92.30
8	8	5	60	0.55	96.51
9	8	7	60	0.63	97.75
10	10	3	40	0.60	95.64
11	10	5	40	0.72	97.21
12	10	7	40	0.77	98.52
13	10	3	50	0.57	95.32
14	10	5	50	0.73	97.07
15	10	7	50	0.75	98.56
16	10	3	60	0.51	94.51
17	10	5	60	0.70	96.64
18	10	7	60	0.72	97.50
19	12	3	40	0.70	98.57
20	12	5	40	0.86	99.74
21	12	7	40	0.89	99.86
22	12	3	50	0.61	96.75
23	12	5	50	0.75	98.77
24	12	7	50	0.78	98.87
25	12	3	60	0.59	96.45
26	12	5	60	0.72	97.51
27	12	7	60	0.75	97.89

The significance of the fit of the second degree polynomial for the bulk density and shattering resistance was assessed by carrying out analysis of variance (ANOVA) with results shown in Table 14.

Table 14: Analysis of variance for bulk density and shattering resistance.

Source	df	Mean Square	
		Bulk density	Shattering resistance
Model	9	0.031*	8.18*
A-pressure	1	*0.028**	*7.11**
B-Dwell time	1	*0.049**	*24.94**
C-Die length	1	*2.920E-003**	*0.37*^{ns}
AB	1	*4.408E-003**	*0.99**
AC	1	*6.614E-003**	*1.09**
BC	1	*4.475E-004ns*	*2.72**
A^2	1	*4.093E-003**	*0.89**
B^2	1	*0.014**	*2.25**
C^2	1	*4.802E-005*^{ns}	*8.869E-004*^{ns}
CV %		4.39	0.52
R-Squared		0.9428	0.9346
Adj R-Squared		0.9170	0.9051
Pred R-Squared		0.8540	0.8164
Standard deviation		0.029	0.51

Remark:*indicates the effect of the specific factor has a significant effect on the specified Response variable at alpha = 5%, ns indicates insignificant terms at some alpha value's = Coefficient of variation, R^2 = coefficient of determination.

The coefficient of determination (R^2) of the model was 0.9428 and 0.9346 (Table 14), which indicate that the model adequately represented the real relationship between the variables under consideration. An R^2 value of 94.28% and 93.46% means the variability was highly explained by model. Furthermore the pre R square of 0.854 and 0.8164 are in reasonable agreement with the Adj R- square of 0.917 and 0.9051, for bulk density and shattering resistance respectively. The Adj R- square of 91.7% and 90.51% variability implies, the model can be used to predict, bulk density and shattering resistance of the briquettes .respectively (Tadesse, 2018). The coefficient of variation obtained were 4.39% and 0.52%, which indicates the degree of precision with which the treatments' were carried out. A low value of C.V suggests a high reliability of the experiment. Adequate precision value of 23.20 and 21.23 measures the signal to noise ratio greater than 4 is generally desirable (Belay, 2014). From the regression Bulk density and shattering model, A, B, AB, AC, A^2, and B^2 and A, B, BC, and B^2 are significant terms with probability of 95% (Table14).

4.10. Analysis Result of Optimization for Briquetting

4.10.1 Effect of pressure and dwell time on density of briquette

Density as physical property of the briquette is defined as structural packing of the molecules of the substance in a given volume. Simultaneous increase in Pressure and dwell time

increases the bulk density of the briquette (Figure 20). The increase in the bulk density of briquette with increase in compaction pressure was due to the reduction in empty space during compaction, and improving the cohesion between the compacted particles (Portilho et al., 2020). The density of briquette with die length of 60mm, varies from 0.56 – 0.89g/cm^3 with the increase in dwell time from 3-7 minute, and pressure from 8 -12 Mpa (Figure 20). The bulk density obtained in this work was slightly match with density of briquette reported at a pressure of 10 Mpa, which is 0.932g/cm^3 by (Helwani et al., 2018). Similarly in agreement with this finding ,Studies have also shown that an increase in dwell time leads to an increase in the dry bulk density of biomass briquettes (Belay, 2014).

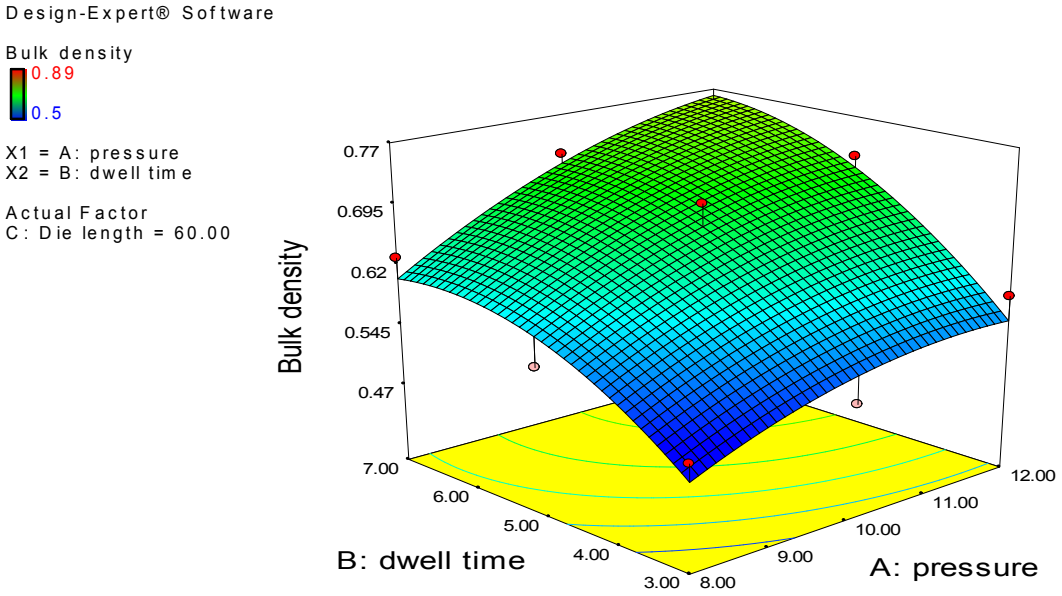

Figure 20: 3D response surface plot of pressure and dwell time on density of briquette

4.10.2 Effect of pressure and dwell time on shatter resistance of briquettes

The durability of torrefied sugarcane dry and top composite were measured after briquettes produced. Figure 21 showed that the mechanical Interlocking as well as the increased adhesion between the particles and formation of intermolecular bonds in the contact area enhances when the pressures increase from 8-12Mpa and dwelling times changes from 3 to 7 minutes. The result obtained agrees with (J. S. Tumuluru et al.,2015), in which the strength of briquettes increases as compression pressure increases from 7.5 to 12.5 MPa. At the optimized pressure (12 Mpa), the shattering resistance increased from 95% to 99.85 % as dwelling time increased from 3 to 7 min (Table 13). This could be as a result of adequate compacting period

and the presence of sufficient lignin content that are being melted at the die temperature (130^{o}C), resulting in the binding of the particles due to die temperature (130^{o}C) (Acharya et al., 2015). Similar result reported by Asisdiq, indicates that the increase in time and temperature factors increase shatter resistance (Asisdiq et al., 2017).

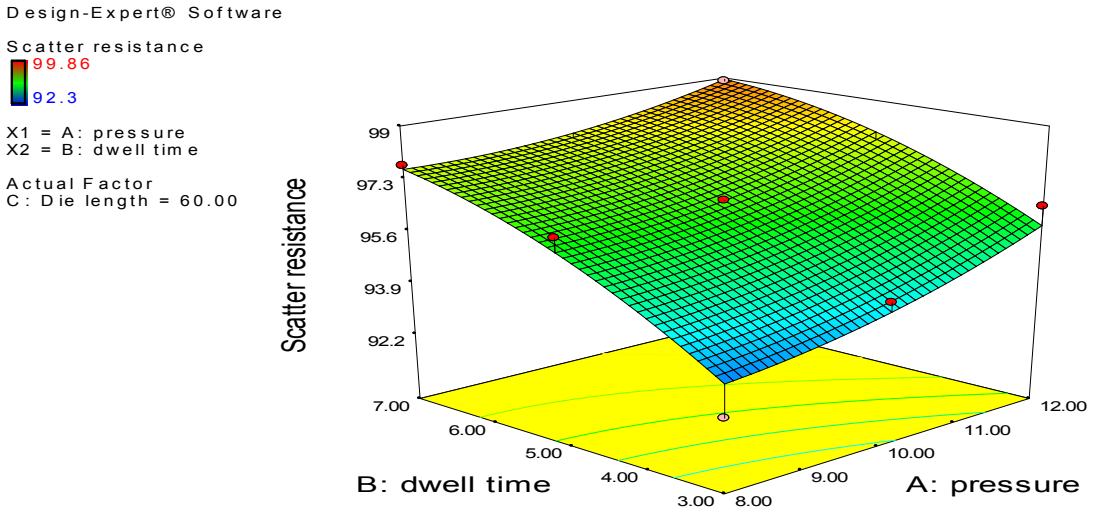

Figure 21: 3D response surface plot of pressure and dwell time on shatter resistance

4.10.3 Effect of die length on density of briquette

This parameter is crucial for the proper construction of compaction devices. It was found that the briquette density and relaxation are influenced by the die height. The highest density(o.89g/cm^3) at maximum compaction pressure (12Mpa) and lowest die height (40mm).As the length of the die increases from 40mm to 60 at constant run time (7 min), the density of the produced briquette reduced from 0.89 g/cm^3 to 0.74 g/cm^3. According to the work of Nurek and Gendek, regardless of the process temperature and moisture, the briquette density increases as the die height is reduced (Nurek et al., 2021).

4.10.4 Calorific value of torrefied briquette

A major indicator of the quality of the briquettes is the calorific value which measures the briquettes energy content. The highest heating value of briquettes produced from torrefied sugar cane dry and top composite at die length 40mm and pressure 12Mpa at 7 minute holding time was found to be 22.04 MJ/kg after densification (Appendix X). The result indicated that the briquette produced from the torrefied composite had higher calorific value after

densification compared the untorrefied briquettes. The increase in calorific value of the briquettes with torrefied sugar cane dry and top composite was mainly due to the degradation or transformation of component such as lignin hemicelluloses to biochar and this leads to the increase in energy density (Portilho et al., 2020).

4.11. Optimized Briquettes Variables and Its Results

Determining the optimum operating condition for torrefied briquettes was aimed to obtain a high quality of solid fuel at a minimum operating cost of production. The optimized operating condition selected by the model for processing briquettes was pressure (12 Mpa), dwell time (6.57 minute) and die length (40mm). The briquette produced at this optimized operation condition fulfilled the requirement of quality briquette solid fuel of bulk density close to 0.86 g/cm^3 and shattering resistance of 99.65% (Table 15).

Table 15: optimization model result of briquetting

Number	pressure	dwell time	Die length	Bulk density	Scatter resistance	Desirability	
1	12.00	6.57	40.00	0.869417	99.6581	0.960	Selected
2	12.00	6.60	40.00	0.869665	99.6529	0.960	
3	12.00	6.53	40.00	0.869038	99.665	0.960	

From the Table 15, when the pressing pressure, dwell time, and die length become 12Mpa, 6.57min, and 40mm respectively, we gate a maximum bulk density and shattering resistance of 0.87 g/cm^3 and 99.66%. Based on these, the optimum briquetting condition implemented in this work was 12Mpa, 7 minute and 40mm die length, which had bulk density and shattering resistance of 0.89 g/cm^3 and 99.87%. The bulk density result highly correlates with wok reported by (Tadesse, 2018, Belay, 2014). By Applying the desirability function (DF) method, the design expert software each dependent variables has DF= 1, as shown in Table 15. However among those, the one that meet validity were 0.960 (Table 15).

Figure 22: optimized briquettes produced.

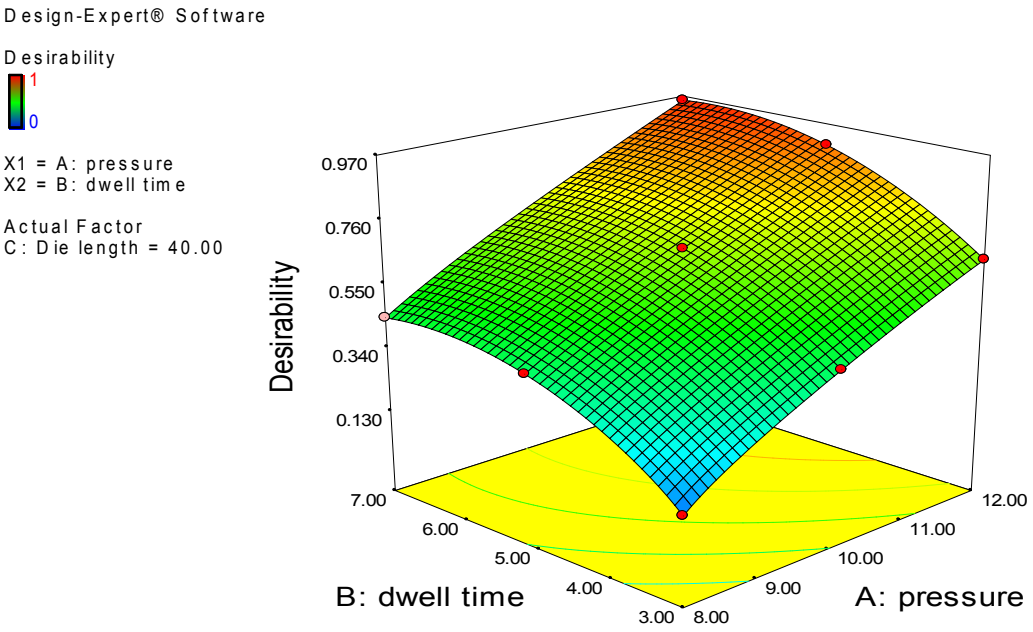

Figure 23: 3D response surface plot for optimum briquetting yield

5. CONCLUSIONS AND RECOMMENDATIONS

5.1. Conclusions

The development of renewable energy sources such as biomass for energy generation is of a particular interest nowadays, as it allows mitigation of greenhouse gases provides means of energy independence and may even offer new employment possibilities. This would also promote appropriate utilization of the biomass instead of discarding it to the environment, which causes adverse effects on human beings and environments. To this end, this study investigates the possibility of developing quality briquette of better energy values from sugar cane dry and top leaves composite biomass using torrefaction pretreatment techniques for optimum energy yields. The torrefaction treatment and subsequent briquetting of sugar cane dry and top leaves composite for optimum energetic yields was carried out with it parametric settings investigated using full- factorial experimental design.

The different analysis results of sugarcane waste composite, torrefied samples and final briquette samples confirmed that:

- The composition of the dry and top leaves revealed that the biomass can be used as one of alternative renewable energy source if proper pretreatments are performed.
- The torrefaction treatments improved the HHV value of dry and top leaves composite sample from 17.07 to 20.03MJ/kg.
- The optimum torrefaction conditions that gave the highest energetic yield (20.03 MJ/kg) are TT of 290^{o}C, RT of 60min and PS of 2mm. The physicochemical composition of sugar cane leave & top composite that contributes to the highest energy yield at the optimized condition might be large as depicted in the FC increase from 5 to 23.36% (Table 8) and obtained from FTIR and TGA analysis.
- Torrefaction temperature has the largest influence on energetic yields (MY, HHV, and energy yield).

- Produce briquette of the required properties from torrefied sugar cane leave & top composite by varying densification pressure, holding time, and length of the die.

- Pressure, die length and dwell time has significant effect on strength of torrefied briquettes.
- The density of briquettes increases with the increase in compaction pressure.
- The optimum briquetting processing variables that gave the highest bulk density are pressure of 12MP, dwell time of 7min and die length of 40mm. At this optimized condition the HHV obtained was 22.04MJ/kg, the bulk density was 0.89g/cm^3 and shattering resistance was 99.86%, which indicated the briquette produced meet the required properties of solid biofuel.

5.2 Recommendations

Considering the promising results obtained from this study, we recommend the following for the future research:

- The economic loss associated to burning of sugarcane leaves and tops in the sugar industry need to be investigated as the research clearly showed it is a valuable resource to be used as alternative energy source if it is properly managed. This is besides of burning of these resource found to have adverse effect on environment and personnel involved during cutting**.**
- Further research should also be conducted on the economic feasibility of producing briquetting at large scale for further use.
- Further study to make use of the waste boiler flue gas obtained during steam production in the sugarcane factory as energy source for torrefaction process in case of industrial scale of manufacturing of briquette from sugarcane dry and top.
- Life cycle assessment for the selected batch should be made to know energy recovery (Mass balance)

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Appendix I: Torrefaction process treatments and their response based on the design.

Run	Factor			Response			
	PS(mm)	**TT(^{O}c)**	**RT(min)**	**M (%)**	**V M (%)**	**FC (%)**	**Ash (%)**
1	6.00	260.00	60.00	2.4	75.98	11.75	9.87
2	4.00	260.00	30.00	3.21	76.57	10.89	9.33
3	4.00	230.00	30.00	3.75	80.1	7.24	8.91
4	2.00	290.00	60.00	1.32	63.12	23.36	12.2
5	4.00	260.00	30.00	3.21	76.57	10.89	9.33
6	4.00	260.00	60.00	2.35	73.06	14.62	9.97
7	2.00	260.00	60.00	2.2	68.17	19.43	10.2
8	4.00	290.00	60.00	1.61	67.7	18.59	12.1
9	4.00	290.00	0.00	6.93	82.18	5	5.89
10	4.00	230.00	60.00	2.97	78.6	9.3	9.13
11	2.00	290.00	0.00	5.75	82.43	5.72	6.1
12	4.00	290.00	30.00	2.15	68.23	18.72	10.9
13	2.00	230.00	60.00	2.87	76.33	11.65	9.15
14	6.00	230.00	0.00	6.45	82.01	4.58	6.96
15	2.00	290.00	30.00	2.05	66.42	21.13	10.4
16	2.00	260.00	0.00	5.75	82.43	5.72	6.1
17	6.00	290.00	30.00	2	71.13	15.37	11.5
18	6.00	230.00	60.00	3.65	80.44	7.3	8.61
19	2.00	260.00	30.00	2.95	74.39	13.21	9.45
20	6.00	260.00	30.00	3.71	77.96	9.74	8.59
21	4.00	260.00	0.00	6.93	82.18	5	5.89
22	6.00	290.00	0.00	6.45	82.01	4.58	6.96
23	6.00	290.00	60.00	1.76	69.96	12.48	15.8
24	4.00	260.00	30.00	3.21	76.57	10.89	9.33
25	4.00	230.00	0.00	6.93	82.18	5	5.75
26	6.00	260.00	0.00	6.45	82.01	4.58	6.96
27	6.00	230.00	30.00	3.92	82.37	6.21	7.5
28	2.00	230.00	30.00	3.55	79.68	7.83	8.94
29	2.00	230.00	0.00	5.75	82.43	5.72	6.1
30	4.00	260.00	30.00	3.21	76.57	10.89	9.33

Where PS= refers to particle size, TT = Torrefaction temperature, RT =Retention time, M = Moisture, VM =Volatile matter, FC =Fixed Carbon

Appendix II: ANOVA Summary for moisture

Source	Mean Squares	df	F-Square	p-value	Prob > F
Model	*94.03*	*9*	*10.45*	*90.72*	*< 0.0001**
A-Particle size	*1.18*	*1*	*1.18*	*10.21*	*0.0045**
B-Temperature	*5.36*	*1*	*5.36*	*46.52*	*< 0.0001**
C-Residence Time	*73.04*	*1*	*73.04*	*634.28*	*< 0.0001**
AB	*0.048*	*1*	*0.048*	*0.42*	*0.5253^{ns}*
AC	*0.039*	*1*	*0.039*	*0.33*	*0.5694^{ns}*
BC	*1.92*	*1*	*1.92*	*16.67*	*0.0006**
A^2	*0.42*	*1*	*0.42*	*3.62*	*0.0715^{ns}*
B^2	*0.069*	*1*	*0.069*	*0.60*	*0.4493^{ns}*
C^2	*12.37*	*1*	*12.37*	*107.45*	*< 0.0001**
Residual	*2.30*	*20*	*0.12*		
Lack of Fit	*2.30*	*17*	*0.14*		
Pure Error	*0.000*	*3*	*0.000*		
Cor Total	*96.33*	*29*			

Remark *= significant, NS = insignificant

Appendix III: ANOVA Summary for volatile matter

Source	df	Mean Square	F Value	p-value Prob > F
Model	9	100.46	38.53	< 0.0001*
A-Particle size	1	45.03	17.27	0.0005*
B-Temperature	1	279.74	107.30	<0.0001*
C-Residence Time	1	415.68	159.45	<0.0001*
AB	1	1.88	0.72	*0.4058*
AC	1	33.40	12.81	0.0019*
BC	1	99.71	38.25	< 0.0001*
A^2	1	0.67	0.26	0.6181^{ns}
B^2	1	2.78	1.07	*0.3137^{ns}*
C^2	1	27.78	10.66	0.0039*
Residual	20	2.61		
Lack of Fit	17	*3.07*		
Pure Error	3	*0.000*		
Cor Total	29			

Appendix IV: ANOVA Summary for Fixed carbon

Source	df	Mean Square	F Value	p-valueProb > F
Model	9	87.99	26.12	< 0.0001
A-Particle size	*1*	*76.80*	*22.80*	*0.0001*
B-Temperature	*1*	*200.80*	*59.61*	*< 0.0001*
C-Residence Time	*1*	*378.86*	*112.46*	*< 0.0001*
AB	*1*	*9.49*	*2.82*	*0.1089*
AC	*1*	*31.66*	*9.40*	*0.0061*
BC	*1*	*57.12*	*16.95*	*0.0005*
A^2	*1*	*0.78*	*0.23*	*0.6356*
B^2	*1*	*0.40*	*0.12*	*0.7350*
C^2	*1*	*37.10*	*11.01*	*0.0034*
Residual	20	3.37		
Lack of Fit	*17*	*3.96*		
Pure Error	*3*	*0.000*		
Cor Total	29			

Appendix V: ANOVA Summary for Ash

Source	Sum of Squares	df	Mean Square	F-Value	p-value Prob > F
Model	141.60	9	15.73	31.24	< 0.0001*
A-Particle size	*0.94*	*1*	*0.94*	*1.86*	0.1874^{ns}
B-Temperature	*24.04*	*1*	*24.04*	*47.72*	*< 0.0001**
C-Residence Time	*90.32*	*1*	*90.32*	*179.31*	*< 0.0001**
AB	*3.72*	*1*	*3.72*	*7.38*	*0.0133**
AC	*1.875E-003*	*1*	*1.875E-003*	*3.722E-003*	0.9520^{ns}
BC	*14.24*	*1*	*14.24*	*28.26*	*< 0.0001**
A^2	*0.38*	*1*	*0.38*	*0.76*	0.3937^{ns}
B^2	*1.63*	*1*	*1.63*	*3.24*	0.0869^{ns}
C^2	*7.40*	*1*	*7.40*	*14.69*	*0.0010**
Residual	10.07	20	0.50		
Lack of Fit	*10.07*	*17*	*0.59*		
Pure Error	*0.000*	*3*	*0.000*		
Cor Total	151.68	29			

Appendix VI: ANOVA for Scatter resistance

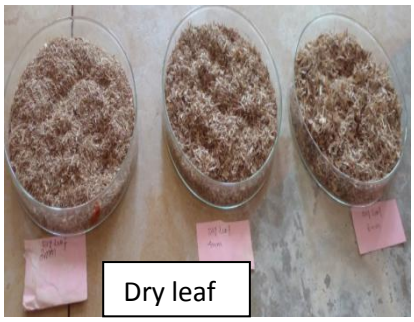
Source	Sum of Squares	df	Mean Square	F -Value	p-value Prob > F
Model	73.59	9	8.18	31.7	< 0.0001
A-pressure	*7.11*	*1*	*7.11*	*27.58*	*< 0.0001*
B-dwell time	*24.94*	*1*	*24.94*	*96.82*	*< 0.0001*
C-Die length	*0.37*	*1*	*0.37*	*1.42*	*0.2467*
AB	*0.99*	*1*	*0.99*	*3.85*	*0.0638*
AC	*1.09*	*1*	*1.09*	*4.25*	*0.0525*
BC	*2.72*	*1*	*2.72*	*10.55*	*0.0040*
A^2	*0.89*	*1*	*0.89*	*3.45*	*0.0780*
B^2	*2.25*	*1*	*2.25*	*8.72*	*0.0079*
C^2	*8.869E-004*	*1*	*8.869E-004*	*3.443E-003*	*0.9538*

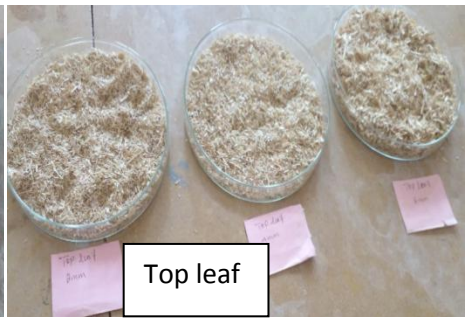
Appendix VII: ANOVA for Bulk density

source	Sum of Squares	df	Mean Square	F -value	p-value Prob > F
Model	0.28	9	0.031	36.60	< 0.0001
A-pressure	*0.028*	*1*	*0.028*	*32.69*	*< 0.0001*
B-dwell time	*0.049*	*1*	*0.049*	*56.77*	*< 0.0001*
C-Die length	*2.920E-003*	*1*	*2.920E-003*	*3.41*	*0.0798*
AB	*4.408E-003*	*1*	*4.408E-003*	*5.14*	*0.0346*
AC	*6.614E-003*	*1*	*6.614E-003*	*7.71*	*0.0116*
BC	*4.475E-004*	*1*	*4.475E-004*	*0.52*	*0.4784*
A^2	*4.093E-003*	*1*	*4.093E-003*	*4.77*	*0.0410*
B^2	*0.014*	*1*	*0.014*	*16.72*	*0.0006*
C^2	*4.802E-005*	*1*	*4.802E-005*	*0.056*	*0.8153*

Appendix IX: Experimental pictures

Milling **Particle Size separation using sieve shaker**





Different particle size 2, 4 and 6mm dry and top leaves

Prepared 2,4and 6mm to be blend based on grain size

Mixing dry and tops based on their in 60 :40 ratio



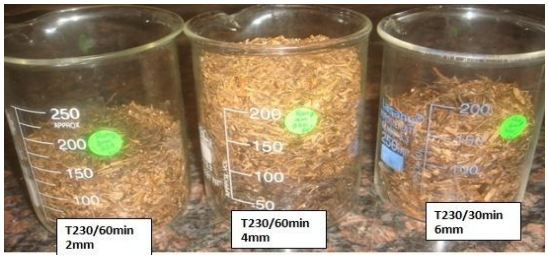
Refluxing process for Extractive washing an filtration after reflux

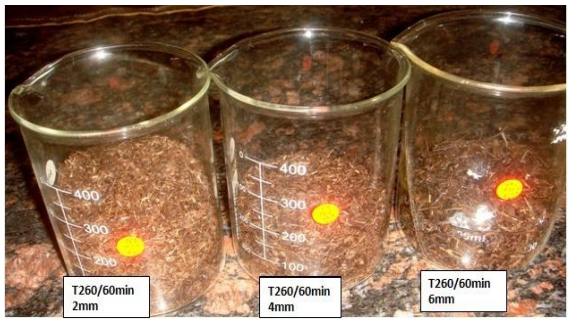


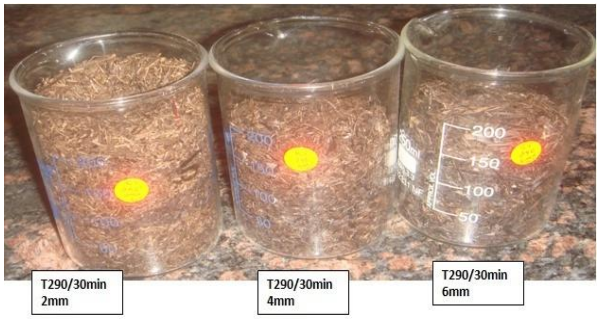
Preservation for hemicelluloses analysis refluxing & filtration process for the preserved sample

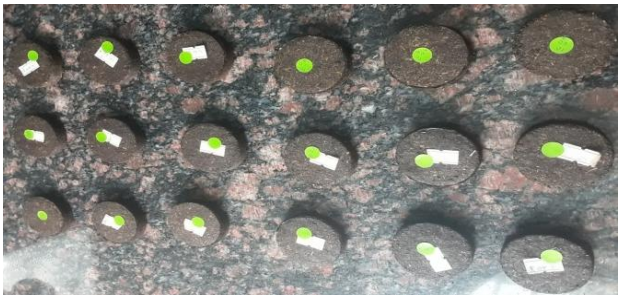

Fixed carbon, volatile matter ash analysis

Sugarcane dry and top leaves composite sample torrefied at different (particle size, residence time and set temperature.









Briquettes produced

Appendix X: Tabulated experimental results

Research & Training Division
Laboratory service

Caloric Value Analytical Result

Experiment title : Application of Torrefaction for the Production of quality Briquette from sugar cane leaves and top composite as alternative energy source.

S/N	Lab code	Sample type	Gross Caloric Value (MJ/Kg)	Avarage HHV(MJ/Kg)
1	001/22	Composite	17.07	
2	002	TT230/30	17.35	
3	003	TT230/60	17.89	
4	004	TT260/30	17.50	
5	005	TT260/60	18.62	
6	006	TT290/30	19.07	
7	007	TT290/60	20.03	
8	008	Sugar cane top	16.83	
9	009	Sugar cane Dry	16.88	
10	010	Torrefied Briquette-1	22.06	22.04
11	011	Torrefied Briquette-2	22.02	
12	012	Torrefied Briquette-3	22.05	

Conducted by: Fisseha Tarekegn (Chemist -II) Sign ____ Date 17/01/23

Approved by: Tilahun Zeleke Laboratory Service Head Sign ____ Date 17/01/23

Proximate analysis result of sugarcane dry and top composite the control (Un torrified) and torrified at (230, 260 and 290^{o}C), particle size (2, 4 and 6mm) and dwelling time (30 and 60 minute).

TT (^{O}C) / Time (min.)	Particle size											
	2mm				4mm				6mm			
	M%	V.M %	F.C %	ASH %	M %	V.M %	F.C %	ASH %	M%	V.M %	F.C %	ASH %
Un torrified	5.75	82.43	5.72	6.10	6.93	82.18	5.00	5.89	6.45	82.01	4.58	6.96
TT t- 230/30	3.55	79.68	7.83	8.94	3.75	80.10	7.24	8.91	3.92	82.37	6.21	7.50
TTt-230/60	2.87	76.33	11.65	9.15	2.97	78.60	9.30	9.13	3.65	80.44	7.30	8.61
TT t -260/30	2.95	74.39	13.21	9.45	3.21	76.57	10.89	9.33	3.71	77.96	9.74	8.59
TT t-260/60	2.20	68.17	19.43	10.20	2.35	73.06	14.62	9.97	2.40	75.98	11.75	9.87
TT t-290/30	2.05	66.42	21.13	10.40	2.15	68.23	18.72	10.90	2.00	71.13	15.37	11.50
TTt-290/60	1.32	63.12	23.36	12.20	1.61	67.70	18.59	12.10	1.76	69.96	12.48	15.80

Mass yield, HHV, Energy yield, and Energy density of raw and torrefied at (230 °C, 260 °C, and 290°C) composite sample of particle size 2mm, 4mm, and 6mm and for holding time (30,60min).

TT (°C) / RT (min.)	Particle size					
	2mm	4mm	6mm	2mm		
	Mass Yield %			HHV(MJ/kg)	Energy yield (W %)	Energy Density
Raw				17.07		
TT /t-230/30	82.53	84.21	91.62	17.35	83.36	1.01
TT/ t-230/60	82.72	80.31	80.99	17.89	86.86	1.05
TT/t-260/30	83.34	79.93	86.47	17.50	85.84	1.03
TT/t-260/60	74.04	77.68	79.86	18.63	80.70	1.09
TT/t290/30	71.84	66.02	64.93	19.07	79.74	1.11
TT/t-290/60	55.9	57.9	61.41	20.03	65.40	1.17

The 30 Experimental run made using the factors pressure, dwell time and die length and response variables (bulk density and shatter) calculated using equation (21 and 22).

Run	Factors			Responses	
	pressure (Mpa)	Dwell time (min)	Die length(mm)	Bulk density kg/cm3	Shattering resistance %
1	10.00	7.00	50.00	0.75	98.56
2	8.00	5.00	60.00	0.55	96.51
3	10.00	3.00	60.00	0.51	94.51
4	10.00	5.00	50.00	0.73	97.07
5	8.00	7.00	50.00	0.62	96.79
6	10.00	3.00	40.00	0.6	95.64
7	8.00	5.00	40.00	0.6	96.54
8	12.00	5.00	50.00	0.75	98.77
9	12.00	7.00	40.00	0.89	99.86
10	10.00	7.00	50.00	0.75	98.56
11	10.00	5.00	50.00	0.73	97.07
12	10.00	5.00	60.00	0.7	96.64
13	8.00	7.00	40.00	0.61	96.76
14	12.00	3.00	50.00	0.61	96.75
15	8.00	5.00	60.00	0.55	96.51
16	12.00	5.00	40.00	0.86	99.74
17	8.00	3.00	50.00	0.52	94.18
18	12.00	7.00	60.00	0.74	98.89
19	10.00	5.00	40.00	0.72	97.21
20	8.00	3.00	60.00	0.5	92.3
21	10.00	5.00	50.00	0.73	97.07
22	10.00	7.00	60.00	0.72	97.5
23	12.00	3.00	40.00	0.7	98.57
24	12.00	3.00	60.00	0.59	96.45
25	10.00	5.00	50.00	0.73	97.07
26	12.00	5.00	60.00	0.72	97.51
27	8.00	3.00	40.00	0.56	95.52
28	10.00	3.00	50.00	0.57	95.32
29	12.00	7.00	50.00	0.78	98.87
30	8.00	7.00	60.00	0.63	97.75