Addis Ababa University

Addis Ababa Institute of Technology

School of Chemical and Bio Engineering

ENHANCING THE PHYSICAL AND COMBUSTION PROPERTIES OF SAWDUST BRIQUETTES VIA HYDROTHERMAL PRE-TREATMENT AND MOLASSES BINDER

By

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Declaration

I declare that this thesis entitled "***Enhancing the Physical and Combustion Properties of Sawdust Briquettes Via Hydrothermal Pre-Treatment and Molasses binder***" is my own original work done under the supervision of Dr. Sintayehu Nibret at School of Chemical and Bio engineering in Addis Ababa Institute of Technology, Addis Ababa University and I have not previously submitted it entirely or in part for obtaining any qualification at any other university.

Amanuel Mengistu ______________________

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Acronyms

AC	Ash content
ANOVA	Analysis of variance
ASTM	American standard test method
CI	Confidence interval
DOE	Design of experiment
FC	Fixed carbon
HHV	Higher heating value
HPT	Hydrothermal pre treatment
kJ	Kilo joules
m^3	Meter cube
MC	Moisture content
MPa	Mega pascal
T	Temperature
VIF	Variance inflation factor
VM	Volatile matter
Wb	Wet basis
wt%	Weight percent

Abstract

Renewable alternative energy sources have become more popular in recent years and biomass is one of them. Biomass is used as an alternative energy resource and available in huge quantity. Wood is a primary energy source widely used for domestic consumption all over the world. Sawdust of Cupressus Lusitanica is mostly available in various wood workshops throughout Addis Ababa, Ethiopia. The main objective of this study is to enhance the physical and combustion properties of sawdust briquettes through hydrothermal pretreatment (HPT) of the raw biomass and addition of molasses as a binding agent. The effects of HPT temperature (taken at 240°C, 280°C, 320°C) and molasses wt% (0%, 10%, 20%) on the physical and combustion properties of the resulted briquettes prepared from the sawdust of Cupressus Lusitanica with particle size range(1mm-0.5mm) were studied. Dwell time of 45sec and briquetting pressure of 10MPa were kept constant during briquette production. The density, moisture content (MC), volatile matter (VM), ash content (AC), fixed carbon (FC), and high heating value (HHV) of the resulted briquettes were investigated and compared with raw sawdust and briquettes from other studies. This study revealed that the physical and combustion properties of biomass briquettes can be improved by hydrothermal pretreatment of the raw biomass whereas binder addition only improves the mass density of the briquettes at the expense of their combustion properties. Hydrothermal treatment was beneficial to the physical properties of the resulted biomass briquettes; density and durability of the untreated briquettes B_{00}(HPT = none, binder wt% = 0%), B_{01}(HPT = None, binder wt% = 10%), B_{02}(HPT = none, binder wt% = 20%) were lower compared to the HPT briquettes. The optimum operating conditions were determined to be HPT = 320^{O}C and binder wt% = 0%; the response variables at this optimum are as follows: density = 747.47kg/m^3, durability = 99.99, MC = 9.61%, VM = 52.2%, AC = 2.87%, FC = 35.4%, HHV = 20.74 MJ/Kg.

1. Introduction

1.1 Background

The rising price of fossil fuel and environmental concern encourage the exploration of alternative energy sources that are renewable. Solar energy in its direct form, wind, geothermal, water, and biomass are all examples of renewable energy. Biomass, which is the generic term for organic waste, has gained the most traction among them. This is due to its availability and very inexpensive energy conversion. Another advantage of biomass over other alternative energy sources is the fact that utilization of biomass resources can solve the disposal problems caused by the large amount of agricultural and forestry waste annually and eliminate the potential for environmental pollution. In addition, biomass energy gives off zero net carbon emission since biomass consumes the same amount of CO_2 from the atmosphere during growth as is released during combustion [1].

There is a high demand for biomass energy. It has been estimated that more than 2.4 billion people, generally among the world's poorest, rely directly upon biomass, e.g. wood, crop residues, dung and other biomass fuels for their heating and cooking needs[2].In Ethiopia, the national electrification rate is around 25%;being 85% in the urban areas and 10% in the rural areas. So, it is expected that Ethiopia will depend on biomass as a primary energy source for quite a number of years to come[3].

When compared to other biomass sources, wood from commercial forest land has the most potential. The majority of this potential is found in byproducts of wood processing such as sawdust and other residues of thinning and logging operations. Sawdust is composed of small chippings of wood obtained from forest wastes or manufacturing wastes. However, directly combusting such kind of loose biomass have disadvantages such as difficulty to transport, high risk of fermentation and spontaneous combustion, low caloric value and energy density, and emission of toxic air pollutants (PAH, PM and NOx) into the atmosphere due to incomplete combustion[4].

Therefore, it is essential to convert sawdust into cleaner and higher caloric value renewable bio-energy. Briquetting (biomass briquetting specifically) is the process of compacting loose biomass materials into rigid, densely packed products[5]. Briquettes can either be formed under high pressure without binders or under low pressure using binders.

Hydrothermal pretreatment (HPT) is the process of heating biomass with hot compressed subcritical water in an oxygen free environment for the purpose of breaking down macromolecules and lowering volatile matter of the biomass [6].

Compared with traditional techniques such as biological process and pyrolysis, HPT process can be directly applied to biomass with high moisture. It has been used successfully in converting high moisture content biomass, including microalgae, sewage sludge, and food waste, into a lignite-like solid product (hydrochar). Compared to raw biomass, hydrothermally treated biomass is highly hydrophobic, more homogeneous and energy-dense and briquettes formed from this pretreated biomass are of a superior quality[7].

1.2 Statement of the problem

There is a current global need for clean and renewable energy source. Fossil fuels are nonrenewable and come from finite sources, which are dwindling because of high cost and environmentally damaging retrieval techniques. In order to achieve better results in terms of energy cost and efficiency, the supply of new and alternative energy sources has become a necessity. Briquetting loose biomass provides a good energy source as well as an environmentally beneficial combustible fuel.

Excellent mechanical strength is achieved with greater compressive pressure. However, a compression technique that uses high pressure consumes more energy. According to Jaya Shankar et al, high pressure and temperature during densification can create solid bridges by merging one particle to another at the point of contact; it is also stated that this method requires higher capital and operating expenditures.

Whereas low pressure densification requires binders to increase physical and mechanical properties of the briquettes. However, binder addition tends to lower the proximate and combustion properties of the briquettes. M. Muluken showed that by increasing pulp binder from 0% to 20 wt%, the density of the resulting briquettes was increased from 218.24to 322.62kg/m^3 while the HHV decreased from 18.38to 14.50MJ/kg. Another major drawback of compacting raw biomass is that it requires drying unit operation which is energy intensive and needs long retention time thereby increasing production cost and acting as a bottle neck of the entire briquette production process.

In order to address the aforementioned drawbacks, Y. Zhai et al attempted to produce hydrochar briquettes from food waste under varying hydrothermal pretreatment (HPT) conditions, and was pelletized using molasses and molasses/lime binders for fuel pellet production. The results indicated that pellets produced from HPT biomass had better physical and thermal properties. The compressive strength and impact resistance index of hydrochar samples produced at 230°C and 260°C exhibited superior physical properties. Prior to pelletization, molasses and lime were added resulting in further improvement in durability and density while diminishing combustion characteristics. The molasses pellets exhibited lower ignition temperature and a longer combustion period than the molasses/lime pellets.

The above-mentioned critical limitations of fossil fuels and less efficient conventional biomass briquette manufacturing techniques accompanied by lack of research on HPT as a viable option for sawdust briquette production highlight the need for this research.

1.3 Objective

1.3.1 General objective

The main objective of this study is to improve physical and combustion properties of sawdust briquettes through hydrothermal pretreatment of the biomass and addition of molasses as a binding agent.

1.3.2 Specific objectives

The specific objectives of this thesis work are to:

i. Investigate the effects of HPT and molasses binder on the physical and combustion properties of sawdust briquettes.
ii. Select optimum operating parameters for the production of sawdust briquettes
iii. Enhance physical and combustion properties of sawdust briquettes.

1.4 Significance of the study

HPT addresses the issue of longer processing times due to drying unit operation and low durability observed in biomass briquette. HPT of biomass would result in low cost and superior quality briquettes which will compete with conventional wood charcoal in the market place thereby combating deforestation. Since HPT of raw biomass could address both problems mentioned above, this study can be a facile and potential way to produce cheaper and superior quality briquettes.

1.5 Scope of the study

This study explores the possibility of producing better quality biomass briquettes from hydrothermally pretreated sawdust feedstock while using molasses as a binder and compares the physical and proximate results with conventional briquettes of similar feedstock.

This work is limited to studying the effects varying the HPT temperature and molasses binder wt% on the physical and combustion properties of the produced briquettes. However, other process variables such as: particle size distribution, feed stock/ raw material, moisture content, binder type, briquetting pressure, retention time and die geometry were kept constant.

2. Literature review

2.1 Conventional Biomass Fuel Briquette Production Methods

Briquetting is the process of densifying loose biomass such as charcoal, sawdust, peat, or paper utilized for fuel. Briquettes can be manufactured with or without the use of binder. Densification without a binder is more convenient, but it necessitates the use of sophisticated and expensive presses and drying equipment, which raises initial capital investment cost.

The briquette manufacturing process varies widely depending on the type of feed biomass and briquetting machine used. Generally, the process has the following basic unit operations: drying, size reduction, sieving, mixing with binder, and densification. Pellets are densified products with diameters and lengths ranging from 3 to 12mm and 6 to 25mm, respectively, whereas briquettes have dimensions in the range of 25 to 100mm diameter and 50 to over 100mm length [9].

According to L. Wamukonya et al., in order for briquette manufacturer to succeed in less industrialized countries, the machinery should be locally built, simple, and low-cost in order to minimize dependency on foreign currency [10].

Biomass densification employs a variety of processes to convert biomass leftovers into a usable fuel. Based on the type of equipment used, briquetting technologies can be classified into five.

I. *Piston press densification*

Raw material is supplied into a cylinder, which is subsequently pressed by a piston against a die in a discontinuous manner. As the compressed material is forced through the die, frictional forces heat it up. The diameter of the briquette is proportional to the machine's production[11].

There are two types of piston press:

a. Die and Punch Technology, also known as ram and die technology, involves punching biomass into a die with a reciprocating ram at high pressure, compressing the mass and resulting in a densified product.
b. Hydraulic press: the hydraulic press compacts the biomass in both vertical and horizontal directions. [12].

II. Screw press densification

The material is continuously supplied through a screw, which drives it into a heated cylindrical die, in order to induce lignin flow. Unlike the impact of a piston, pressure builds up slowly along the screw. The briquettes formed by this technology have a carbonized outer surface and a hole in the center. [13],[14].

III. Roll press densification

In a briquetting roller press, the feedstock falls in between two rollers, rotating in opposite directions and is compacted into pillow-shaped briquettes. This type is used for briquetting carbonized biomass to produce charcoal briquettes.

IV. Pelletizers

Pelletizing is similar to briquetting, except that smaller dies are used (approximately 30 mm). The pelletizer has a number of dies that are placed as holes in thick steel or a ring, and the material is driven into the dies by two or three rollers. Pelletizers create cylindrical briquettes with a diameter of 5mm to 30mm. Pellets are an ideal fuel for industrial applications that require automatic feeding. Pelletizers can generate up to 1000kg of pellets per hour, however they require a large initial capital investment and a lot of electricity.[15].

V. Low pressure or manual presses

There are different types of manual presses used for briquetting biomass feedstocks. They are specifically designed for the purpose or adapted from existing implements used for other purposes. Clay brick making presses are a good example. Low-pressure briquetting techniques are ideal for briquetting plant waste like bagasse and sawdust. The resulting briquette is denser than the original material, but it has limited mechanical strength and crumbles easily, necessitating the application of a binder. [12].

2.2 Experimental methods

Physical and combustion properties of the formed briquettes depend on feed/raw material properties like moisture content, particle size, shape, distribution, binder type, and ratio. Besides feed biomass properties, the quality of the final product also depends on various process variables like temperature, pressure, retention or hold time, die geometry and speed. The effects of one or combination of these factors have been studied to determine the optimal point for each process variable to manufacture briquettes having better physio-chemical properties.

Youndrie et al. used pressure (3000,5000 and 7000 psi), particle size (20,40, and 60mesh) and moisture content (5,10, and 15%) as independent variables[16]. Youndrie employed Box-Behnken design experimental method and used relaxation density and durability as the response variables. Then, he found the optimal point at a pressure of 3002.8psi, particle size of 34.9 meshes and raw material water content of 7.79% which gave a maximum density of 1.0 g/cm^3.

Y. Song et al. densified four biomass samples using laboratory hydraulic press briquetting machine under three pressure levels (7.5, 10, 12.5 MPa), three levels of temperature (90, 110, 130ºC), three moisture content levels (9, 12, 15% w.b), and three levels of particle size (19.1, 25.04, 31.75 mm) at dwell time of about 30s[17]. The response variables were density, durability, dimensional stability, and moisture content. The experiments were designed using the Box-Benken method. Results of the analysis indicated that moisture content plays a significant role in briquettes' durability, stability, and density. At high pressure (12.5 MPa) and low moisture (9-12%), compact briquettes with high density were obtained.

2.3 Comparative Study based on the Type of Feed Material

P. Jittabut examined the physical and thermal properties of briquettes of rice straw and sugarcane leaves with molasses as a binder. Species density, compressive strength, and moisture content were the physical parameters investigated. Results revealed fixed carbon in the range of 9.06- 13.63 percent, volatile matter in the range of 68.14-74.67 percent, ash content in the range of 7.84-12.85 percent, and moisture content in the range of 4.2-6.2 percent [18].

The ignition time and water boiling test of coal briquette blends containing Pennisetum Purpureum (elephant grass) and Imperata cylindrical (spear grass) were compared by T.U. Onnegbu et al. [19]. Using cassava starch as a binder and calcium carbonate (Ca (OH)2) as a desulfurizing agent, several samples of briquettes were made by combining differing loads of plant materials with coal in the ratios of 0:100, 10:90, 20:80, 30:70, 4:60, 50:50, and 100:0. Ignition time was reduced as wt % of plant material increases, although coal blends including Pennisetum purpureum fared better. Burning rates as well as particular fuel consumptions were determined during the water boiling test. The recorded boiling time values were 11.43±0.43 min (briquettes), 14.94±0.22min (charcoal), 9.25±0.42 min (firewood), and 8.99±0.22 min (mangrove).

2.4 Comparative Study based on Physical and Proximate Properties

S. Wu et al. prepared a charcoal briquette from waste cotton stalk (CS) biomass samples pretreated by two different thermal methods; dry torrefaction (DT) and HPT at 200, 230, and 260 °C. The intermediate samples were then densified and carbonized at 400 °C to prepare charcoal briquette without any binders[20]. Test results indicated that the physical properties including the mass densities and compressive strengths of charcoal briquettes with HPT are better than DT based briquettes and unpretreated charcoal briquettes, even those of the commercial barbecue charcoal with binder addition. Charcoal briquettes using HPT exhibited a similar combustion character index (S) with the commercial barbecue charcoal and hydrothermally pretreated CS at 230 °C was the best material for the charcoal briquette preparation process.

J.T. Oladeji et al. investigated the properties of briquettes made from corncobs and rice husk residues [21]. Corncob briquettes had lower relaxation ratio of 1.70 and a moderate moisture content of 13.47 percent. Other positive attributes of corncob briquette over rice husk were long after glow time of 370 seconds and slow propagation rate of 0.12 cm/s. Furthermore, it has a higher volatile matter percentage and HHV of 86.53 and higher of 20,890 kJ/kg than the rice husk, which has 67.98 percent, 13,389 kJ/kg correspondingly.

The calorific values, ignition time, burning rate, specific fuel consumption, fuel efficiency, and water boiling time of biomass briquettes were evaluated by R. M. Davis et al. [22]. Water hyacinth briquettes and plantain peel were utilized as binders in the study. Briquettes were compared to mangrove wood, charcoal, and Anthronotha macrophylla. The thermal fuel efficiency of the briquettes made from this densification variable outperformed charcoal, firewood, and red mangrove wood, according to the findings. Furthermore, charcoal had the highest fuel efficiency (31.29 %), followed by fuel briquettes (28.17%). The calorific values ranged from 4166.67 kcal/kg (firewood) to 6552 kcal/kg (charcoal). The caloric values of the different biomass varied greatly. The water boiling time, ignition time, and burning rate of the various energy sources had significant variation. Briquettes took 11.43 minutes to boil, charcoal took 14.94 minutes to boil, firewood took 9.25 minutes to boil, and wood took 8.99 minutes to boil (mangrove).

The studies referenced above do reveal that worldwide there has been limited research conducted on the application of hydrothermal pretreatment in improving the production process and quality of the end products.

According to the papers cited above, despite various advantages of HPT only limited research has been done on its application to improve the manufacturing process and product quality of biomass briquettes.

3. Materials and Methods

3.1 Materials

Sawdust of *Cupressus Lusitanica* from a local wood workshop was used as the main raw material for the manufacture of the briquettes. Molasses was used as a binding agent to increase the durability of the final product. Sawdust of 0.75mm particle size was separated using a sieve shaker. HPT was carried out inside an autoclave by adding distilled water and sieved sawdust; the autoclave was purged using nitrogen gas to create an inert environment. Densification of loose biomass was carried out using hydraulic piston press. Caliper was used to measure the dimensions of the briquettes formed while dryer was used to remove moisture from the samples. Furnace was used in the determination of the AC and VM of the as prepared briquettes and bomb calorimeter was used to measure the HHV of the samples.

3.2 Experimental Framework of the Study

The experiments were planned according to the frame work shown in figure 1. This study has two major parts: briquette production and characterization of the feed biomass and briquette. Initially, molasses and sawdust of *Cuperessus Lusitanica* were collected. Physical and combustion properties of the sawdust were determined. Afterwards, portion of the remaining sawdust was sampled and mixed with binder while the rest was mixed with distilled water and placed in the autoclave to undergo HPT. Some of the pretreated biomass was mixed with varying amounts of molasses and briquetted while the remaining were densified without the use of binder. Finally, physical and combustion properties of the sawdust briquettes were investigated.

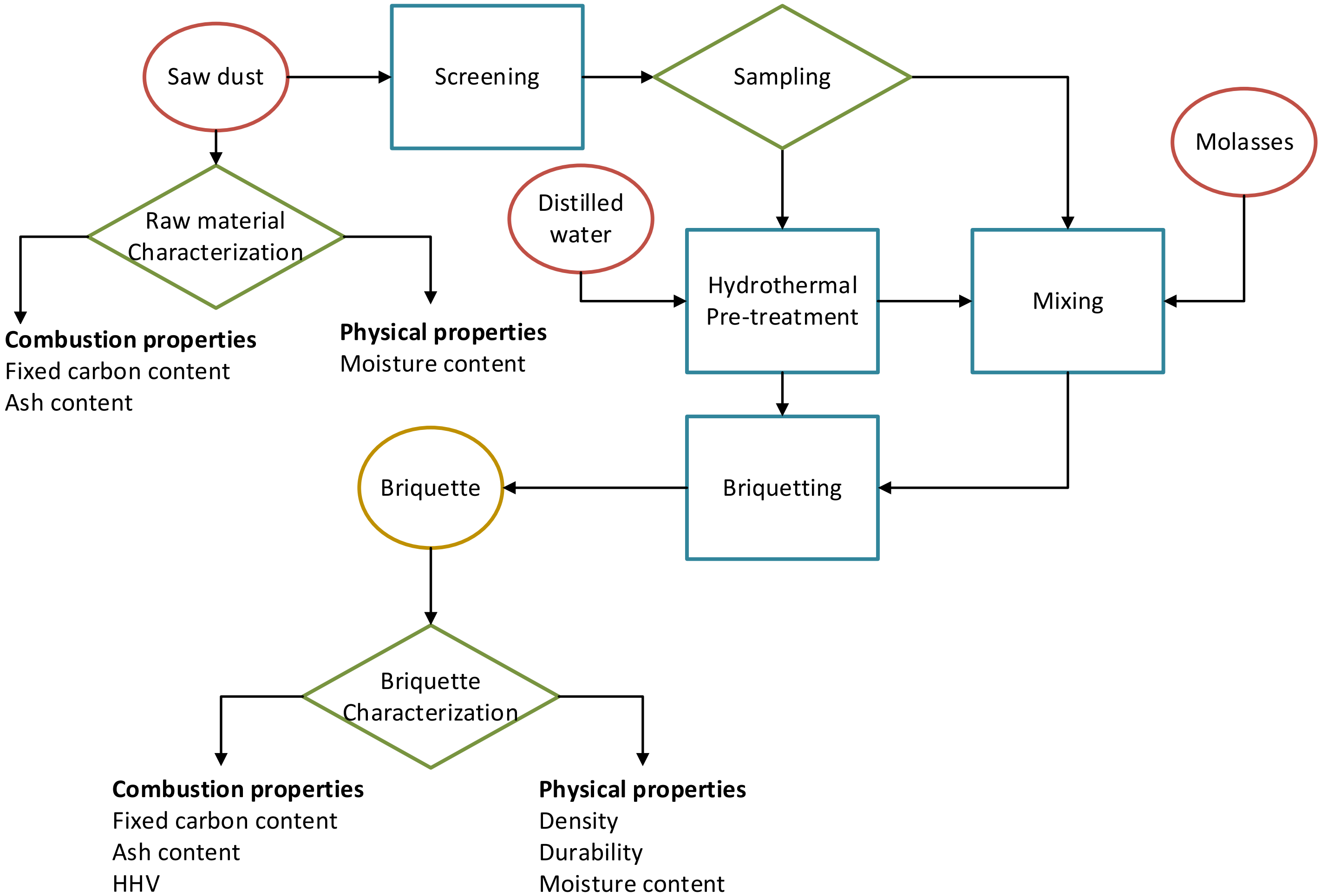

Figure 1: Experimental frame work

3.2.1 Biomass Briquette Manufacturing Procedure

i. Sample Collection

Saw dust of *Cupressus Lusitanica* was collected from a local wood workshop in Addis Ababa while molasses was collected from National Alcohol and Liquor Factory which is also located in Addis Ababa.

ii. Sieving

The raw sawdust was characterized in terms of MC, density, VM, and AC. Then the remaining sawdust was screened on a sieve shaker to obtain a particle size between 1mm -0.5mm. Sawdust retained in the remaining sieve trays was discarded. The sieve tray arrangement is shown in figure 2a; the sieve sizes from top to bottom are 3mm, 2mm, 1mm, 0.5mm, and 0mm(pan).

Figure 2: Screening apparatus

A. Sieve shaker; B. Sawdust particle size (1-0.5mm)

iii. Hydrothermal Pretreatment

The HPT of the biomass was carried out in an autoclave. The reactor is mainly composed of a cylindrical outer vessel, an inner compartment for carrying the sample, heater, inlet/outlet valve, pressure gage, and a thermoregulator. The design temperature range of the autoclave is 0-400 °C, the precision of the temperature control is ±2°C, and the pressure range is 0-20 MPa.

Figure 3: Sawdust before and after HPT

A. Sawdust mixed with distilled water; B. HPT sawdust

First, 200±1 g of sawdust was weighed and mixed with 1000 mL deionized water and was loaded into the reactor and the top lid was closed. Then, nitrogen at a rate of 200 mL/min was pumped through the inlet valve into the reactor for 20 min in order to displace the remaining atmospheric air inside of the reactor through the outlet valve. After purging, the inlet and outlet valves were closed and the reactor was heated to temperatures of 240, 280, and 320^{o}C at an average heating rate of 5 °C/min. After holding for 20 min at the respective temperature, the heater was turned off and outlet valve was opened to release the gas formed inside the reactor. Finally, after the reactor was cooled, the pretreated biomass was taken out and placed in a ventilated place at room temperature.

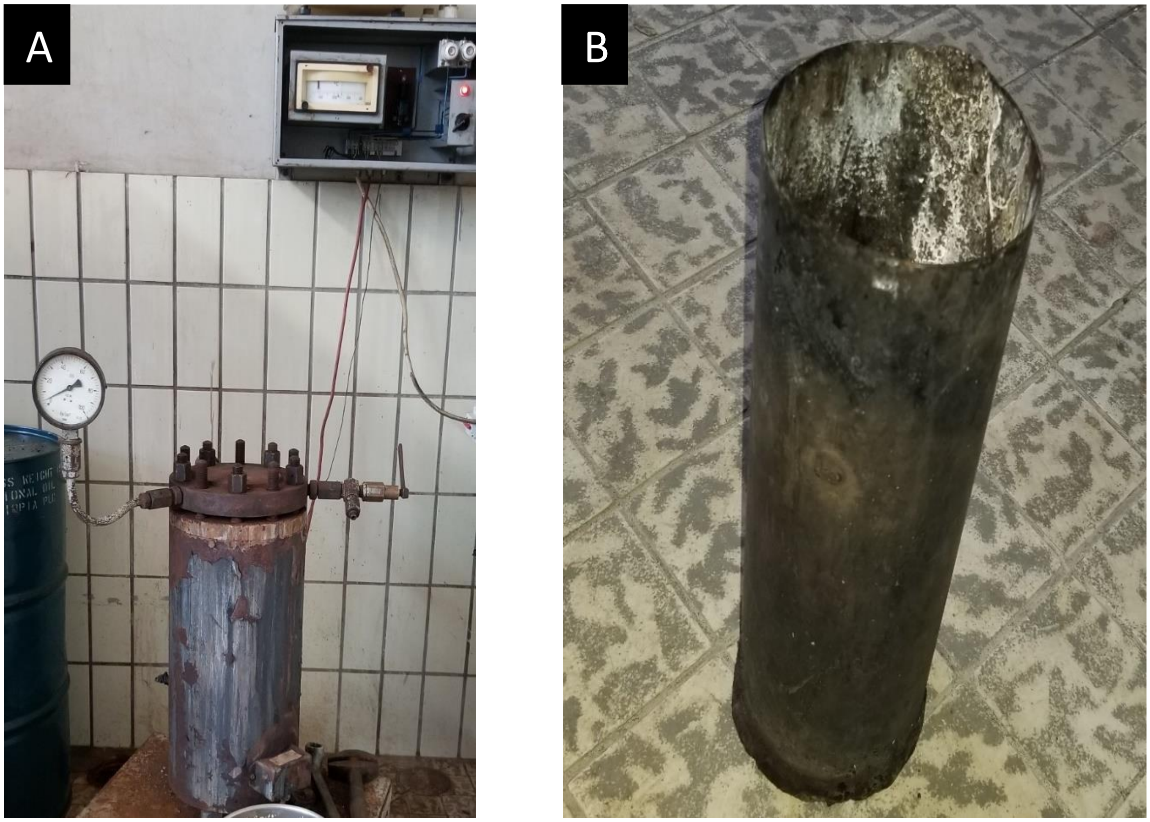

Figure 4: Autoclave

A. Autoclave; B. Vessel

iv. Binder Addition

Molasses was added to the raw and pretreated biomass. First, 200g of biomass is weighed and transferred to a mixing bowl. Then molasses is added at a weight percent of 10% and 20%. Finally, the prepared biomass samples were labeled accordingly.

v. Densification

Each mixture was hand fed into a cylindrical die of dimensions 40mm in diameter and 150mm height that was milled to the desired diameter from mild steel and thoroughly polished to provide a smooth internal surface. The die also has holes along its vertical axis in order to remove excess moisture during compression of the biomass. In the mold, the biomass was in contact with a piston-like compressive metal with a plunger. Then the mold is placed in a manually operated hydraulic piston press and compressed at a pressure of 10MPa for a holding time of 45sec. Finally, the produced briquettes were labeled and stored.

3.2.2 Data analysis

3.2.2.1 Experimental Design

Briquette production is affected by various process variables (pressure, temperature, die geometry, and die length) and feed stock material property (moisture content, binder type and ratio). From all these variables, the effects of HPT temperature and binder weight percent are the two parameters considered in this study. Variation on the above parameters resulted in a significant change on the quality of briquettes. To produce the best quality briquettes, the optimal point of each variable needs to be determined.

Full factorial experimental design was selected to study the effects of these two parameters on the properties of the biomass briquettes. Design-Expert software was used for designing of the experiments (DOE). The levels of the independent variables shown in table1 were selected based on previous researches. The HPT temperature ranges from 240°C to 320 °C and previous studies showed that increment in pretreatment temperature enhances most of the physical and combustion properties; except for HHV. On the other hand, a previous work showed that increment in molasses wt.% increases the density in addition to the VM which will reduce the calorific value[23]. Considering the previous study result, the weight percent for molasses binder were selected to be 0%, 10 %, and 20 %. Coded and actual levels of the two process variables is shown in table 1.

Table 1: Coded and actual levels of parameters

Independent variables	**Symbols**	**Coded and actual levels**			
		0	1	2	3
HPT temperature (°C)	A	none	240	280	320
Molasses wt%	B	0	10	20	-

The total number of runs was determined to be 12 using equation1:

$$N = k_1 \times k_2 \tag{1}$$

$$where : k_1(number\ of\ levels\ for\ the\ first\ factor, A) = 4$$

$$k_2(number\ of\ levels\ for\ the\ second\ factor, B) = 3$$

3.2.2.2 Response Variables

a. Density

Actual measurements of the mass, diameter, and length of the generated briquettes were used to determine the density. The mass was estimated using a digital weighing scale, while the volume was computed using a vernier caliper by taking the briquette's linear measurements (length and diameter). Equation 2 is used to compute the density of the biomass briquette.

$$\rho br = \frac{Mbr}{Vbr} \quad (2)$$

where ρbr = density of briquette, Mbr = mass of briquette, and Vbr = volume of the briquette.

b. Durability

The briquettes' durability was tested in accordance with ASTM D440-86. Each briquette sample was weighed before being dropped three consecutive times from a height of 2 meters onto a concrete floor. The durability (%) can be calculated as the ratio of the briquette's final weight retained after three drops to the original weight of the briquette (equation 3).

$$D(\%) = \frac{m_2}{m_1} \times 100 \quad (3)$$

where D (%) = Durability, m_1 = initial mass of briquette before test (g), and m_2= mass of briquette after test (g).

c. Moisture Content

The percentage MC was found by weighing 2 g of the briquette sample and oven drying it at 105°C until the mass of the sample was constant. Moisture content was determined using equation4.

$$MC = \frac{[m_1 - m_2]}{m_1} \times 100\% \quad (4)$$

where, MC = moisture content, m_1 = mass of sample before drying, and m_2 = mass of sample after drying.

d. Volatile Matter Content

The percentage VM was determined by pulverizing 2 g of the briquette sample in a crucible and placing it in an oven until a constant weight was obtained. The briquettes were then kept in a furnace at a temperature of 550°C for 10 min and weighed after cooling in desiccators. The percentage VM was then calculated using equation 5.

$$\text{VM}(\%) = \frac{(\text{W2} - \text{W3}) * 100}{\text{W2}} \quad (5)$$

where, VM (%) = percentage VM, W2 =oven dried sample weight, and W3 = weight of oven dried sample after heating in furnace.

e. Ash Content

The percentage AC was calculated by heating 2 g of briquette sample in the furnace at 550 °C for 4 hours and weighing after cooling in desiccators to obtain the ash weight. The AC was determined using equation 6:

$$\text{AC}(\%) = \frac{(\text{W2}) * 100}{\text{W1}} \quad (6)$$

where, W1 = weight of oven dried sample (g), W2 =weight of oven dried sample after complete combustion in furnace i.e., weight of ash (g), and AC (%) = percentage AC.

f. Fixed Carbon Content

The percentage of FC was computed by subtracting the sum of VM, AC, and MC from 100 as shown in equation 7:

$$FC\ (\%) = 100 - (AC + MC + VM) \quad (7)$$

g. Calorific Value

Calorific value of the samples was tested at *Geological Survey of Ethiopia*. Calorific value is determined by burning a weighed sample in an adiabatic oxygen bomb calorimeter under controlled conditions. Calorific value is computed from temperature observations made before and after combustion.

The bomb calorimeter housing with thermometers and the bomb cover with crucible holder make up the apparatus. Using an electrical digital weighing scale, the samples were weighed in the crucible to masses ranging from 0.8g to 1.2g. The crucible's sample was then placed inside it, and a 14-mm-long resistance wire was connected across the circuit and immersed in the crucible's sample to complete the circuit. The whole set up was put into the bomb and the bomb is filled with oxygen gas to a pressure of 30atm. Then the vessel inside the calorimeter is filled with distilled water of a weight 2000g±0.5g. Finally, the bomb was immersed inside the water and ignited. Differential thermometer was used to measure the temperature rise and the rise in temperature is correlated to calorific value using equation8.

$$H = \frac{[(t \times w) - e]}{g} \tag{8}$$

where, H = heating value, t = temperature rise, w = water equivalent of benzoic acid, g = weight of sample, e = correction factor of fuse wire which is 5.19KJ/cm for No. 34 B and S gauge wires.

4 Results and discussion

4.1 Raw material characterization

The collected sawdust was sieved in the range of 1~0.5 mm before analysis. The proximate analysis parameters were MC, AC, VM, FC, and calorific value. The result of this analysis is presented in table 2 and compared with proximate values of other solid fuels.

Table 2: Proximate analysis of sawdust and other biomasses

Parameters	Sawdust	Molasses	Sawdust briquettes available in market [11]	Biomass briquettes from charcoal dust[24]	Anthracite Coal [25]
MC (%)	8.94	18.5	9.44	10	0.4
AC (%)	3.61	11.67	3.36	7	6.1
VM (%)	77.63	63.36	83.43	20	6.2
FC (%)	9.82	6.47	3.37	63	87.4
HHV (kJ/kg)	17,560	21,200	18,622.98	28,857.05	23,960

4.1.1 Moisture content

As it can be seen from table 2, the MC of the sawdust sample was greater when compared with hard coal. Wood biomass has a higher porosity index than coal, allowing it to store more moisture between dead cells and within cell walls [26]. The preferred value of MC for alternative fuels, according to C. J. Donahue et al., is less than 20%[25]. It can be seen from table 2 that the MC of the collected sawdust is within the acceptable range, according to table 2.

4.1.2 Ash content

AC represents the incombustible component remaining after a sample of fuel is completely burned. The amount of ash remaining after combustion is directly proportional to the amount of inorganic and incombustible matter present in the solid fuel. AC affects the quality of solid fuel by lowering its calorific value. The AC of solid fuels must be less than 15% [10] [24]. The result in table 2 shows that the AC of the sawdust sample is within the acceptable range and therefore can be used as feedstock for briquette production.

4.1.3 Volatile matter

The VM other than water in charcoal comprises all those liquid and tarry residues not fully driven-off in the process of carbonization. If the carbonization is prolonged and at a high temperature, then the content of volatiles is low. When the carbonization temperature is low and residence time is short, then the VM content increases[5]. This can be seen from table 2 where VMs of raw sawdust and sawdust briquette are significantly higher than that of carbonized wood.

As seen in table 2, the VM present in sawdust was higher than that of coal. This is an indication that sawdust can ignite under lower temperatures when compared to coal[27]. During pyrolysis, these high volatiles result in a quicker combustion rate [15].

4.1.4 Fixed carbon content

The FC content is usually estimated as a "difference", that is to say, the mass that remains after volatiles have been released, omitting ash and moisture content. The amount of carbon accessible for char combustion during pyrolysis is represented as FC. [11], [18]. The FC in the material gives a rough estimate of the heating value. Concerning the result of FC, sawdust was found to have a lower FC content than that of coal and wood charcoal.

4.1.5 Higher heating value

HHV is associated with the oxidation state of natural fuels, in which carbon atoms predominate and tiny fluctuations in hydrogen content are obscured [4],[28]. The FC of sawdust was discovered to be lower than that of coal and wood charcoal, resulting in a reduced heating value.

4.2 Characterization of biomass briquettes

The briquettes were manufactured by varying HPT temperature and wt.% of molasses binder at 4 and 3 levels, respectively. Full factorial experimental design that results in 12 possible combinations/runs was employed. The produced briquettes are displayed in figure 5.

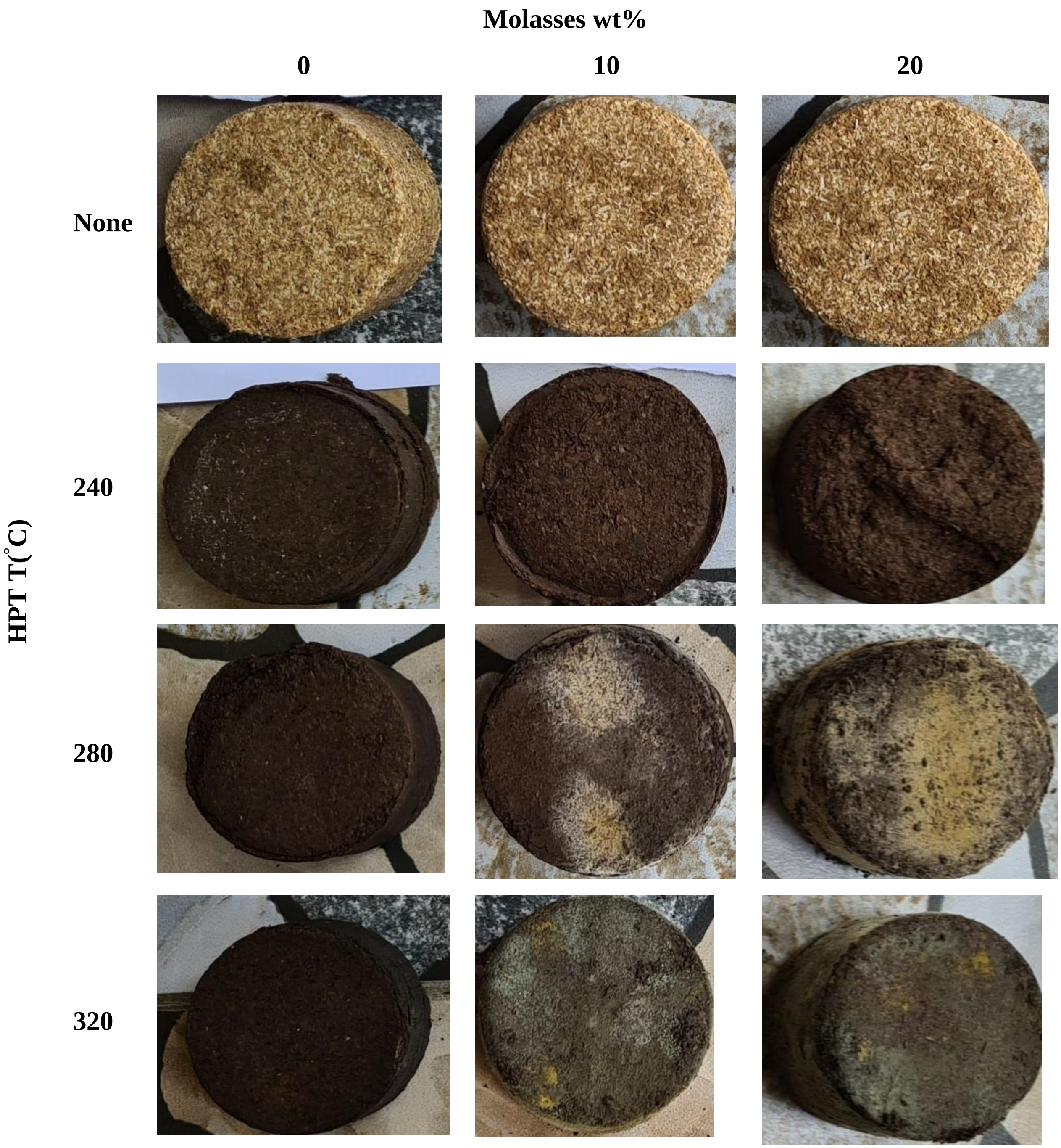

Figure 5:Sawdust briquettes prepared at different temperatures and amount of binder

Physical, proximate, and HHV analysis were applied to comparatively evaluate the produced briquettes. The purposes of these analyses are to indicate the density, durability, MC, VM, AC, the percentage by weight of the FC, and HHV of the briquettes. The 4×3 full factorial experimental design with their respective responses is shown in table 3.

Table 3: Two factor, full factorial design of process variables and responses

Run	**Process variables**		**Response**						
	Temp. (°C)	Binder (wt%)	Density (kg/m^3)	Durability (%)	MC (%)	VM (%)	AC (%)	FC (%)	HHV (MJ/kg)
5	None	0	573.34	74.69	8.94	77.6	3.61	9.82	20.38
2	None	10	638.66	85.33	9.9	76.2	4.42	9.48	19.64
7	None	20	751.76	91.20	10.9	74.8	5.22	9.15	19.06
6	240	0	699.86	99.96	11.1	71.8	1.78	15.3	20.40
11	240	10	727.33	99.98	11.8	71	2.37	14.8	19.77
9	240	20	792.87	99.98	12.5	70.1	3.25	14.1	19.16
1	280	0	780.00	99.99	10.7	57	2.39	29.9	20.52
12	280	10	791.70	99.97	11.5	57.6	3.32	27.6	20.01
3	280	20	838.26	99.98	12.3	58.2	4.25	25.2	19.60
4	320	0	747.47	99.99	9.61	52.2	2.87	35.4	20.74
10	320	10	794.34	99.97	10.5	53.3	3.75	32.5	20.57
8	320	20	816.13	99.98	11.4	54.4	4.63	29.6	20.12

4.2.1 Density

Denser briquettes, keeping other variables constant, contain more heat per unit volume and tend to burn for longer periods. The mass density of pellets has a significant influence on transportation costs, energy density, and mechanical properties of the solid fuel. The density of biomass briquettes is determined by the feed biomass type, die shape, briquetting pressure, and, to a degree, briquetting temperature and duration [26].

M. S. Nasir reported a maximum density of sawdust-starch composite briquettes of 670 kg/m^3 at binder wt% of 40 % and pressure of 9 MPa[29]. The maximum density achieved in this work is 838.26 kg/m^3. This variation could be the result of the type of binder used, applied pressure between the two studies, and the presence of HPT of the biomass in this study.

4.2.1.1 Analysis of variance

Using full factorial ANOVA, the influence of HPT temperature and binder wt percent on the density of biomass briquettes was explored; the results are presented in table 4 and discussed.

Table 4: ANOVA table for density (Quadratic Model)

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	60970.49	5	12194.10	17.91	0.0015	significant
A-Temperature	33292.51	1	33292.51	48.91	0.0004	
B-Binder wt%	24051.40	1	24051.40	35.33	0.0010	
AB	4283.76	1	4283.76	6.29	0.0460	
A^2	70.06	1	70.06	0.1029	0.7592	
B^2	380.95	1	380.95	0.5596	0.4827	
Residual	4084.22	6	680.70			
Cor Total	65054.71	11				

The 17.91 Model F-value indicates that the model is significant. An F-value of this magnitude has a 0.15 percent chance of occurring due to noise. Model terms with P-values less than 0.05 are considered significant. A, B, and AB are important model terms. The model terms are not important if the value is bigger than 0.1000.

Table 5: Fit statistics for density (Quadratic Model)

Std. Dev.	**26.09**		**R^2**	**0.9372**		**Adeq Precision**	**14.1993**
Mean	745.98		**Adjusted R^2**	0.8849			
C.V. %	3.50		**Predicted R^2**	0.7856			

The **Predicted R^2** of 0.7856 is in reasonable agreement with the **Adjusted R^2** of 0.8849; i.e., the difference is less than 0.2. **Adeq Precision** measures the signal to noise ratio. A ratio greater than 4 is desirable.

Table 6: Coefficients in terms of coded factors for density (Quadratic Model)

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	709.85	1	23.11	653.29	766.40	
A-Temperature	71.89	1	10.28	46.74	97.04	1.13
B-Binder wt%	59.09	1	9.94	34.76	83.41	1.16
AB	-29.74	1	11.85	-58.75	-0.7314	1.16
A^2	8.10	1	25.25	-53.69	69.89	1.13
B^2	11.95	1	15.98	-27.14	51.05	1.0000

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multi-collinearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.

$$Density = 709.85 + 71.89A + 59.09B - 29.74AB + 8.10A^2 + 11.95B^2 \qquad (9)$$

4.2.1.2 Effect of Process Variables on Density of Briquettes

i. ***Effects of hydrothermal pretreatment on density of briquettes***

The effect of HPT temperature on density of briquettes is shown in figure 6a. As it can be seen from table 3 and figure 6a, the density of HPT sawdust briquettes shows an increasing trend up to HPT temperature of 280 ˚C after which the density starts to gradually decrease. This may be due to the degradation of lignin caused by higher HPT temperature, which weaken the bonding effect of lignin as crucial natural binder in biomass during densification. Beall F et al. analyzed wood samples for methoxy group after isothermal heating at temperatures between 150 °C and 500 °C[30]. They proposed that lignin decomposition begins at about 290 °C with a maximum rate of decomposition occurring between 350 °C and 450 °C. The slight decrease in density at HPT temperature of 320 °C in this study could be the result of lignin decomposition.

ii. ***Effects of binder wt% on density of briquettes***

Different wt% of molasses was combined for each level of hydrothermal pretreatment of the sawdust. Y. Zhai et al. showed that the density of the briquettes increased from 872.1 kg/m^3 to 912.9 kg/m^3 after the addition of 20 wt% molasses[31]; the increase in density was due to the high viscosity of molasses that helps to glue together the sawdust particles. In this study the highest density that could be achieved was 838.26 kg/m^3; the difference in density could be due to the difference in briquetting pressure applied or the biomass used. Figure 6b shows that at a fixed HPT temperature, as binder wt% increases the density of the briquettes also increases.

iii. ***Interaction effect of Hydrothermal pretreatment temperature and binder wt% on density of briquettes***

The interaction effect of HPT temperature and binder wt% on density of briquettes is shown in figure 6c. It can be seen from table 4 that the P value of the interaction, AB, is 0.024 and this P value verifies a significant interaction effect between the two parameters.

As HPT temperature and binder wt% increases, the density of briquettes shows an increasing trend until HPT temperature reaches 280 °C where it attains the maximum value. However, the density seems to decrease when HPT temperature is further increased to 320 °C due to the degradation of lignin after a temperature of 290 °C. On the other hand, the amount of binder has a direct correlation with density throughout the test runs.

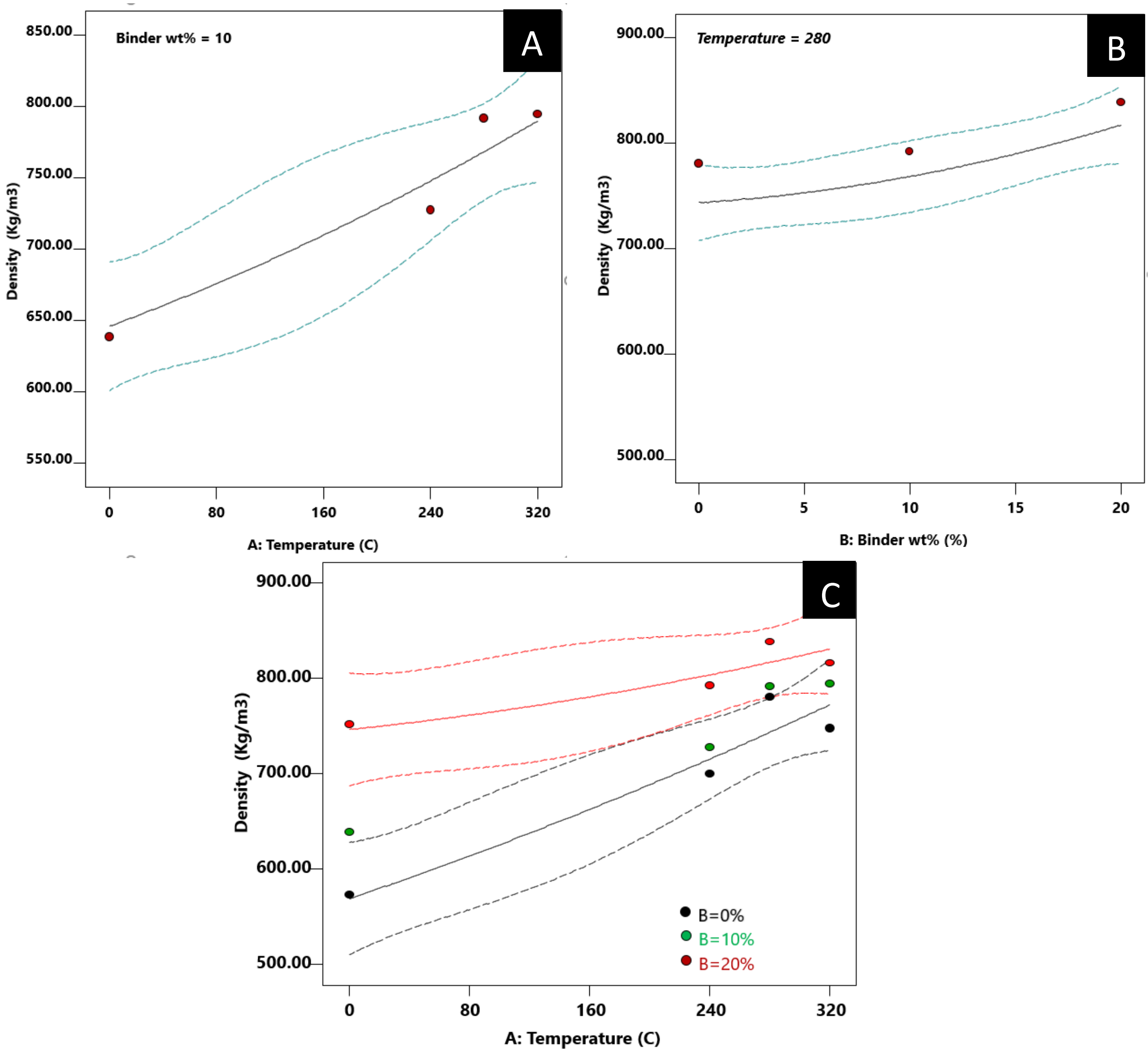

Figure 6: Effects of HPT Temperature and binder wt % on Density of briquettes

A. Effect of HPT temperature on density; B. The effect of binder wt% on density of briquettes; C. Interaction effect of HPT temperature and binder wt% on density of briquettes

4.2.2 Durability

Effects of process variables i.e., HPT temperature and binder wt% on the durability of briquettes is discussed below.

i. Effect of HPT temperature on durability of briquettes

The effect of HPT temperature on durability of briquettes is shown in figure 7a. It can be seen from figure 7a and table 3 that durability of HPT briquettes remains constant at almost 100 % without significant difference. When compared to the untreated briquettes, HPT briquettes exhibited significant increase in durability. However, difference in durability between HPT briquettes cannot be identified due to the high shatter resistance of HPT briquettes.

ii. Effects of binder wt% on durability of briquettes

The effect of binder wt% on durability of untreated and treated briquettes can be seen in figure 7b and 7c. It can be seen from figure 7b and table 3 that durability of untreated briquettes seems to improve as binder wt% increase whereas durability of HPT briquettes seems to show no correlation with binder wt%. However, previous studies revealed an increase in durability with binder wt%[24], [31], [32]. The lack of correlation between durability of HPT briquettes and binder wt% in this study indicated the need for other testing methods.

iii. *Interaction effect of HPT and binder wt% on durability of briquettes*

Interaction effect of HPT temperature and binder wt% on durability of briquettes can be observed in figure 7d. It can be seen from the ANOVA table that P value of AB interaction is 0.0002 indicating significant interaction effect between HPT temperature and binder wt% on the response (durability of briquettes). The interaction plot shows that durability of briquettes exhibited slightly higher increase when molasses and HPT temperature are increased at the same time than when they were increased separately. This could be the result of the high viscosity and cohesive properties of molasses which further strengthens the binding effect of lignin molecules inherently found in the biomass.

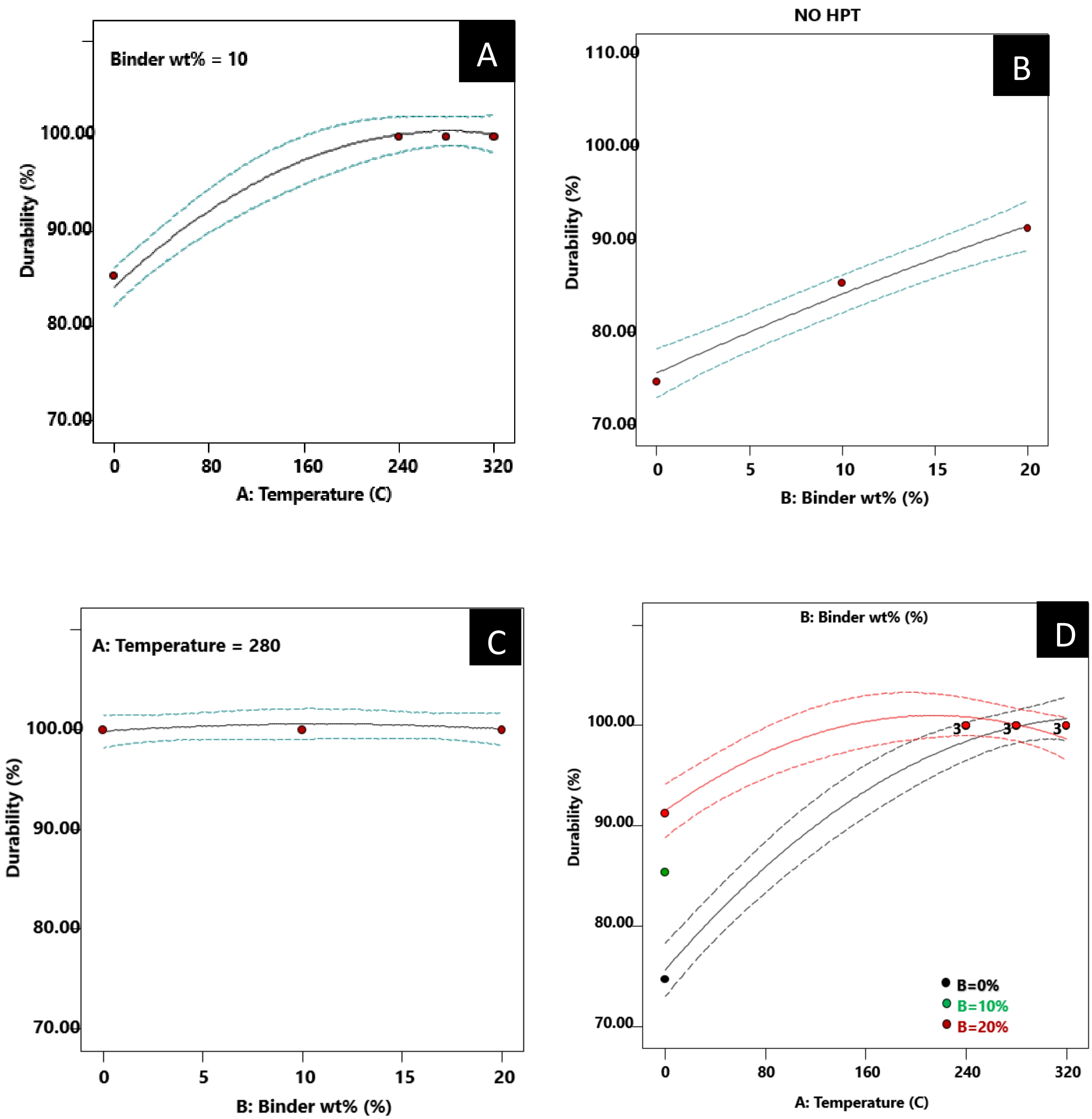

Figure 7: Effects of HPT Temperature and binder wt % on Durability of briquettes

A. Effect of HPT temperature on durability; B. The effect of binder wt% on durability of untreated briquettes; C. The effect of binder wt% on durability of HPT briquettes; D. Interaction effect of HPT temperature and binder wt% on durability of briquettes

4.2.3 Moisture content

Heating value of biomass briquette is affected by the weight% of moisture it contains. Moisture decreases the low heating value (LHV) of the fuel.

Mechanical strength and binding mechanism can be influenced by the MC of the feedstock and biomass briquette. According to P. Jittabut, briquettes having the highest MC of 19.2% resulted in lowest shatter resistance with durability of 64.19 wt%[18].

The influence of HPT temperature and binder wt% on the MC of biomass briquettes was analyzed using the general factorial ANOVA and the results are presented in ANNEX 2.

i. Effects of HPT temperature on moisture content

The effect of HPT temperature on MC of briquettes can be seen in figure 8a. As it can be seen from table 3 and figure 8a, MC of the briquettes shows a decreasing trend as the HPT temperature increases. Beall F. et al. performed thermogravimetric analysis to investigate the thermal degradation reactions of wood, cellulose, hemicelluloses, and lignin and discovered that as HPT temperature increases the amount of steam released after the HPT process increases and the rate of hydrolysis reaction also increases which decreases the amount of moisture remaining within the biomass[30]. However, the minimum MC is achieved in briquettes without HPT due to the absence of addition of water during pretreatment.

ii. Effects of binder wt% on moisture content

Table2 shows that molasses has a higher moisture content when compared to sawdust. As the weight% of molasses increases, the moisture content of the sawdust briquette also increases as shown in figure 8b.

iii. Interaction effects of HPT temperature and binder wt% on moisture content

The interaction effect of HPT temperature and binder wt% on MC of briquettes is shown in figure 8c. According to the ANOVA table, the interaction effect of the two factors (HPT temperature and binder wt%) was insignificant with a P value of 0.6246.

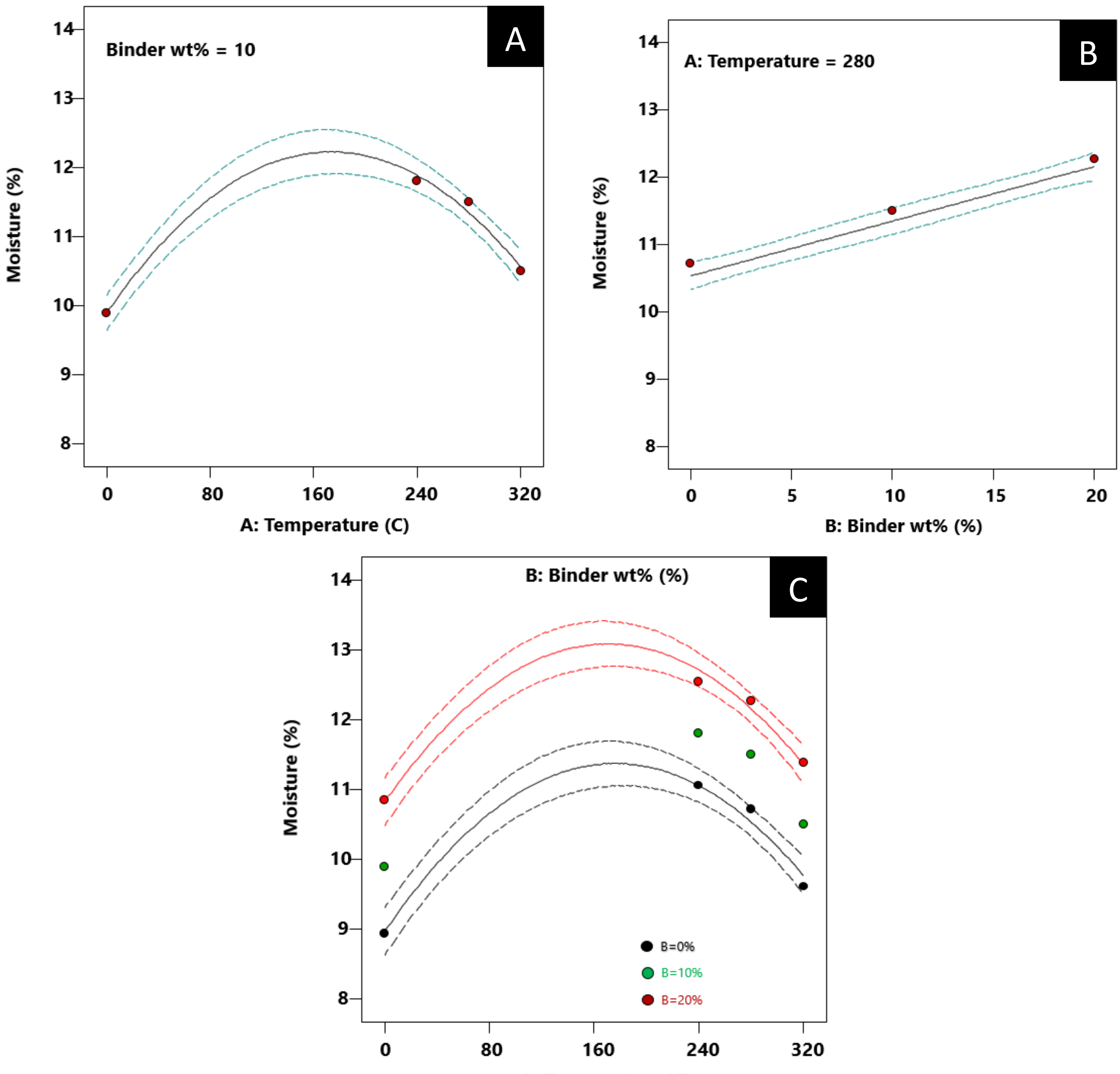

Figure 8: Effects of HPT Temperature and binder wt % on Moisture content of briquettes

A. *Effect of HPT temperature on Moisture content; B. The effect of binder wt% on Moisture content of briquettes; C. Interaction effect of HPT temperature and binder wt% on Moisture content of briquettes*

4.2.4 Volatile matter

VM content affects combustion properties of fuels. Higher VM causes smoke, long flames and decrease the calorific value. The influence of HPT temperature and binder wt% on VM content of biomass briquettes was analyzed using the general factorial ANOVA and the results are presented in ANNEX 3.

4.2.4.2 Effects of process variables on volatile matter of briquettes

i. Effect of HPT temperature on volatile matter of briquettes

The effects of HPT temperature on volatile matter of briquette can be seen in figure 9a. As it can be seen from table 3 and figure 9a, an increase in HPT temperature decreases the VM of biomass briquettes. The hydrothermal pretreatment step decreases the percentage of VM by removing components that have lower evaporation temperature. O. Uner et al. describes how an increase in temperature yields charcoal having lower VM[33].

ii. Effect of binder wt% on volatile matter content of briquettes

It can be seen from figure 9b that an increase in binder wt% results in a slight decrease of VM of the briquettes. This is due to the difference in VM of molasses and sawdust; molasses has a slightly lower VM than sawdust. Consequently, as molasses wt% increases, VM of the briquette decreases.

iii. Interaction effect of HPT temperature and binder wt% on Volatile matter

The interaction effect of HPT temperature and binder wt% on VM content of briquettes is shown in figure 9c. The interaction effect of the two factors (experimental variables) was insignificant with a P value of 0.3316.

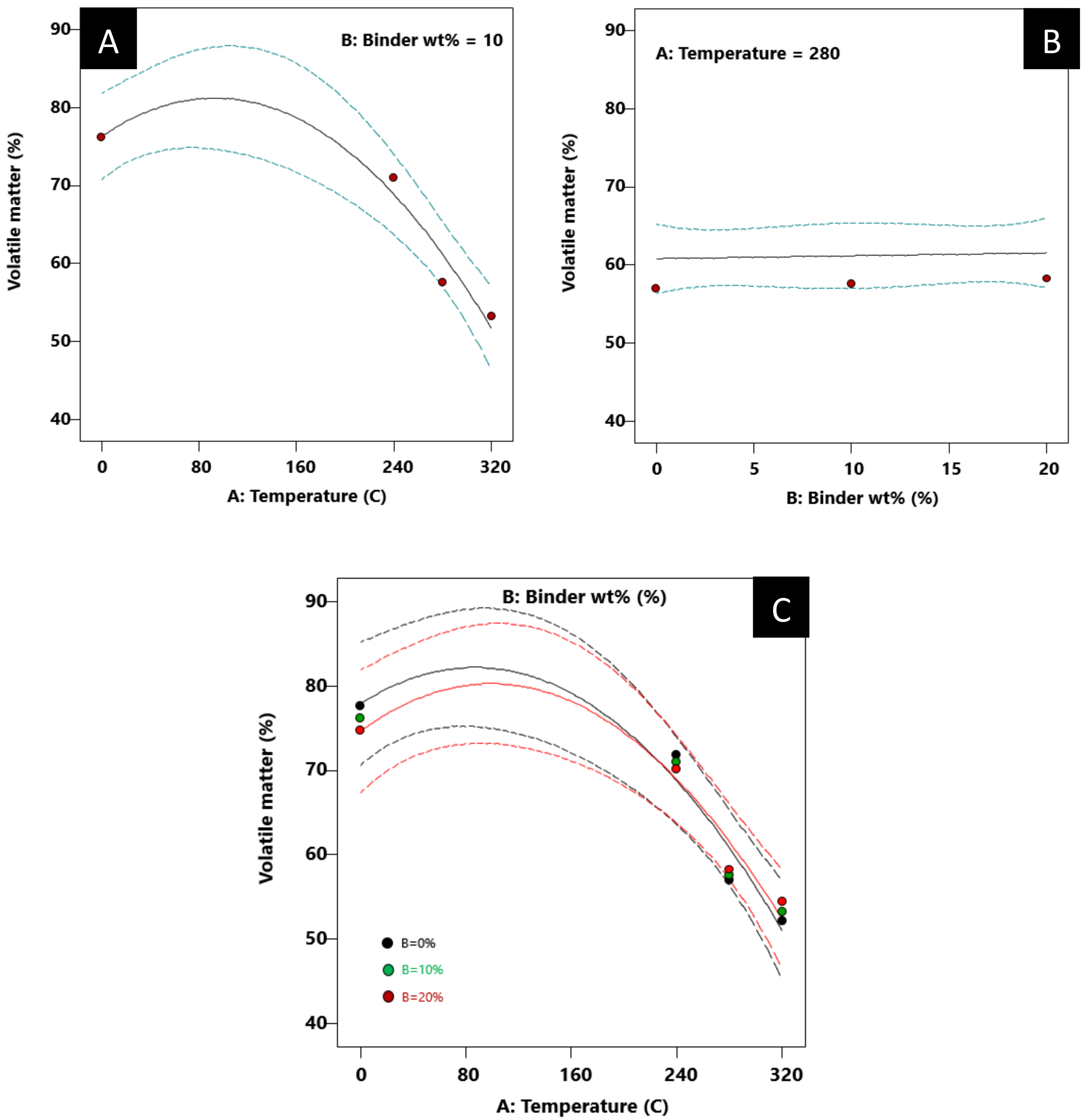

Figure 9: Effects of HPT Temperature and binder wt % on Volatile matter of briquettes

A. Effect of HPT temperature on Volatile matter; B. The effect of binder wt% on Volatile matter of briquettes; C. Interaction effect of HPT temperature and binder wt% on Volatile matter of briquettes

4.2.5 Ash content

Ash is formed from the incombustible components of solid fuels; higher ash content results in lower calorific value. The influence of HPT temperature and binder wt% on the AC of biomass briquettes was analyzed using the general factorial ANOVA and the results are presented in ANNEX 4.

i. Effect of HPT temperature on ash content of briquettes

It can be seen from figure 10a that AC is highest for briquettes without HPT=0 and decreases at HPT of 240 °C and starts to increase as HPT temperature rises. It can be seen from experimental results that biomass that didn't undergo HPT has a relatively lower MC while having lower FC that resulted in a higher AC. During HPT, the ash components in the biomass do not change and all ash components of biomass are still present in treated biomass. The change in the AC is relative to the change in the original biomass components. As the biomass loses some of the moisture and volatiles during the process, the AC is more of a relative increase with respect to the original components. J.T Oladeji et al. have reported that the ash yields of corn stover and miscanthus samples at HPT temperature of 200 °C showed a significant decrease by 83% and 74% , respectively, and about 90% of calcium, magnesium, sulfur, phosphorus, and potassium were removed[21].

ii. Effect of binder wt% on ash content of briquettes

Table 2 shows that molasses has a higher AC compared to sawdust and as the weight% of molasses increases, the AC of sawdust briquette also increases. This can be seen in figure 10b.

iii. Interaction effect of HPT temperature and binder wt% on ash content

The interaction effect of HPT temperature and binder wt% on AC of briquettes is shown in figure 10c. The interaction effect of the two parameters was insignificant, as can be seen from the ANOVA table, with a P value of 0.8071.

M. S. Nasir obtained sawdust briquette having the least AC of 2.87 % without HPT[34]. The lowest AC recorded in this study was 1.78 % at HPT temperature of 240 °C and 0 wt% molasses resulting in better quality briquettes. As HPT temperature and molasses wt% increase, the AC of briquettes shows an increasing trend with the exception for those without HPT where maximum ash content is attained.

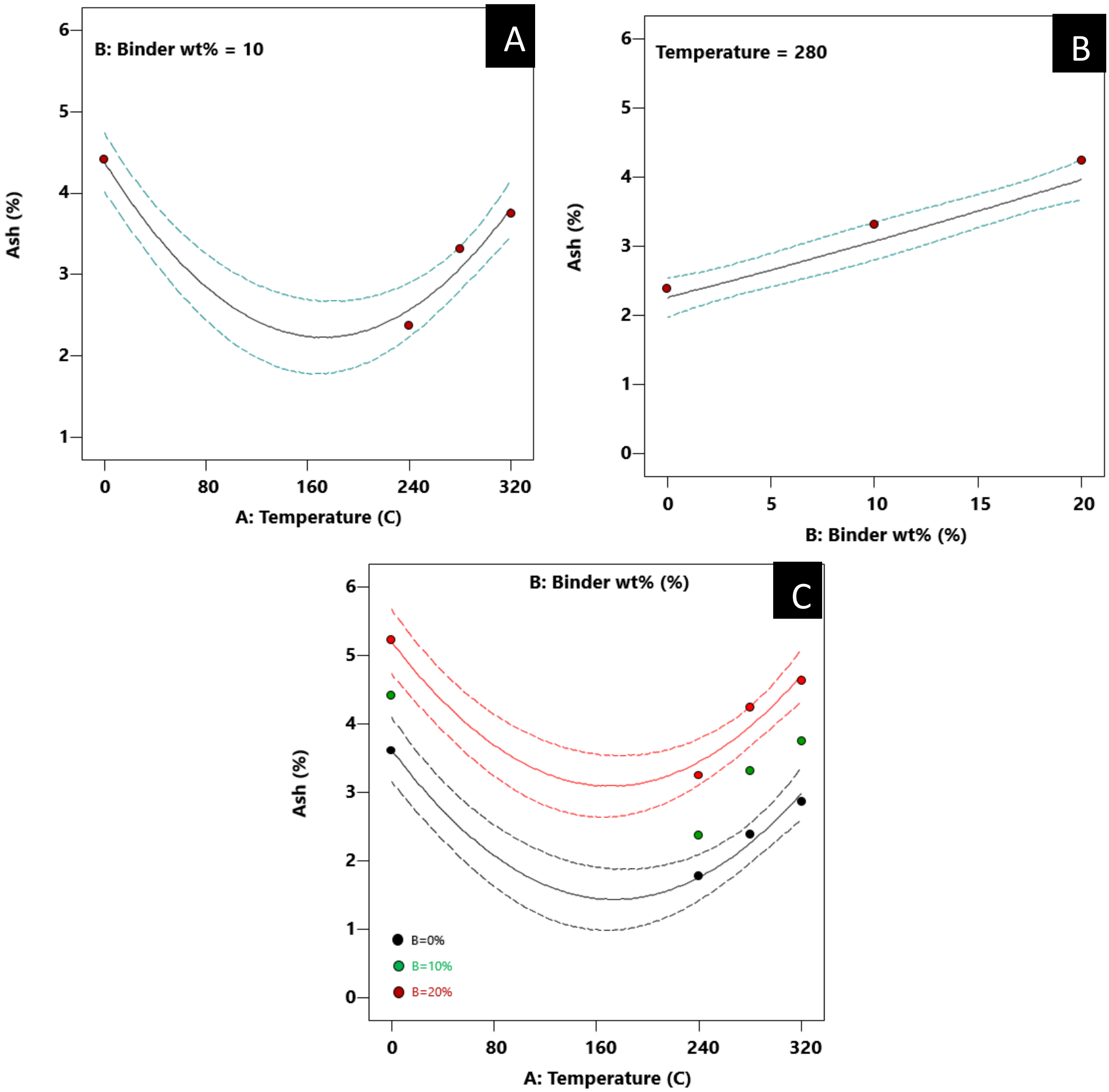

Figure 10: The effect of HPT temperature and binder wt% on Ash content of briquettes

A. Effect of HPT temperature on Ash content; B. The effect of binder wt% on Ash content of briquettes; C. Interaction effect of HPT temperature and binder wt% on Ash content of briquettes

4.2.6 Fixed carbon content

4.2.6.1 Analysis of variance

The influence of HPT temperature and binder wt% on the FC content of biomass briquettes was analyzed using the general factorial of ANOVA and the results are presented in ANNEX 5.

i. Effect of HPT temperature on fixed carbon of briquettes

HPT increases FC of biomass briquettes through the removal of moisture and VM. Y. Zhai et al. analyzed hydro-char produced from food waste under varying hydrothermal carbonization temperature[31]. The hydro char pellets exhibited improved fuel quality including higher FC when both the hydrothermal carbonization temperature and residence time increased. The FC for 260 °C hydrothermal carbonization temperature; 1hr residence time had the highest FC of 38.01 percent, showing that the carbonization process was driven more by hydrothermal temperature than by residence time. In this investigation, the greatest FC was 35.35 percent; this slight decrease can be associated with the difference in the type of feed biomass used. The effect of HPT temperature on FC content is shown in figure 11a.

ii. Effect of binder wt% on fixed carbon content of briquettes

Table 2 shows that molasses has a higher FC content when compared to sawdust. Therefore, as the wt% of molasses increases, the FC content of the sawdust briquette slightly increases. However, it can be seen from table 19 that the P values of B and B^2 are greater than 0.05 indicating that binder wt% is an insignificant factor for FC content of the briquettes. This is shown in figure 11b.

iii. Interaction effect of HPT temperature and binder wt% on fixed carbon

The interaction effect of HPT temperature and binder wt% on FC content of briquettes is shown in figure 11c. The interaction effect of the two experimental variables was insignificant with a P value of 0.2761.

As HPT temperature and molasses wt% increase, the FC content of briquettes shows an increasing trend. However, ANOVA results indicate that binder wt% has no significant effect on FC content thereby implying no interaction effects of the two process variables on the FC.

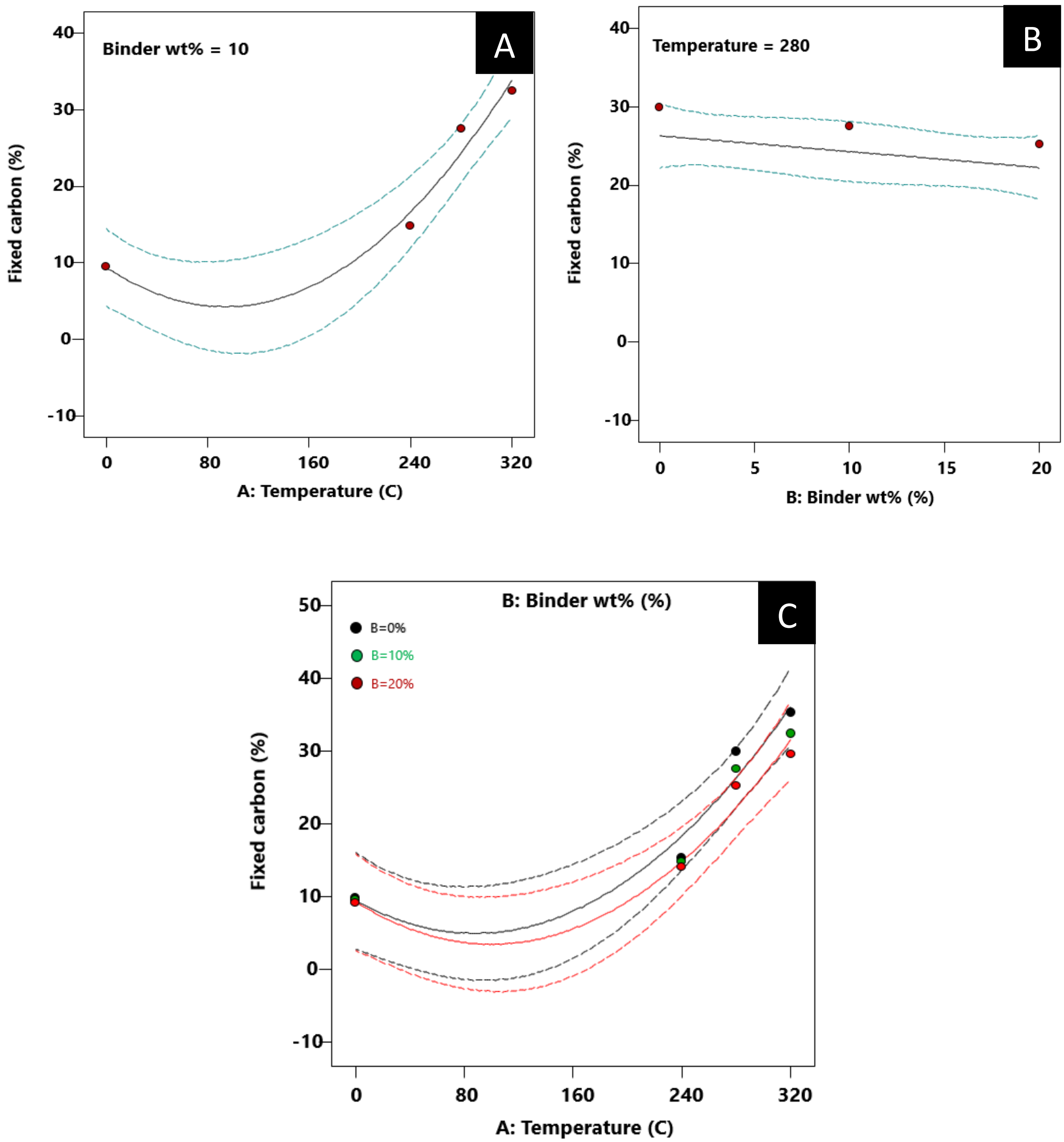

Figure 11: The effect of HPT temperature and binder wt% on Fixed carbon content of briquettes

A. Effect of HPT temperature on fixed carbon content; B. The effect of binder wt% on d fixed carbon content of briquettes; C. Interaction effect of HPT temperature and binder wt% on fixed carbon content of briquettes

4.2.7 High heating value

The influence of HPT temperature and binder wt% on the HHV of biomass briquettes was analyzed using the general factorial ANOVA and the results are presented in ANNEX 6.

i. Effect of HPT temperature on HHV of briquettes

HPT increases HHV of biomass briquettes by increasing FC content through the removal of VM. It can be seen from figure 12a that the HHV improves as HPT temperature increases. Y. Zhai et al. produced hydro-char from food waste under varying hydrothermal conditions, and was carbonized then pelletized[31]. HHVs of the pellets increased consistently with increasing hydrothermal severity, reaching about 32.36 MJ/kg at 260 °C and 8 h. Highest HHV attained in this study is 24.68 MJ/Kg. The decrease in HHV could be the result of difference in feed material and absence of carbonization in this study.

ii. Effect of binder wt% on HHV content of briquettes

Table 2 shows that molasses has lower HHV when compared to sawdust. Therefore, as the wt% of molasses increases, the HHV of sawdust briquette decreases. M. Muluken shows that an increase in binder wt% results in the decrease in HHV of the briquettes[13]. The results in this study also seem to comply with M. Muluken as shown in figure 12b.

iii. Interaction effect of HPT temperature and binder wt% on HHV

The interaction effect of HPT temperature and binder wt% on HHV of briquettes is shown in figure 12c. it can be seen from table 7 that the p value of the interaction, AB, is 0.0002 indicating a significance interaction effect between the two parameters

As HPT temperature and molasses wt% increase, the HHV of briquettes shows an increasing and decreasing trend respectively.

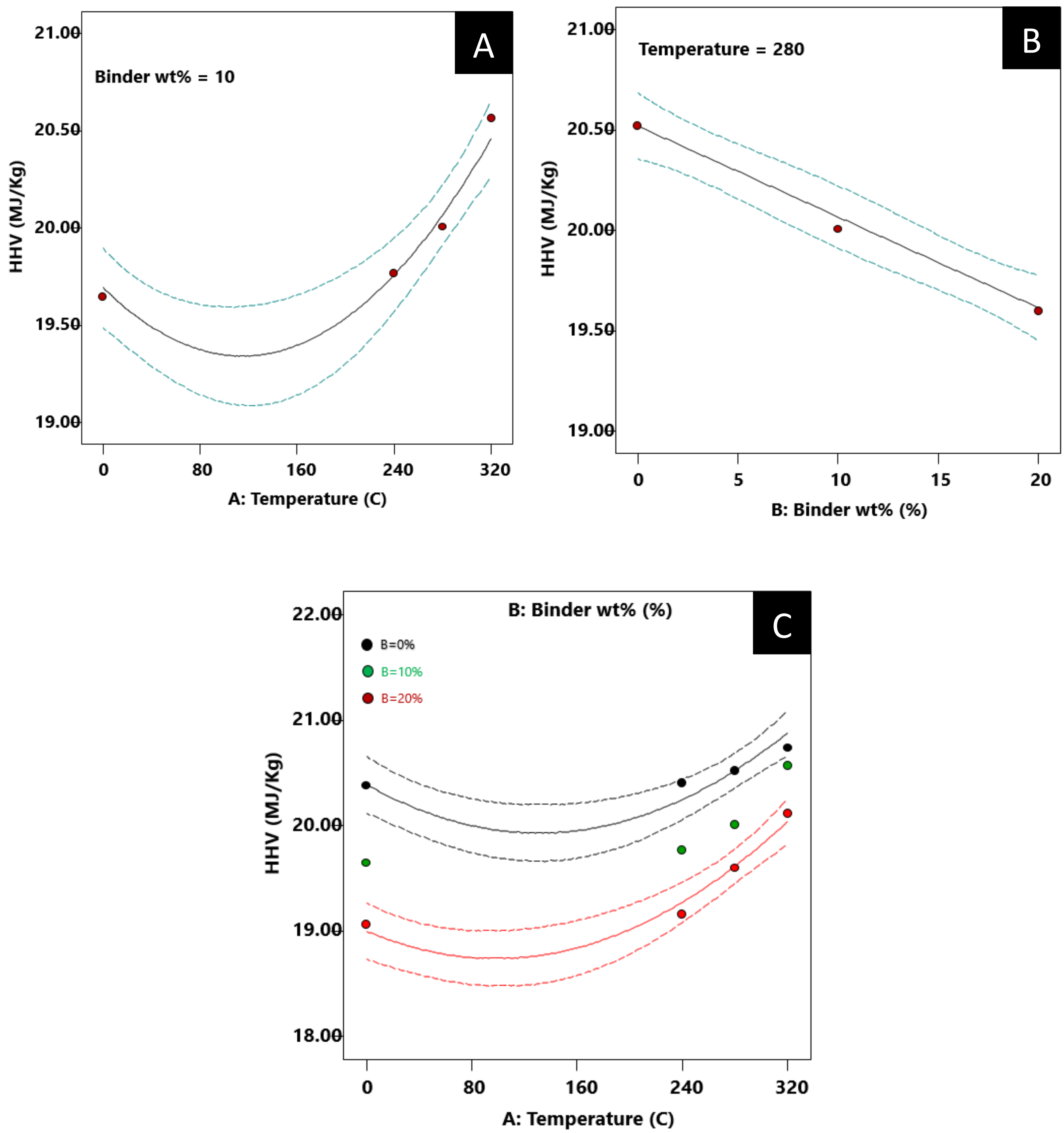

Figure 12: The effect HPT temperature and binder wt% on HHV of briquettes

A. Effect of HPT temperature on HHV; B. The effect of binder wt% on HHV of briquettes; C. Interaction effect of HPT temperature and binder wt% on HHV of briquettes

4.3 Optimization

Determining the optimum operating conditions for the briquettes is aimed at manufacturing solid fuels having superior overall quality. In this regard, the constraints used for optimization are shown in table 7.

Table 7: Constraint

Name	A	B	Density	Durability	MC	VM	AC	FC	HHV
Goal	in range	in range	Max	Max	Min	Min	Min	Max	Max
Lower Limit	None	0	573	74.7	8.94	52.2	1.78	9.15	19.1
Upper Limit	320	20	838	100	12.5	77.6	5.22	35.4	20.7

Based on the criteria given in table 7, numerical optimization was carried out in order to predict optimum operating points and 8 solutions were obtained. The forecasted solution having the highest desirability was selected.

The selected solution had a desirability of 0.87 and the process parameters were HPT = 320^{O}C and binder wt% = 0%; the predicted response variables at those points can be seen from table 8.

Table 8: Generated solution for optimization

No	**HPT**	**Binder wt%**	**Density**	**Durability**	**MC**	**VM**	**AC**	**FC**	**HHV**	**Desirability**
1	320.00	0.00	772.44	100.70	9.77	51.06	2.99	36.18	20.88	0.87
2	318.21	0.00	771.13	100.68	9.81	51.53	2.96	35.70	20.86	0.87
3	320.00	0.76	772.92	100.71	9.83	51.11	3.05	36.01	20.85	0.86
4	320.00	8.63	786.05	100.41	10.46	51.62	3.70	34.22	20.52	0.78
5	0.00	10.93	654.41	84.87	9.98	76.13	4.46	9.43	19.63	0.17
6	0.00	10.84	653.60	84.80	9.97	76.14	4.45	9.43	19.64	0.17
7	0.00	11.06	655.62	84.97	9.99	76.11	4.47	9.43	19.62	0.17
8	0.00	9.93	645.42	84.08	9.89	76.29	4.38	9.44	19.70	0.17

T=320^{O}C   Binder wt%= 0

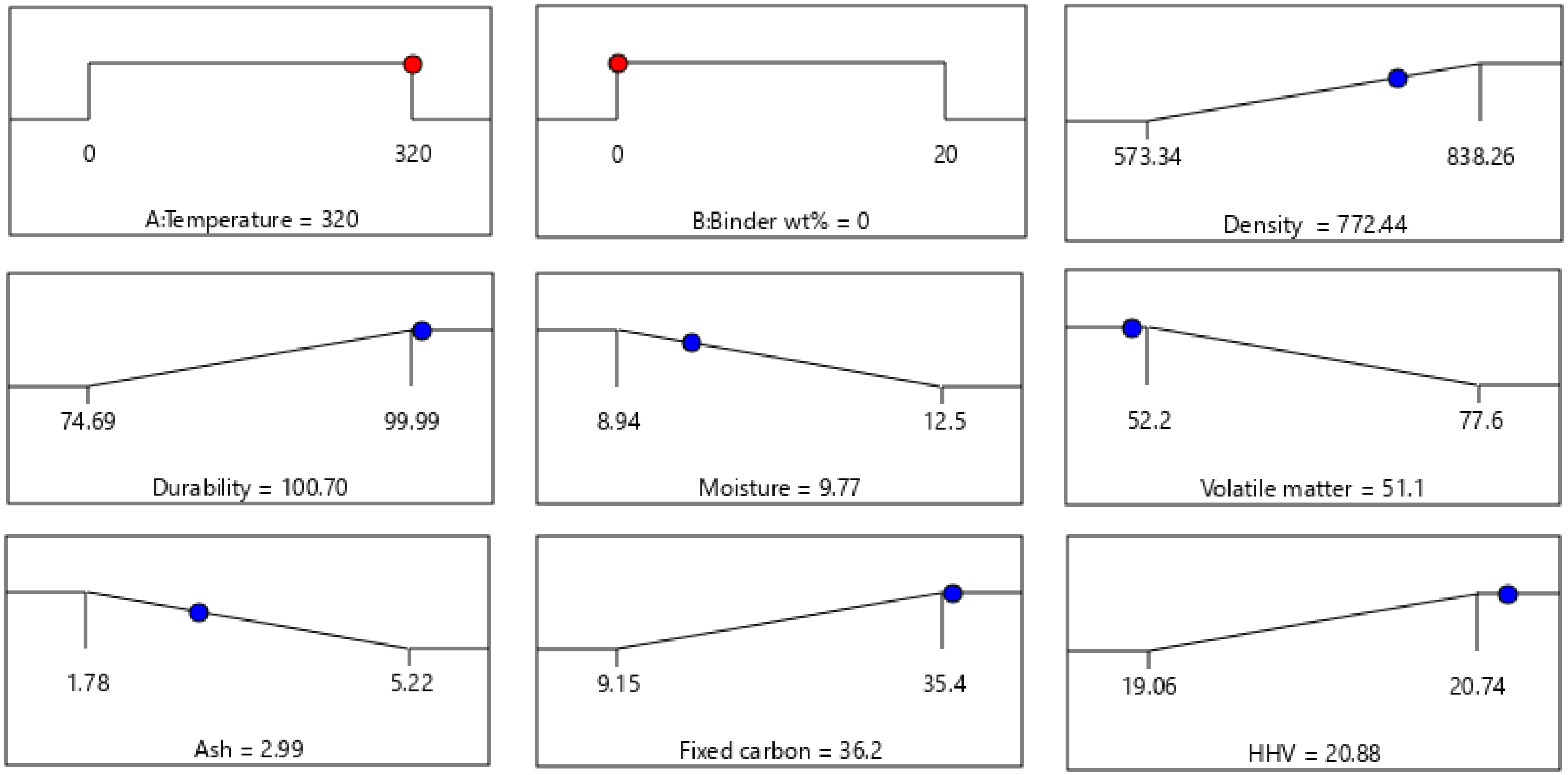

Figure 13: Ramps for the selected optimum point solution

Validation

To validate the optimum condition predicted by the model, triplicate experiments were conducted at the optimum points.

Table 9: Validation of optimization model

Response variables	Test run 1	Test run 2	Test run 3	SD	Average	Predicted value	Error
Density (kg/m3)	747.05	747.50	747.86	0.41	747.47	772.44	0.03
Durability (%)	99.99	99.99	99.99	0	99.99	100.70	0.01
MC (%)	9.05	10.24	9.54	0.49	9.61	9.77	0.02
VM (%)	52.74	52.09	51.78	0.40	52.2	51.1	0.02
AC (%)	2.47	2.45	3.69	0.58	2.87	2.99	0.04
FC (%)	36.10	34.96	35.14	0.50	35.4	36.2	0.02
HHV (MJ/kg)	20.74	20.74	20.74	0	20.74	20.88	0.01

As can be seen from table 9, the maximum error is 0.04 which indicates that the generated model is an adequate prediction of the response variables.

5 Conclusion and recommendation

5.1 Conclusion

This study revealed that the physical and combustion properties of biomass briquettes can be improved by hydrothermal pretreatment of the raw biomass whereas binder addition only improves the mass density of the briquettes at the expense of their combustion properties. The effect of hydrothermal pretreatment and binder wt% on the properties of the resulted sawdust briquettes was investigated and the results were compared with briquettes formed without pretreatment and binder addition. The physical and combustion characteristics of the briquettes were critically analyzed based on density, MC, VM, AC, FC content and HHV.

HPT was beneficial to the physical properties of the resulted biomass briquettes. The densities of the untreated briquettes B_{00} (573.34kg/m^3), B_{01}(638.66kg/m^3), and B_{02}(751.76kg/m^3) were lower when compared to the HPT briquettes. HPT also improved the combustion characteristics by lowering AC, FC content and VM of the resulted biomass briquettes. The HHV had also increased after HPT.

The optimum operating conditions were determined to be HPT = 320^{O}C and binder wt% = 0%; the response variables at this optimum are as follows: density = 747.47kg/m^3, durability = 99.99, MC = 9.61%, VM = 52.2%, AC = 2.87%, FC = 35.4%, HHV = 20.74 MJ/Kg

This study showed that HPT of sawdust and molasses binder addition improves the physical and combustion properties of briquettes and also bypass the raw material drying unit operation.

5.2 Recommendations

Incorporating HPT in biomass briquette production enhances quality of the briquettes. However, energy balance needs to be performed and efficient autoclaves need to be utilized prior to scale up in order to minimize production cost.

Design and construction of efficient carbonizer should be done in order to carbonize the briquettes and further enhance the combustion properties of the HPT briquettes.

More research on the effects of HPT on biomass other than sawdust should be done in order to utilize other biomass waste for briquette production.

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7. ANNEX

Annex 1: Analysis of variance for durability

Table 10: ANOVA for the quadratic model of the durability model of briquettes

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	725.04	5	145.01	104.97	< 0.0001	significant
A-Temperature	419.51	1	419.51	303.69	< 0.0001	
B-Binder wt%	82.58	1	82.58	59.78	0.0002	
AB	97.01	1	97.01	70.23	0.0002	
A^2	30.31	1	30.31	21.94	0.0034	
B^2	0.9322	1	0.9322	0.6748	0.4428	
Residual	8.29	6	1.38			
Cor Total	733.33	11				

- The Model F-value of 104.97 indicates that the model is statistically significant. An F-value of this magnitude has a 0.01 percent chance of occurring as a result of noise.
- Model terms with P-values less than 0.05 are considered statistically significant. A, B, AB, and A^2 are important model terms in this situation. The model terms are insignificant if the value is greater than 0.100.

Table 11: Fit statistics for durability model

Std. Dev.	**1.18**		**R^2**	**0.9887**		**Adeq Precision**	**30.3986**
Mean	95.92		**Adjusted R^2**	0.9793			
C.V. %	1.23		**Predicted R^2**	0.9011			

- The **Predicted R^2** of 0.9011 is in reasonable agreement with the **Adjusted R^2** of 0.9793; i.e., the difference is less than 0.2.
- **Adeq Precision** measures the signal to noise ratio. A ratio greater than 4 is desirable. It is preferable to have a ratio of more than 4. In this case the signal-to-noise ratio of 30.399 is adequate.

Table 12: coefficients in terms of coded factors for durability model

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	97.54	1	1.04	94.99	100.08	
A-Temperature	8.07	1	0.4631	6.94	9.20	1.13
B-Binder wt%	3.46	1	0.4478	2.37	4.56	1.16
AB	-4.48	1	0.5340	-5.78	-3.17	1.16
A^2	-5.33	1	1.14	-8.11	-2.55	1.13
B^2	-0.5912	1	0.7197	-2.35	1.17	1.0000

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multi-collinearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.

$$Durability = 97.54 + 8.07A + 3.46B - 4.48AB - 5.33A^2 - 0.5912B^2 \quad (10)$$

The given mathematical equation was calculated as the sum of a constant, first order effects, second order effects, and interaction effects for the response variable (durability) as a function of HPT temperature (A) and binder wt percent (B).

Annex 2: Analysis of variance for MC

Table 13: ANOVA for quadratic model of Moisture content

Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	
Model	12.62	5	2.52	112.15	< 0.0001	significant
A-Temperature	0.7237	1	0.7237	32.17	0.0013	
B-Binder wt%	5.13	1	5.13	228.00	< 0.0001	
AB	0.0221	1	0.0221	0.9836	0.3596	
A^2	4.24	1	4.24	188.31	< 0.0001	
B^2	0.0000	1	0.0000	0.0000	1.0000	
Residual	0.1350	6	0.0225			
Cor Total	12.75	11				

- The Model F-value of 112.15 indicates that the model is statistically significant. An F-value of this magnitude has a 0.01 percent chance of occurring as a result of noise.
- Model terms with P-values less than 0.05 are considered statistically significant. A, B, and A^2 are important model terms in this situation. The model terms are insignificant if the value is greater than 0.100.

Table 14: Fit statistics for Moisture content model

Std. Dev.	**0.1500**		**R^2**	**0.9894**	**Adeq Precision**	**35.4182**
Mean	10.92		**Adjusted R^2**	0.9806		
C.V. %	1.37		**Predicted R^2**	0.9637		

The **Predicted R^2** of 0.9637 is in reasonable agreement with the **Adjusted R^2** of 0.9806; i.e., the difference is less than 0.2.

Adeq Precision measures the signal to noise ratio. It is preferable to have a ratio of more than 4. In this case the signal-to-noise ratio of 35.418 is adequate. The design space can be navigated using this model.

Table 15: Coefficient in terms of coded factors for Moisture content model

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	12.22	1	0.1329	11.90	12.55	
A-Temperature	0.3352	1	0.0591	0.1906	0.4798	1.13
B-Binder wt%	0.8629	1	0.0571	0.7230	1.00	1.16
AB	-0.0676	1	0.0681	-0.2343	0.0992	1.16
A^2	-1.99	1	0.1452	-2.35	-1.64	1.13
B^2	0.0000	1	0.0918	-0.2247	0.2247	1.0000

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multi-collinearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.

$$MC = 12.22 + 0.3352\,A + 0.8629B - 0.0676AB - 1.99A^2 \qquad (11)$$

The given mathematical equation was calculated as the sum of a constant, first order effects, second order effects, and interaction effects for the response variable (moisture content) as a function of HPT temperature (A) and binder wt percent (B).

Annex 3: Analysis of variance for VM

Table 16:ANOVA for quadratic model of Volatile matter

Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	
Model	1003.17	5	200.63	19.37	0.0012	significant
A-Temperature	971.90	1	971.90	93.84	< 0.0001	
B-Binder wt%	1.62	1	1.62	0.1568	0.7058	
AB	6.30	1	6.30	0.6087	0.4649	
A^2	230.75	1	230.75	22.28	0.0033	
B^2	0.0000	1	0.0000	0.0000	1.0000	
Residual	62.14	6	10.36			
Cor Total	1065.31	11				

- The Model F-value of 19.37 indicates that the model is statistically significant. An F-value of this magnitude has a 0.01 percent chance of occurring as a result of noise.
- Model terms with P-values less than 0.05 are considered statistically significant. A and A^2 are important model terms in this situation. The model terms are insignificant if the value is greater than 0.100.

Table 17:Fit statistics for Volatile matter model

Std. Dev.	**3.22**		**R^2**	**0.9417**		**Adeq Precision**	**11.7980**
Mean	64.52		**Adjusted R^2**	0.8931			
C.V. %	4.99		**Predicted R^2**	0.8416			

- The **Predicted R^2** of 0.8416 is in reasonable agreement with the **Adjusted R^2** of 0.8931; i.e., the difference is less than 0.2.
- It is preferable to have a ratio of more than 4. In this case the signal-to-noise ratio of 11.798 is adequate. The design space can be navigated using this model.

Table 18:Coeficients in terms of coded factors for Volatile matter

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	78.70	1	2.85	71.72	85.67	
A-Temperature	-12.28	1	1.27	-15.39	-9.18	1.13
B-Binder wt%	-0.4855	1	1.23	-3.49	2.51	1.16
AB	1.14	1	1.46	-2.44	4.72	1.16
A^2	-14.70	1	3.11	-22.32	-7.08	1.13
B^2	0.0000	1	1.97	-4.82	4.82	1.0000

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multi-collinearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.

$$Volatile\ matter\ =\ 78.7 - 12.28A - 0.4855B - 1.14AB - 14.7A^2 \qquad (12)$$

The given mathematical equation was calculated as the sum of a constant, first order effects, second order effects, and interaction effects for the response variable (volatile matter) as a function of HPT temperature (A) and binder wt percent (B).

Annex 4: Analysis of variance for AC

Table 19:ANOVA for quadratic model of Ash content

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	11.41	5	2.28	52.32	< 0.0001	significant
A-Temperature	0.5177	1	0.5177	11.87	0.0137	
B-Binder wt%	4.69	1	4.69	107.56	< 0.0001	
AB	0.0068	1	0.0068	0.1559	0.7066	
A^2	3.69	1	3.69	84.61	< 0.0001	
B^2	0.0035	1	0.0035	0.0803	0.7864	
Residual	0.2617	6	0.0436			
Cor Total	11.67	11				

- The Model F-value of 52.32 indicates that the model is statistically significant. An F-value of this magnitude has a 0.01 percent chance of occurring as a result of noise.
- Model terms with P-values less than 0.05 are considered statistically significant. A, B, and A^2 are important model terms in this situation. The model terms are insignificant if the value is greater than 0.100.

Table 20:Fit statistics for ash content

Std. Dev.	0.2089		R^2	0.9776		Adeq Precision	23.3716
Mean	3.49		**Adjusted R^2**	0.9589			
C.V. %	5.99		**Predicted R^2**	0.9368			

- The **Predicted R^2** of 0.9368 is in reasonable agreement with the **Adjusted R^2** of 0.9589; i.e., the difference is less than 0.2.
- It is preferable to have a ratio of more than 4. In this case the signal-to-noise ratio of 23.372 is adequate. The design space can be navigated using this model.

Table 21:coeficients in terms of coded factors for ash content

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	2.24	1	0.1850	1.79	2.70	
A-Temperature	-0.2835	1	0.0823	-0.4848	-0.0821	1.13
B-Binder wt%	0.8253	1	0.0796	0.6306	1.02	1.16
AB	0.0375	1	0.0949	-0.1947	0.2697	1.16
A^2	1.86	1	0.2021	1.36	2.35	1.13
B^2	0.0363	1	0.1279	-0.2767	0.3492	1.0000

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multi-collinearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.

$$Ash\ content = 2.24 - 0.2835A + 0.8253B + 0.0375AB + 1.86A^2 + 0.0363B^2 \quad (13)$$

The given mathematical equation was calculated as the sum of a constant, first order effects, second order effects, and interaction effects for the response variable (Ash content) as a function of HPT temperature (A) and binder wt percent (B).

Annex 5: Analysis of variance for FC

Table 22:ANOVA for quadratic model of Fixed carbon

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	1015.58	5	203.12	23.49	0.0007	significant
A-Temperature	963.59	1	963.59	111.46	< 0.0001	
B-Binder wt%	9.96	1	9.96	1.15	0.3244	
AB	5.98	1	5.98	0.6917	0.4374	
A^2	234.95	1	234.95	27.18	0.0020	
B^2	0.0031	1	0.0031	0.0004	0.9854	
Residual	51.87	6	8.65			
Cor Total	1067.45	11				

- The Model F-value of 23.49 indicates that the model is statistically significant. An F-value of this magnitude has a 0.01 percent chance of occurring as a result of noise.
- Model terms with P-values less than 0.05 are considered statistically significant. A, A^2 are important model terms in this situation. The model terms are insignificant if the value is greater than 0.100.

Table 23:Fit statistics for fixed carbon

Std. Dev.	**2.94**		**R^2**	**0.9514**		**Adeq Precision**	**12.9220**
Mean	21.07		**Adjusted R^2**	0.9109			
C.V. %	13.96		**Predicted R^2**	0.8645			

- The **Predicted R^2** of 0.8645 is in reasonable agreement with the **Adjusted R^2** of 0.9109; i.e., the difference is less than 0.2.
- It is preferable to have a ratio of more than 4. In this case the signal-to-noise ratio of 12.922 is adequate. The design space can be navigated using this model.

Table 24:coeficients in terms of coded factors for fixed carbon

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	6.84	1	2.60	0.4624	13.21	
A-Temperature	12.23	1	1.16	9.40	15.07	1.13
B-Binder wt%	-1.20	1	1.12	-3.94	1.54	1.16
AB	-1.11	1	1.34	-4.38	2.16	1.16
A^2	14.84	1	2.85	7.87	21.80	1.13
B^2	-0.0342	1	1.80	-4.44	4.37	1.0000

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multi-collinearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.

$$Fixed\ carbon = 6.84 + 12.23\,A - 1.20B - 1.11AB + 14.84A^2 - 0.0342B^2 \quad (14)$$

The given mathematical equation was calculated as the sum of a constant, first order effects, second order effects, and interaction effects for the response variable (Fixed carbon) as a function of HPT temperature (A) and binder wt percent (B).

Annex 6: Analysis of variance for HHV

Table 25:ANOVA for quadratic model of HHV

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	3.31	5	0.6619	47.00	< 0.0001	significant
A-Temperature	0.9458	1	0.9458	67.15	0.0002	
B-Binder wt%	2.14	1	2.14	151.76	< 0.0001	
AB	0.0918	1	0.0918	6.52	0.0433	
A^2	0.4932	1	0.4932	35.02	0.0010	
B^2	6.378E-08	1	6.378E-08	4.529E-06	0.9984	
Residual	0.0845	6	0.0141			
Cor Total	3.39	11				

- The Model F-value of 47.00 indicates that the model is statistically significant. An F-value of this magnitude has a 0.01 percent chance of occurring as a result of noise.
- Model terms with P-values less than 0.05 are considered statistically significant. A, B, AB, and A^2 are important model terms in this situation. The model terms are insignificant if the value is greater than 0.100.

Table 26:Fit statistics for HHV

Std. Dev.	0.1187		R^2	0.9751		Adeq Precision	22.4079
Mean	20.00		**Adjusted R^2**	0.9544			
C.V. %	0.5935		**Predicted R^2**	0.8600			

- The **Predicted R^2** of 0.8600 is in reasonable agreement with the **Adjusted R^2** of 0.9544; i.e., the difference is less than 0.2.
- It is preferable to have a ratio of more than 4. In this case the signal-to-noise ratio of 22.408 is adequate. The design space can be navigated using this model.

Table 27:Coeficients in terms of coded factors for HHV model

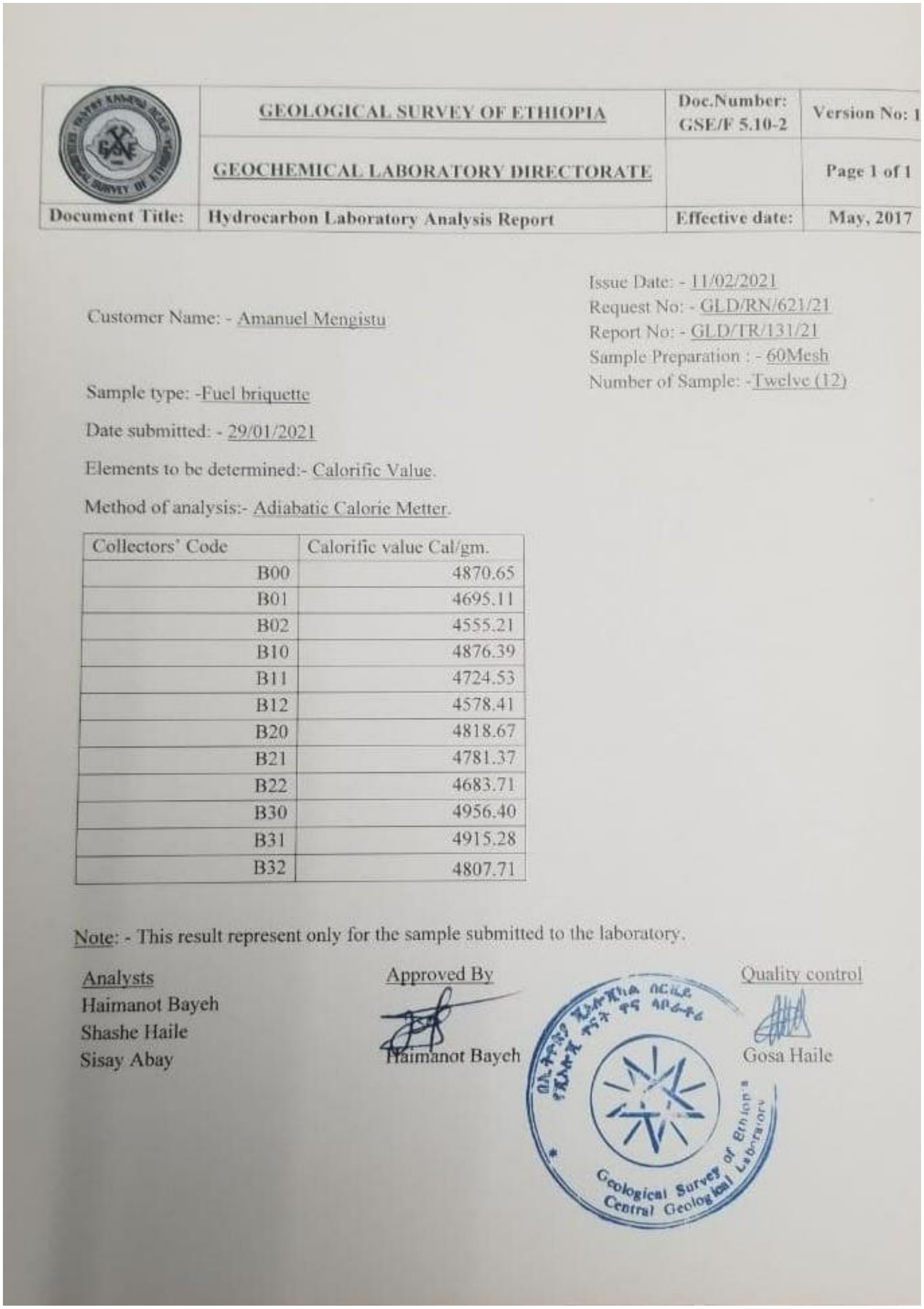
Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	19.40	1	0.1051	19.14	19.66	
A-Temperature	0.3832	1	0.0468	0.2688	0.4976	1.13
B-Binder wt%	-0.5570	1	0.0452	-0.6677	-0.4464	1.16
AB	0.1377	1	0.0539	0.0057	0.2696	1.16
A^2	0.6797	1	0.1149	0.3987	0.9608	1.13
B^2	0.0002	1	0.0727	-0.1777	0.1780	1.0000

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multi-collinearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.

$$HHV = 19.4 + 0.3832A - 0.5570B + 0.1377AB + 0.6797A^2 + 0.0002B^2 \qquad (15)$$

The given mathematical equation was calculated as the sum of a constant, first order effects, second order effects, and interaction effects for the response variable (High heating value) as a function of HPT temperature (A) and binder wt percent (B).

Annex 7: HHV values of briquettes from the laboratory of Geological survey of Ethiopia



	GEOLOGICAL SURVEY OF ETHIOPIA	Doc.Number: GSE/F 5.10-2	Version No: 1
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Document Title:	Hydrocarbon Laboratory Analysis Report	Effective date:	May, 2017

Issue Date: - 11/02/2021
Request No: - GLD/RN/621/21
Report No: - GLD/TR/131/21
Sample Preparation : - 60Mesh
Number of Sample: -Twelve (12)

Customer Name: - Amanuel Mengistu

Sample type: -Fuel briquette

Date submitted: - 29/01/2021

Elements to be determined:- Calorific Value.

Method of analysis:- Adiabatic Calorie Metter.

Collectors' Code	Calorific value Cal/gm.
B00	4870.65
B01	4695.11
B02	4555.21
B10	4876.39
B11	4724.53
B12	4578.41
B20	4818.67
B21	4781.37
B22	4683.71
B30	4956.40
B31	4915.28
B32	4807.71

Note: - This result represent only for the sample submitted to the laboratory.

Analysts
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Shashe Haile
Sisay Abay

Approved By
Haimanot Bayeh

Quality control
Gosa Haile

Figure 14: HHV values of briquettes