Analysis of Physico-Chemical Properties of Christ Thorn Jujube Seeds (*Ziziphus Spina Christi*) Bioethanol-Gasoline Blends

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Abstract - Establishing sustainable and possible renewable fuel substitutes for gasoline has become necessary, most especially with the recent fuel subsidy removal in Nigeria which has significantly affected the economy and increases the cost of living. In line with this, production of bioethanol from Christ thorn jujube seeds was carried out successfully with average percentage yield of 52%. The produced bioethanol was blended in gasoline and investigation into the physicochemical properties of the bioethanol and the blends were carried out. From the results obtained the density and the specific gravity of the bioethanol were 777.2kg/m³ and 0.7772 and the blends increases as the percentage of the bioethanol increased and range between 752.4 – 760.8 kg/m³ and 0.7524 – 0.7608 respectively. The kinematic viscosity of the bioethanol produced was 1.64cSt and the blends also increases as the percentage of the bioethanol increases in the gasoline and range between 0.79 – 0.93cSt. The flash points, the pour points and the cloud points all increases with increased in the bioethanol content in the blends and they were all within the acceptable range. The heating value of the gasoline upon blending with the bioethanol decreases though, with an advantage of increasing the oxygen content of the blends which may improve the combustion process. The heating value of the gasoline decreases from 44.40 - 44.21MJ/kg upon blending with 5% bioethanol (sample B5). The boiling point (78.5°C) of the bioethanol obtained was within the acceptable storage and handling range of 78°C. The PH value of sample B20 was lower than the acceptable value, thus it cannot be use as fuel for spark ignition engine. The physicochemical properties of the bioethanol produced from Christ thorn jujube seeds suggest possibility of running an engine test for performance and emission level evaluations to ascertain it usage as a blend.

Indexed Terms- Physicochemical properties, Christ thorn jujube seeds, Bioethanol, Gasoline, Density, Flash point, Cloud Point and Calorific value.

I. INTRODUCTION

1.1 Background of the Study

Access to inexpensive and reliable energy is essential to reducing poverty and improving health, productivity and enhancing competitiveness. Energy is crucial for the sustainability of modern societies and its uses are inevitable for human survival. It is the essential ingredient for socio economic development and hence, is a true indicator for economic growth. Thus, energy all over the world still remained an important role in the development and growth of a nation. However, the world depends so much on fossil fuels to meet its energy requirements of which it emits pollutants to the environment and will be exhausted in the near future. According to [1] about 80% of the total global energy consumption were energy from the fossil fuels. [2] reported that around 75 % of the fossil fuels are used for heat and energy production, about 20 % as fuel, and just a few percent for the production of chemicals and materials. Renewable energy (solar, hydro, biomass and geothermal) and nuclear power energy contribute 13.5% and 6.5% of the total energy respectively [1]. However, according to a study by [3] about 40% of the total energy consumption is dedicated to transport which largely require liquid fuels such as gasoline, diesel or kerosene which are all sourced from crude oil.

In most developed countries, governments encourage the use of renewable energy resources with the following major goals: (*i*) to secure access to energy, (*ii*) to mitigate climate changes, (*iii*) to develop/maintain agricultural activities and (*iv*) to ensure food safety. Affordable energy, climate change and social stability, as the three pillars of sustainability, are directly related to the above-mentioned major goals [2]. These will greatly alter the current of global warming and all fossil-based problems by replacing fossil fuels with renewable resources, which are to some extent uniformly distributed and cause fewer environmental and social concerns.

Recent survey suggests that there was an enormous interest in the production and usage of liquid biofuels (biodiesel or bioethanol) as promising substitutes for fossil fuels. Bioethanol is considered one of the most fascinating biofuels due to its encouraging impact on the environment and can be used directly in vehicles or blended with the gasoline, thereby reducing greenhouse gas emission and fuel consumption. The blending of bioethanol with gasoline might not require modifying the engine; rather it will help to enhance ignition or engine performance. Also, with the current fuel subsidy removal in Nigeria which significantly affected the economy and the already known environmental consequences of using fossil fuels, necessitates the search for alternative fuels most especially for use in spark ignition engines. Thus, the need to investigate the physicochemical properties of bioethanol extracted from Christ thorn (*Ziziphus Spina Christi*) jujube seeds as an alternative feedstock in spark ignition engines.

Many researches have been carried out on the use of plants and their seeds for the production of biofuels. Bioethanol Production from lignocellulose raw materials is sustainable, fascinating and attractive because lignocellulosic biomass is renewable and non-competitive with food crops. It brings considerable reduction in greenhouse gas emission, almost equally distributed on the Earth and it can be obtained from different residues or directly harvested from forest, with virtually no or lower price. The average lignocellulosic biomass contains 43 % cellulose, 27 % lignin, 20 % hemicellulose and 10 % other components. Effective means of enzymatic hydrolysis of lignocellulosic biomass most be insured and the choice of pretreatment to a larger extent depends on the nature of the raw material and the formation of byproducts during the selected pretreatment, and its choice has a large impact on all subsequent stages in the bioethanol production [2].

According to research on assessment of bioethanol production from Christ thorn fruit's pulp and seed conducted by [3], the carbohydrate composition for a continuous fermentation time of 96 hours of both the pulp and seed were 49.7% and 25.74% respectively. The percentage yield of the bioethanol produced was not clearly stated, though, the concentration was found to increase steadily with fermentation times. This implies that that the sugar is being fermented by the activity of the selected enzyme (yeast; *saccharomyces cerevisae*). Other physicochemical properties such as calorific value, density of the fuel, kinematic viscosity, pour point, cloud point and octane number which are essential for automotive vehicles were not characterized in the work.

[4] also worked on performance and emission analysis of Indian jujube seeds oil as biodiesel, the research found that, the combustion parameters for the biodiesel and its blends gave the best result when compared with standalone diesel. The emission analysis for the biodiesel and its blend gave the best result when compared to the sole fuel. [1] produced and characterized bioethanol from Snot Apple (*Azanzagarckeana*). The results showed a percentage yield of 52% and other physicochemical properties (density, specific gravity, kinematic viscosity, pour point, cloud point, and octane number) were appreciably higher in comparison with that of gasoline. The work concluded that the bioethanol produced can be blended comfortably with gasoline.

1.2 Christ Thorn (*Ziziphus Spina Christi*) Jujube seeds

The plant Christ thorn (*Ziziphus spina-christi*) jujube seeds are mostly found in abundant across the Sub-Saharan African and Sahel regions and according to [3] this extend from Senegal, Sudan and Arabia where the annual rainfall is about 50 – 300mm or on periodically inundated sites. It grows in dry conditions and are conspicuously thorny, often the thorns in pairs one being straight and other curved. It has numerous small, sometimes minute teeth like leaves [5]. The leaves and the seeds are presented in plates I (a and b).


(a)


(b)

Plate I: Christ Thorn Jujube Leaves and Seeds

1.3 Production of Bioethanol

A wide range of technologies are available to separate biomass resources into their building blocks, like carbohydrates, proteins, fats, *etc.* The plant that produces lignocellulose-containing raw materials could be a good example of biorefinery concept where cellulose and hemicellulose produce simple (fermentable) sugars and lignin produces target compounds (*e.g.,* polymers, resins, pesticides, levulinic acid and other materials) [2]. Further studies by [2] reported that the biorefinery process usually comprises the following stages: pretreatment and preparation of biomass, separation of biomass components and subsequent conversion and product purification steps.

Bioethanol, as an alternative to the fossil fuels, is mainly produced by yeast fermentation from different feedstocks. It is a high-octane number fuel and its physicochemical features are considerably different compared to the gasoline [2]. Ethanol is a member of a group of organic chemicals called alcohols. Ethanol has the chemical formula of CH_3CH_2OH, with the $-OH$ (hydroxyl group) being the functional group [3]. Because of the presence of the $-OH$ hydrogen bonding is quite common. Ethanol is a primary alcohol since the hydroxyl group is attached to a carbon atom which is itself attached to only one other carbon atom. Ethanol is a clear, colourless liquid with a characteristic of sweet smell.

II. MATERIALS AND METHODS

2.1 Production of Bioethanol from Christ Thorn Jujube Seed

The production of bioethanol from Christ thorn jujube seeds includes the following process: collection and preparation of the Christ thorn jujube seeds, grinding and removal of the pulp from the seeds into powdered form, hydrolysis, fermentation, filtration, distillation and characterization of the produced bioethanol.

2.1.1 Collection and preparation of the Christ thorn jujube seeds

The Christ thorn jujube seeds as shown in Plate Ib, is readily available in local market and also can be collected directly from the bush, however, this was obtained from Yelwa market, Bauchi state, Nigeria and about Two and half $(2.5kg)$ was used for the purpose of this research work. In preparing the seeds, it was washed to remove the dirt and then sun dried properly to ensure that the moisture content was removed. Mortar and piston were used to mill the sample in order to separate the pulp from the seed as shown in plate II. The milled sample was then sieved using a 3mm size sieve and subsequently used for the production of the bioethanol.

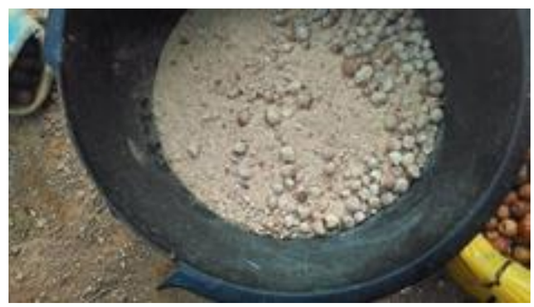

Plate II: Milled Christ Thorn Jujube Seeds

2.1.2 Fermentation process

From the mass of the grinded sample, 1kg was put into a rubber gallon and hydrolyzed with 0.1M dilute sulphuric acid for an hour, after which it was washed in distilled water and filtered for fermentation process. This is to solubilize the hemicellulose, thereby making the cellulose more accessible to enzymes [2]. About 40g of yeast (yeast; *saccharomyces cerevisiae*) was added and mixed thoroughly with 2,500ml of water in a plastic container. The container was sealed to ensure no oxygen enters or gases comes out from the container. The process lasted for 84 hours to ensure complete fermentation has taken place. After 84 hours the sample was filtered and sieved to remove the sediments and yeast cells. 1800cm^3 was obtained from the fermented solution for production of bioethanol, hence, the filtrate of the sample are the dilute solution of bioethanol and other organic substance which was distilled at temperature range of 60-80^{o}C as shown in plate III. The distilled ethanol was purified by redistilling it to obtained pure ethanol and later was analyzed to determine the physicochemical properties of the ethanol produced. The percentage yield of the bioethanol in the sample was determined using equation 1 below;

$$\%\ \text{yield of bioethanol} = \frac{volume\ of\ ethanol\ produced}{volume\ of\ sample} \times 100\% \quad (1)$$

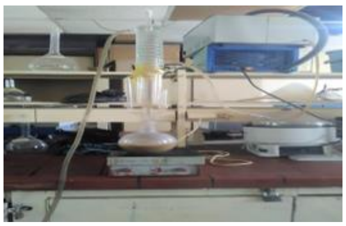

Plate III: Filtration of Bioethanol

2.2 Physicochemical Analysis

The following tests were conducted to determine the physicochemical properties of the produced bioethanol and its blends. The formulation of the blends (gasoline-Christ thorn jujube seeds bioethanol) on volume basis were 0%, 5%, 10%, 15% and 20% as shown in Table 1 below. B0 means 100% gasoline, B100 indicating 100% bioethanol, B5 means 5% bioethanol and 95% gasoline blend etc.

Table 1: Formulation of the Gasoline-Christ Thorn Jujube Seeds Bioethanol Blends

S/No	Sample	Bioethanol (%)	Gasoline (%)
1	B0	0	100
2	B100	100	0
3	B5	5	95
4	B10	10	90
5	B15	15	85
6	B20	20	80

2.2.1 Determination of density

Density which is the mass per unit volume of the liquid bioethanol (kg/m^3), was obtained by employing the ASTM standard D-1217 methods. This was done by obtaining the weight of a 25ml empty bottle using the electronic weighing balance, and the weight of the 25ml bottle filled with bioethanol sample. The density was determined using equation 2.

$$Density = \frac{mass}{volume} = \frac{W_2 - W_1}{V} \quad (2)$$

Where W_1 = weight of empty bottle
W_2 = weight of ethanol and bottle
V = volume of the ethanol

2.2.2 Determination of specific gravity (S.G)

The specific gravity is the density of a substance to the density of water at a specific temperature, this is according to ASTM standard D-1217. It was determined using equation 3 below;

$$S.G = \frac{Density\ of\ ethanol}{Density\ of\ water} \quad (3)$$

where density of water = 1000kg/m^3

2.2.3 Kinematic viscosity

This is the measure of resistive flow of a fluid subjected to gravity. This can be measured using ASTM standard D-446 in which a capillary viscometer graduated long tube with a stopper at the lower end to prevent the liquid from flowing out is used. The distance between the two markers in the tube was recorded and the time taken for the sphere to travel from the first marker to the second marker was also recorded. It was to ensured that the ethanol was pour above the marker so as to allow the sphere to achieve terminal velocity. A stop watch was provided to record the time taken for steel sphere to go down the length of the tube when released. However, the formula for determining the viscosity of the sample is presented in equation 4 below [1].

$$Viscosity\ (\mu) = \frac{2(\Delta\rho\ gr^2)}{9V} \quad (4)$$

Where μ = dynamic viscosity of the ethanol produced
ρ = density of the ethanol produced
g = acceleration due to gravity
r = radius of sphere
V = terminal velocity of the sample was poured into the long tube

2.2.4 Calorific value

This is the energy contained in a fuel and is determined by using ASTM standard D-240. This is to measure the heat produced by complete combustion of a specific quantity of the fuel in a calorimeter. The quantity of heat generated is expressed in joules per kilogram (J/kg). To measure the quantity of energy in the produced bioethanol, a specific quantity was poured in the capsule and a fuse wire was inserted in the sample and placed in the bomb calorimeter. The bomb then charged with oxygen from the oxygen cylinder and covered. The calorimeter bucket then filled with 2 liters of water. Subsequently, the bomb was drop in other word lowered partially in the bucket. The two ignition lead wires were then pushed into the terminals of the bomb head. The wires were oriented away from the stirrer shaft so that they do not tangle. The calorimeter cover was closed and as the fuel is being combusted, the calorific values were read.

2.2.5 Determination of flash point

ASTM D-93 standard method was adapted in measuring the flash point of the samples. A conical flask 150ml with an opening and a cork for inserting the thermometer was put in place. The tip of the thermometer was inserted in such a way that it did not touch the bottom of the flask. The flask was then filled with 10ml of fuel sample and was heated on a hot plate to the lowest temperature at which the vapors leaving the flask opening ignites.

2.2.6 Determination of pour point

ASTM D-2500 standard method of measuring pour point was adapted. Here, a portion of the sample was poured into a test tube and the mercury point of the thermometer was inserted in the test tube. The set up was inserted in a beaker containing ice and left to solidify. When the solidification was confirmed, the test tube was removed and tilted and closely observed till it started to flow. The lowest temperature at which the sample was observed to flow was recorded as the pour point.

2.2.7 Determination of a cloud point

The same ASTM standard used in measuring pour point was also adapted here. A portion of the sample was poured into a test tube and the mercury point of the thermometer was inserted in the test tube. The set up was inserted in a beaker containing ice. The sample was observed closely until it started forming a cloud gel. The temperature was recorded as the cloud point.

III. RESULTS AND DISCUSSION

3.1 Results

3.1.1 Percentage yield

The percentage yield of the bioethanol was obtained by taking average of the three treatments conducted. From the 1800cm^3 fermented solution of the Christ thorn jujube seeds, 500cm^3 of the solution was measured for each of the treatment and used for distillation process. The bioethanol produced were 264cm^3, 258cm^3 and 250cm^3 respectively and the percentage yield computed using equation 1 are presented in table 2 below.

Table 2: Percentage yield of Christ thorn jujube seeds Bioethanol

S/No	Sample Use (cm^2)	Ethanol Produced	% Yield
1	500	264	53
2	500	258	52
3	500	250	50
Mean	500	257	52

3.1.2 Physicochemical properties

The physicochemical properties of Christ thorn jujube seeds bioethanol and its blends are presented in table 3 below. From the results, the density of the bioethanol appeared to be higher than that of the gasoline by 1.06% and upon blending, it increases the density of the gasoline. The higher the percentage of the bioethanol in the blend the higher was the blends' density. Fuel density according to [6] increases with increasing molecular weight of the fuel which increases the overall performance of engines [7], [8] and [6]. An analysis of premium motor spirit (PMS) distributed in Lagos metropolis, Nigeria was carried out by [9] and found out that the density falls within 743.6 – 782 kg/m^3. This values when compare to what were obtained for both bioethanol and its blends were within the operating range. Upon blending, the density increases with increase in bioethanol content. Thus, it is expected to improve the performance of the engine.

The specific gravity of the bioethanol produced was 0.7772, higher than that that of petrol (0.7360) and the blends keep on increasing as the bioethanol content increases in the blend. This value is also comparable to one obtained by [3] from the same Christ thorn jujube seeds bioethanol (0.821) and 0.785 from agricultural waste obtained by [6]. This scenario was expected as the bioethanol is known to be heavier than gasoline. In fact, this is the reason why when a mixture of the two is viewed inside a transparent container; bioethanol is seen to settle at the middle sandwiching gasoline on top and water at the bottom. However, all the samples specific heat capacity falls within the acceptable limit of 0.7500 to 0.8500, recommended by the ASTM standards [10].

Recent review by [11] and [12] suggest that the kinematic viscosity is temperature dependent; it decreases as temperature increases and for gasoline at 40ºC the viscosity was 0.72cSt based on Research Octane Number 95 (RON95 – most standard fuels in Europe are based on RON 95). Trost et al. (2021), also worked on RON98 and discovered that the kinematic viscosity was lower than RON95 at 40ºC (0.69cSt). Similarly, [13] reported that the standard of viscosity for bioethanols is 1.525cSt. From the results in Table 3 the kinematic viscosity of Christ thorn jujube seeds obtained at 35ºC was 1.64cSt and its blends range between 0.79 - 0.93cSt. It was observed that, as the bioethanol content in the blend increases, so also the viscosity of the blend increases. This was as a result of the higher viscosity of the bioethanol. This will greatly influence the fuel atomization quality and size of the fuel droplet in the spray, reducing drag and incomplete combustion of fuel.

Also tested for is the flash point of the produced bioethanol. This described the potential of fuels to catch fire and explosion hazards in liquids [10]. Also [10] stated that liquids with flash points which are less than 37.8ºC are referred to as flammable and combustible liquids. The standard flash point of bioethanol is 12ºC, and the one obtained from this work (as shown in Table 3) was 13.7ºC which can also be characterized as flammable and combustible fuel. However, that of gasoline according to ASTM D93A and [1] is -43ºC. The blend shows that as the bioethanol content increases the flash point decreases.

The pour and cloud points are properties of fuel that are of great importance in identifying at what level the fuel change phase due to paraffin content. [14] quoted 5.2ºC and 23ºC as pour and cloud points ASTM standard values for bioethanol respectively. The result presented in Table 3 showed that the experimental bioethanol has a good pour and cloud points values of 6.3°C and 22.5ºC respectively. These were close to the standard reported by [14]. The blends of the pour points range between -7.6ºC to - 6.0ºC and the cloud points range between 20.4ºC - 21.3ºC. The obtained cloud point is high enough not to cause fuel filter clogging [15].

The calorific value of bioethanol obtained from the Christ thorn jujube seeds was found to be 27.4 MJ/kg which was lower than that of gasoline (44.4 MJ/kg). This could be attributed to the chemical composition in the fuel and probably fraction of water content which might be present after distillation process. Consequently, this lowered the heating values of the blends as the percentage of the bioethanol increases (44.22 - 43.23MJ/kg) as shown in Table 3. The higher the percentage of the bioethanol, the lower the heating value of the blends. This behavior was also reported in [6]. However, this disadvantage could be up sated by the presences of oxygen in the bioethanol which may improve the combustion by pushing the process towards complete combustion [16].

Table 3: Physicochemical Properties of Christ Thorn Jujube Seeds Bioethanol and its Blends

Property	B0	B100	B5	B10	B15	B20
Density (kg/m^3)	746	777.2	752.4	758.7	760.2	760.8
Specific gravity	0.746	0.7772	0.7524	0.7587	0.7602	0.7608
Kinematic Viscosity (cSt)	0.72	1.64	0.79	0.83	0.88	0.93
Flash Point (º C)	-43	13.7	-36	-30	-27	-22
Pour Point (º C)	-8	6.3	-7.6	-7.1	-6.5	-6.0

Cloud Point (° C)	20	22.5	20.4	20.9	21.0	21.3
Calorific value (MJ/kg)	44.40	27.4	44.21	43.95	43.84	43.23
Boiling Point (° C)	61	78.5	-	-	-	-
PH	7	6.6	6.8	6.7	6.5	6.2
Color	Pale Yellow	Colorless	-	-	-	-

CONCLUSION

Production of bioethanol from Christ thorn jujube seeds was carried out successfully with average percentage yield of 52%. The bioethanol produced was colourless and blending with gasoline was achieved with ease. The physicochemical properties of the Christ thorn jujube seeds obtained were within their respective standards. The density and the specific gravity of the blends increases as the percentage of the bioethanol increased and range between 752.4 – 760.8 kg/m^3 and 0.7524 – 0.7608 respectively. The kinematic viscosity of the blends also increases as the percentage of the bioethanol increases and range between 0.79 – 0.93cSt. The flash point, the pour point and the cloud point all increases with increased in the bioethanol content. The heating value of the gasoline upon blending it with the bioethanol decreases though, with an advantage of increasing the oxygen content of the blend which may improve the combustion process. The heating value of the gasoline decreases from 44.40 - 44.21MJ/kg upon blending with 5% bioethanol. The boiling point (78.5^{o}C) of the bioethanol obtained was within the acceptable storage and handling range of 78^{o}C. The PH value of sample B20 was lower than the acceptable value, thus it cannot be use as fuel for spark ignition engine. The physicochemical properties of the bioethanol produced from Christ thorn jujube seeds suggest possibility of running an engine test for performance and emission level evaluation to ascertain it usage as a blend.

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