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STUDY OF PHYSIO-CHEMICAL PROPERTIES, PROXIMATE AND ULTIMATE ANALYSIS OF BIODIESEL EXTRACTED FROM THREE FEED-STOCKS- MELIA AZEDARACH, RICE BRAN AND WATER HYACINTH

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ABSTRACT
After extracting oil from Melia azedarach, Rice bran, and Water hyacinth, their physio-chemical properties are tested which are observed to fulfill the protocol of ASTM Standard for making bio-diesel from these three feedstocks. Melia azedarach, Rice bran, and Water hyacinth bioactive compounds were assayed for estimating the property of elemental analysis (CHNS) and proximate analysis. The percentage composition for nitrogen, carbon, hydrogen, and sulfur is found to be 2.309%, 45.531%, 6.574%, and 0.732% with retention time (minutes) of 0.858, 1.367, 4.367, and 10.583 respectively for Melia azedarach. The ultimate analysis of rice bran for nitrogen, carbon, hydrogen, and sulfur is found to be 20.027%, 49.892 %, 6.058%, and 1.452 % with retention time (minutes) 0.867, 1.375, 4.375, and 10.600 respectively. The ultimate analysis of Water hyacinth for nitrogen, carbon, hydrogen, and sulphur is found to be 2.033%, 36.680 %, 5.251%, and 0.077 % with retention time (minutes) 0.867, 1.375, 4.375, and 10.700 respectively. The Proximate analysis of moisture content is found to be in the range of 5.8-8.70 %, Ash content 5.4-54.10 %, and volatile content 23.85-64.38 % for all three types of feed-stocks. This research paper also compares C, H, N, and S composition and the physio-chemical properties of extracted oil for the production of biodiesel.
Keywords: Biodiesel, Melia Azedarach, Water Hyacinth, Rice Bran, Proximate Analysis, Physio-Chemical Properties
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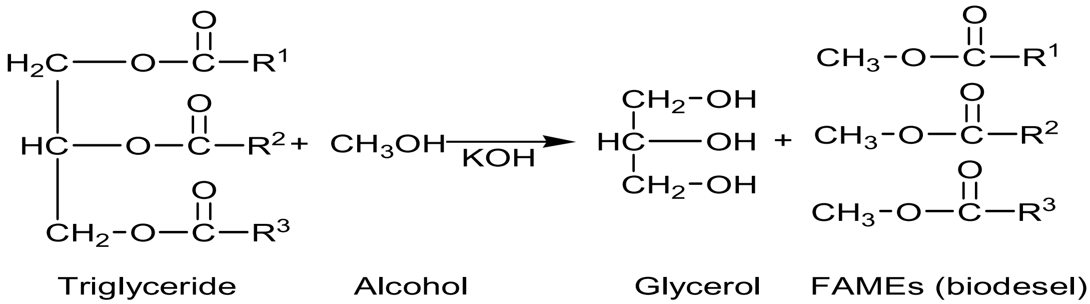
INTRODUCTION
Impeding conventional fossil fuels has a leading involvement because of the day-to-day usage of these fuels. Due to more demand for these conventional fuels, there is a decline in the reserve of fossil fuels, and it is also one of the key factors responsible for environmental pollution.[1-2] Due to more CO_2 emissions which contribute about 55% to global warming from greenhouse gases released from the emission of these fossil fuel-based automobiles. Several techniques including physical, chemical, and biological had been used for capturing CO_2 emissions.[3] In recent years there have been worldwide protests for achieving zero CO_2 emission which diverted the whole fuel industry towards the use of alternative fuel i.e., bio-diesel. Bio-diesel is in demand in the current era as the replacement for fossil fuels because of its novel environmentally friendly properties. It is a biodegradable, green fuel, with low Sulphur content and therefore non-polluting clean fuel. Produced pollution was recorded to be 0.0018 Wt. % which is found to be 29-fold less than gasoline.[4] Experimentally bio-diesel is produced by the trans-esterification of animal oils, vegetable oils and even cooking oils.[5] Biomass material blocks like rice bran can act as substitutes for fossil-based fuels and industrial processes. Bio-fuel are green, eco-friendly, and cleaner, they can be easily exploited to reduce greenhouse gas emissions.

The outermost layer of rice bran contains 10-12% of moisture content and proves to be excellent biomass for the production of biodiesel. It also contains ingredients that are alkaline in nature. Its calorific value is less than wood.[6] Rice bran is known to be a promising fuel that has many perspectives for bio-energy production and biomass-to-energy projects, diminishing famine and hence advancing the eminence of the lifespan of village people.[7] In the current scenario, the majority of the bio-diesel industry works by the use of edible vegetable oils which creates a big economic jolt for the country and also imposes competition among the food industry, which hikes the prices of these oil sources.[8-9] After recognition of this issue and its impact on the great demand for bio-diesel production, there is the invention of second-generation non-edible seeds and third-generation biofuel which can achieve the current target and act as a big energy source potential for the upcoming time.



Scheme-1 Trans-esterification

The non-edible feedstock includes mahua, jatropha (Ratanjot), S. chinesis (Jojoba), Ricinus communis (Castor), Melia azedarach (Eichhornia crassipes), haveabrazilenses (Rubber tree), have the biggest advantage that it can be easily cultivated in that area where other food crops cannot grow, and they are eco-friendly and can produce economically valuable products as a byproduct in contrast to the food crops.[10] Among all of these non-edible sources jatropha, a microalgal oil has high biomass content and acts to be a more reliable energy source. Seeds of Melia azedarach were found to contain various fatty acids like linoleic acid which is recorded to be 64.1% by weight, oleic acid with 21.8 Wt., palmitic acid having 10.1 Wt.%, Stearic acid having 3.5 Wt. %.[11] Melia azedarach L, remarkably best-known as the umbrella tree (Bakayan tree) or chinaberry, which comes under the category of Meliaceae, is a cosmetic tree.[12-14] It can grow vastly anywhere in open areas, roadsides, marshy lands, or near dwellings. It can easily grow in all types of soil especially alkaline and dry.[15] Even though it cultivates barren and plentiful in the cis-Himalayan zone and is also cultured for therapeutic and ornate objectives in India.[16] Like of neem bush, numerous fragments of China berry shrubs like greeneries, floras, and berries, and their bark have healing value as they are used for curative diverse skin ailments including ulcer injuries, herpes, Hansen's disease, dermatitis, scrofula disease, etc.[16] Even if Neem, is also an associate of the identical family, has been considered as a "feedstock" for making bio-diesel.[17] Phytoplankton are also very cheap sources of bio-diesel. The main reason for their popularity is first they are cosmopolitan and second because of their low price and consideration for third-generation bio-diesel e.g., water hyacinth (Eichhornia crassipes). Phytoplankton are very cheap raw material for bio-diesel production with high content of fatty acid." Water hyacinth" (Eichhornia Crassipes), is a marine vegetal and also known as Pontederia Crassipes. It is free- a floating perennial aquatic plant and known to be the wildest budding vegetal replicates using "stolon" which ultimately forms offspring floras. Due to the high biomass rate, 2.5 acres (1 hectare) of standing crop produces more than 1,000,000 cu ft/acre of biogas. It contains 70% of CH_4 and 30% CO_2.[12-14] It is used as an inexhaustible property and as a source of emission of neutral CO_2 by the use of efficient and cost-efficient techniques. Description and analysis of bio-diesel is critical. Data collection of chemicals, as well as physical properties, display information about the degree of quality, toxicity, and stableness of resultant products and the different procedures necessary for scaling to refined bio-oils and chemical substances that could be profitable and retrievable.[18] The bio-diesel exists at various biomass pyrolysis conditions and showed similar chemical compositions. Consequently, the current study engrossed the procedure of lipid extraction from Melia azedarach, rice bran, and water hyacinth.

EXPERIMENTAL

Material and Methods

Sample-1: Water hyacinth (Eichhornia Crassipes), reaped from stream Yamuna, Village-Kairana, District-Shamli (U.P), India.

Sample-2: Rice bran, a sample collected from the rice mill, Gandhi Mandi, G.T. Road, District-Panipat (Haryana), India.
Sample-3: Melia Azedarach, a sample collected from the college Campus of APIIT, S. D India, Toll Plaza, G.T. Road, District-Panipat (Haryana), India.

Processing of Feedstocks

This involves the washing, drying, and grinding of collected plant materials.

Washing

The collected feed stocks Melia Azedarach, Rice bran & Water Hyacinth (Eichhornia Crassipes) must be washed with water and mildly scrubbed to eliminate dirt and other remains. Afterward, the feedstock materials can be further washed with mineral water or tap water. The feedstocks were dissected into stalk, stem & roots, the roots are tending off. The color of the sample was pale yellow for Melia azedarach and dark green for rice bran oil with a smooth wax-like surface.

Drying

Feedstock of Melia azedarach, Rice bran, and Water hyacinth (Eichhornia Crassippes) is then dried in an oven (Precision, Model no.- 99202, hot- Air Oven) at temp. of 1050 ˚C for 24 hours.

Grinding

Feedstock is compounded into powder with the electrical mixer (model-S'5467 TH) and obtained feedstock is stored in a desiccator for further use. The dried sample was analyzed for "ultimate analysis" (C, H, N, and S) and "proximate study" (volatile matter, Ash content, Moisture content, and fixed carbon). The dried sample was analyzed for C, H, N, and S with the help of an elemental analyzer. (Model Flash 2000 series), company name- Thermo Scientific.

Extraction of Oil

The clean, dried, and grinded feedstocks are placed inside the Soxhlet apparatus which is packed with feedstock by employing solvent n-hexane for Melia azedarach and rice bran and methyl alcohol for water hyacinth and then refluxed for 10 hrs. at 45˚C temperature.[19] The obtained extracts were filtered and kept in a Rota evaporator which helps to evaporate the solvent left in the extract.[20] The physiochemical properties of bio-fuels like color, odor, pH, viscosity, flash point, iodine value, and acid value is determined as they play an important role in making bio-fuel function an automobile and are important in deciding their performance in various automobile engines under various environmental and other factors and also helpful in deciding parameters of its grading, storage, transport, and production.[21]

Colour and Smell

The color of extracted Melia azedarach and water hyacinth oil is observed as dark brown and with a strong pungent smell while the color of Rice bran is pale yellow with a buttery smell.

Viscosity (ASTM- d445)

Viscosity is defined as the internal friction offered by the layers of fluid to its flow. The extracted oil viscosity is measured with Redwood Viscometer (Model - SICBRVM-04). Three concordant readings were taken and averaged.

Flash Point (ASTM- d92)

It is characterized as the "lowest temperature" when an oil emits adequate vapors which burn for a moment and gives a flash, on bringing a burning candle carried close to it under specific conditions. Flashpoint determined with Pansky Marten Device (P18200). The oil is heated in such a way that its temperature rises by 3-5 ºC per minute. During heating the mechanical stirrer is rotated slowly for homogeneous heating of oil. When the temp. rises to about 15 ºC at the estimated flash point of oil, checking it started by inserting the burning flame into the oil mug through the opening for it in the lid. The checking is done for every rise in temp. of 3 ºC. The temp. at which a momentary flash produced by the heating of the oil is continued with frequent testing till the oil gives a flame that burns at least for 5 seconds. This temperature is recorded as a fire point. The process is repeated and the mean value of two temperatures is recorded separately for flash and fire points.[22]

pH (ASTM- d6423)
pH value can be defined as the measurement of hydrogen ion concentration (H^+) present in the oil under investigation. It can be determined with the help of a pH paper strip. The given oil is taken in a beaker and one pH paper strip is dipped inside it and leaves it for 1 minute to dry after one minute, we match the color that appears on the strip and note down its corresponding pH value by matching the color from the pH chart and noted its pH value from that chart.[23]

M/V(Density) (ASTM- d4052)
Liquid density is defined as the 'mass of liquid divided by its volume'. It can be measured with the help of a specific density of a 5ml bottle and then determined its mass with the help of an electronic compact scale (DHAUS, DA214) at a temperature of 25 ^{0}C. Three concordant readings were taken and then find out the mean value.[24]

Pour Point (ASTM- d97)
The pour point is determined with the pour point apparatus, the sample oil is placed inside a test tube which is surrounded by a cooling bath consisting of a mixture of ice and sodium chloride (ice + NaCl). The testing bar or tube is semi-filled with oil, and equipped with a thermometer that is passed through the cork inside the testing jar, when the cooling starts the temperature drops uninterruptedly, for every single 1^{0}C drop in temp. the test tube is taken out and temp. is examined. "Temperature, where haziness (Cloudiness) appears, is verified as its cloud point". Subsequently, this freezing is continued and the test tube is observed after every 3 ^{0}C reductions in temperature and slanted to see whether testing oil drift or not, that 'temperature' on which the oil ceases to flow in the test tube after keeping in a horizontal position is used for 5 minutes, is recorded as the "pour point."[25]

Acid No. or Neutralization No. (ASTM - d974)
Acid no. may be stated as "the number of milligrams of KOH required to neutralize the free acid present in 1 gram of oil". Weigh accurately 3-4 grams of oil sample in a weighing bottle and transferred it to the titration flask. Add 50 ml of ethyl methyl ketone and shake the mixture vigorously. Add 5-6 drops of phenolphthalein indicator and titrate it with standard alcoholic KOH slowly with constant shaking until there is an appearance of light pink color. Record the 'volume' of Potassium hydroxide (KOH) used in the experiment. Repeat this same process with a blank sample and record the volume of KOH used.[26]

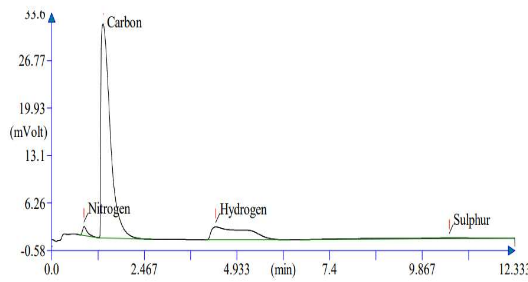
Iodine Value (ASTM- d4607)
The iodine Value may be stated as "the no. of grams of iodine equivalent to the amount of iodine mono-chloride (ICl) consumed by 100 g of the oil". Iodine value can be measured by preparing a known weight of oil sample in CCl_4, after then titrating using a known excess of Wij's solution i.e., Solution of iodine mono-chloride in 'Glacial acetic acid'. One molecule of ICl adds to each double bond present in the oil. The unreacted ICl oxidizes the KI solution to I_2. Iodine that liberates out is then titrated with hypo ($Na_2S_2O_3$) solution using freshly prepared 'starch indicator' which is added near the endpoint.[27]

Saponification Value (ASTM - d94)
Saponification no. may be stated as "the no. of a milligram of KOH required to saponify fatty material present in 1 gm of oil". The saponification value of an oil is determined via reflux of an observed mass of testing 'oil sample' with a standard alcoholic Potassium hydroxide (KOH) solution. 'Unreacted' KOH is determined using titration with standard hydrochloride (HCl) using phenolphthalein indicator.[28]

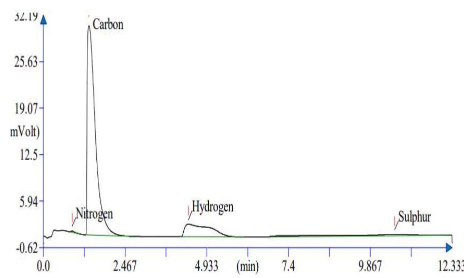
RESULTS AND DISCUSSION

Elemental Study (C, H, N, S)
The chemical constituents of C, H, N, and S for three feedstocks are given in Table-1. The total elemental percentage composition for Melia azedarach is 55.146 with retention time (minutes) for nitrogen, carbon, hydrogen, and sulphur 0.858, 1.367, 4.367, and 10.583 minutes respectively. For rice bran, the total elemental percentage is recorded as t57.429 % with the retention time (minutes) for nitrogen, carbon, hydrogen, and sulphur 0.867, 1.375, 4.375, and 10.600 minutes respectively. The total elemental percentage for water hyacinth is found to be 44.041% with the retention time (minutes) for nitrogen, carbon, hydrogen,

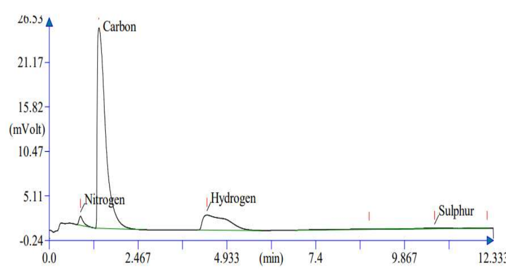
and sulphur 0.867, 1.375, 4.375, and 10.700 minutes respectively. The Sulphur and Nitrogen contents in Melia azedarach, Rice bran, and Water hyacinth are comparatively low and do not diffuse into biofuel which is significant because it is extremely objectionable in fuel handling, chemical process, and usage.[29] It clearly emphasizes from the spectra graphs 1, 2 & 3, that the biofuel produced from these feedstocks could be Eco- friendly and proves to be green fuel as compared to fossil-based fuels.[30]



Graph-1: Ultimate Analysis of Melia Azedarach



Graph-2: Ultimate Analysis of Melia Azedarach



Graph-3: Ultimate Analysis of Melia Azedarach

Table-1: Comparison of Ultimate Analysis of Three Feed Stocks

Element (%)	Rice Bran (%)	Melia Azedarach (%)	Water Hyacinth (%)
C	49.891	45.531	36.680
H	6.058	6.574	5.251
N	0.027	2.309	2.033
S	1.452	0.732	0.077
Total Element	57.429	55.146	44.041

Proximate Analysis

The moisture content of Melia azedarach, Rice bran, and Water hyacinth was observed to be 5.8 ± 0.0, 8.70, and 6.35, which is not in excess. The excess of moistness drops the calorific capacity of the sample and takes away a significant quantity of heat in the form of latent heat energy. Extreme surface moistness cause exertion in handling the sample and causes quenches fire in the furnace. Volatile matter for the three feedstocks Rice bran, Melia azedarach, and Water hyacinth was observed to be 2.00, 23.85, and 64.38 respectively which is reported to be efficient. The high volatile content specifies that a big percentage of fuel is oxidized in gaseous form. The essential requirement for biofuel manufacturing is the presence of 'high volatile content' as in sample carbon signifies the quantity of sample that is available to convert into gas as a fuel & mineral- tar products. As far as Ash content is concerned it is 5-4 ± 0.5 for Melia azedarach, 54.10 for Rice bran, and 2.033 for Water hyacinth. Excessive fraction of ash is objectionable as it lowers the heat of combustion of fuel. High ash leads to higher heat losses and causes ash lumps. Fixed carbon is that carbon that burns in a solid state.[31] Fixed carbon for three feedstocks Rice bran, Melia azedarach, and Water hyacinth are 86.80, 13.35, and 0.077 respectively. Heat of Combustion of fuel depends on the proportion of 'Fixed Carbon', a high percentage of fixed carbon favors high heat of combustion or calorific value.

Table-2: Proximate Analysis of Samples

Analysis parameters (%)	Rice Bran (%)	Melia Azedarach (%)	Water Hyacinth (%)
Moisture content	8.70	44.1 ± 1.14	6.35
Volatile matter	23.85	2.00 ± 0.5	64.38
Ash	54.10	5-4 ± 0.5	2.033
Fixed carbon	13.35	48.5	0.077

Physio-Chemical Analysis of Feed-Stocks

Tables-3, 4, and 5 display the index parameter for biofuel. From the observed results, most of the indexed parameters follow the quantity dimension of biofuel for Melia azedarach, Rice Bran oil, and Water hyacinth. It also follows the quality guidelines of biodiesel within the range limits of American (ASTM) and European fuel standard requirements. According to European Standard (EN 14214 standard), the average specific gravity of biodiesel is reported as 0,8600-0,900 gm/ml. The mean value index of Melia azedarach is found below as compared to the prescribed guidelines of the European standard. This lowering may be due to environmental factors. The reported specific value indices of rice bran oil and water hyacinth is found to be high as compared to the guidelines of European standard. Some environmental factors also matter as in the case of water hyacinth the energy content of the pond is high so the specific gravity of the pond water is also found to be high.[32] The main reason for this difference might be due to fluctuation in the ecological niche. The mean acid value reported for Melia azedarach is 0.45 mg KOH/gm and for Rice bran oil acid value reported to be 0.5 mg KOH/gm and for water hyacinth it is observed to be 0.4 mg KOH/gm. According to the guidelines of ASTM, the maximum acid value is 0.8 mg KOH/ gm, and accordant with the guidelines of EN 14214 (European standard) it is 0.5 mg KOH/gm for bio-diesel production and therefore reported to precede the guidelines of international level- advisable for manufacturing bio-diesel. Melia azedarach and water hyacinth reported densities are 1004.2 kg/m^3 and 1004.3 kg/m^3 having brownish black color and rice bran oil density is recorded to be 0.913 kg/m^3 having pale yellow color, the reason for high density could be due to the presence of impurities and may be due to high moisture value in the oil, these observed densities are recorded high as compared to light fuel-based oil which is estimated to be 850 kg/m^3.[33] High density of this bio-oil displayed the presence of low aromatic content and high oxygen content like hydro-carbon.[33] Due to the high hygroscopic nature of Eichhornia Crassippes (water hyacinth) that is found responsible for heavy moisture amount in their biomass and the Flash point of Eichhornia crassippes observed as 220 ºC and when we differentiate with other types of oil, flash point is little high, if compare with the diesel oil (Flash Point-53 ºC) and with bio-diesel (flash point-160 ºC) Oil extracted out from Eichhornia Crassipes (Water hyacinth) was tested for profiling various fuel properties and after testing there is the minimal concentration of S (Sulphur) and N (Nitrogen) which indicates the suitability of fuel as green oil or Eco- friendly.[34] The iodine value of Melia azedarach, rice bran oil, and water hyacinth was observed as 9.14 g, 105 g, and 20.90 g and it indicates the oxidation stability and the degree of unsaturation exist in the oil sample and reported to convey all the specific guidelines cited in the EN 14214 and in the range limits of EN 14214 T, this points out the presence of 'free acid' content in the obtained bio-fuel. The viscosity value observed for Melia azedarach, rice bran oil, and eichhornia crassipies is 0.0398 poise as its key factor in fuel characterization and its bio-diesel significances. High viscosity favors incomplete combustion, adducted fatty acid chain length and thereby adducted to saturated bonds[34], and it also held for high atomization, excessive carbon deposition in various parts of machine like 'injection nozzles' which is found to be responsible for contaminated lubricating oil with unburnt carbon particles. Thus, in the refined state Melia azedarach, Rice bran oil, and Eichhornia Crassipes (water hyacinth) observed to be inapplicable in modern-day 'diesel engines', can overcome this problem by lowering the viscosity of bio-oil by compounding with either diesel oil or by blending with other types of fuels. The pH value reported to be 6.9 for Melia azedarach and for Rice bran oil it is 2.93 and it is recorded in the parameter range of 1-4.5 for pH chart of extracted oil cited in the literature review. The lowering of pH value is due to the presence of aldehydes which creates acidity, found to be the major reason of "corrosion" in the machinery parts of the engine. To overcome this problem extracted bio-oil require some modification in their chemical composition which can lower down the acidity.[36-37]

Table-3: Physio-Chemical Properties of Melia Azedarach

Properties	Value
Color	Dark Brown
	Pungent like
Saponification No. (mg KOH / g oil)	171.8 + 0.2
Acid No. (mg KOH / g oil)	0.45
Iodine Value	127.2 + 0.3
Viscosity	0.0398 Poise
pH	4.3
Density	0.9
Specific gravity (g/ml)	0.95

Table-4: Physio-Chemical Properties of Rice Bran

Properties	Value
Colour	Pale Yellow
	Nutty flavor like
Saponification No. (mg KOH / g oil)	190
Acid No. (mg KOH / g oil)	0.5
Iodine Value	105 g
Viscosity	0.0398 Poise
pH	3.9
Density	0.913
Flash point	280 ˚C
Specific gravity(g/ml)	0.92 g/ml

Table-5: Physio-Chemical Properties of Water Hyacinth

Properties	Value
Colour	Dark Brown Black
	Acid Smoky Smell
Saponification Value (mg KOH / g oil)	5.42 mg
Acid Value (mg KOH / g oil)	0.4
Iodine Value	20.90 g
Viscosity	19.8 Poise
pH	2.93
Density	1004.3 Kg/m^3
Flash point	220 ˚C
Specific Gravity (g/ml)	0.9129 g/ml

CONCLUSION

From this study, it can be concluded that the prime dimension of bio-diesel of three feed-stocks (Melia azedarach, Rice bran, and Water hyacinth) accomplishes the standardized regulations by American (ASTM) and European standards. The contrary document displays that the oil content of Melia azedarach, Rice bran, and Water hyacinth is affected by the harvest cycle, Agroecology, and period. Bio-oil from the extraction of three feed-stocks was defined for fuel attributes which represent that valuable Petro-chemicals,

chemicals, and medicinal products can be attained from 'hyacinth oil'. Pyrolytic "Water- hyacinth bio-oil" in actual form might not be the right substitute fuel, yet, it proves to be a contender for purification in manufacturing contemporary oils and substances or chemicals. Since its fuel attributes and their biochemistry configuration. Fat content in Melia azedarach fruits makes it an appropriate raw material for biodiesel production as documented by the characteristics of bio-diesel formed from it. Due to the presence of a high degree of unsaturated hydrocarbons which are more prone to oxidation, this limitation can be removed by the accumulation of additives like pigment to the bio-diesel. In all three feed-stocks, bio-fuel has an insignificant concentration of N_2 and S, which symbolizes that it is an 'eco-friendly oil'.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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