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Review

Alternative Methods for Biodiesel Cetane Number Valuation: A Technical Note

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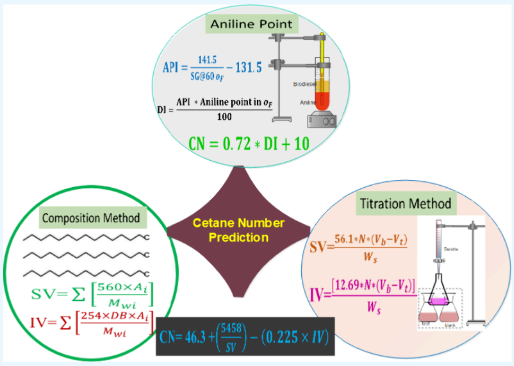
ABSTRACT: Biodiesel is an environmentally beneficial and clean energy source that may replace fossil fuels, which are detrimental to the environment and cannot be replenished. Therefore, the physicochemical parameters of biodiesel must be determined in order to verify its quality. The cetane number is a crucial dimensionless fuel property that gauges the fuel ignition quality in power diesel engines. A higher cetane number results in a shorter ignition delay time, and vice versa. Biodiesel's cetane number may fluctuate due to a variety of fatty acid compositions, including variations in carbon chain length and the degree of unsaturation. The cetane number generally increases with increasing saturation and chain length, while it decreases as chain length is reduced and degrees of unsaturation and branching increase. This is the main reason for why alkanes possess a higher cetane number than alkenes and aromatics. The standard protocols for evaluating the cetane number of biodiesel are ASTM D613 and ISO 5165 test techniques using a monocylindrical cetane engine. However, adhering to these conventional procedures is quite challenging and time-consuming, and the cetane number test result may also be affected by the presence of certain gases and fumes. As a result, many researchers are bothered with cetane number valuation, and occasionally they skip it due to a lack of other options. Consequently, the aim of this paper is to present a set of more straightforward and relevant alternative techniques that can be applied to predict the cetane number of biodiesel when engine-based measurement is not practical. The three techniques with their designed pictographic outlooks conferred in this article include color indicator titration, aniline point, and fatty acid composition-based methods. The reported values of these procedures meet the minimum cut point of the biodiesel cetane number required by ASTM D6751 (≥47) and exhibit minimal variation from the typical standard methods. Nevertheless, the above-mentioned techniques are not applicable to other alternative biofuels except biodiesel products because they have a direct implication on the characteristics of the fatty acid profiles of different oil precursors, such as carbon chain length, degree of saturation or unsaturation, and aromaticity, which make up monoalkyl esters.



1. INTRODUCTION

The environment is worsening and worldwide energy scarcity is increasing rapidly due to rapid industrial and metropolitan growth.[1−6] The high energy demand is largely satisfied with the use of nonrenewable resources originating from coal, petroleum, and natural gas, which depletes and exhausted gradually.[7,8] Apart from gradual depletion, the use of nonrenewable resources has led to atmospheric pollution and global warming.[9−13] As a result, researchers worldwide have become motivated in terms of developing clean renewable alternative energy sources that could be readily available, environmentally sustainable, technologically feasible, and techno-economically competitive for socioeconomic prosperity.[3,14−16] Previously, wide-ranging research has been performed to explore alternative energies for fossil resources,[17] mainly focused on wind, hydropower, and solar.[18] But, according to the 21st Century Renewable Energy Policy Network (REN 21), the production of biodiesel has been superior to that of other biofuels and could be an alternative fuel.[5,17,19−21]

Biodiesel is a liquid biofuel commonly made by the transesterification of triglycerides molecules (hydrophobic substances made from one mole of glycerol with three moles of fatty acids) with alcohol in the presence of a suitable catalyst. The end product contains fatty acids of alkyl esters.[22−27] The common sources of biodiesel are various organic raw materials such as edible and nonedible vegetable oils obtained from soybean, canola, rapeseed, Jatropha, mustard, palm, beauty leaf, microalgae, mahua, rubber seed, animal fats, waste cooking oil, etc.[12,16,28] However, the use of edible oils as biodiesel feedstock may contribute to price fluctuation and lack of supply due to the

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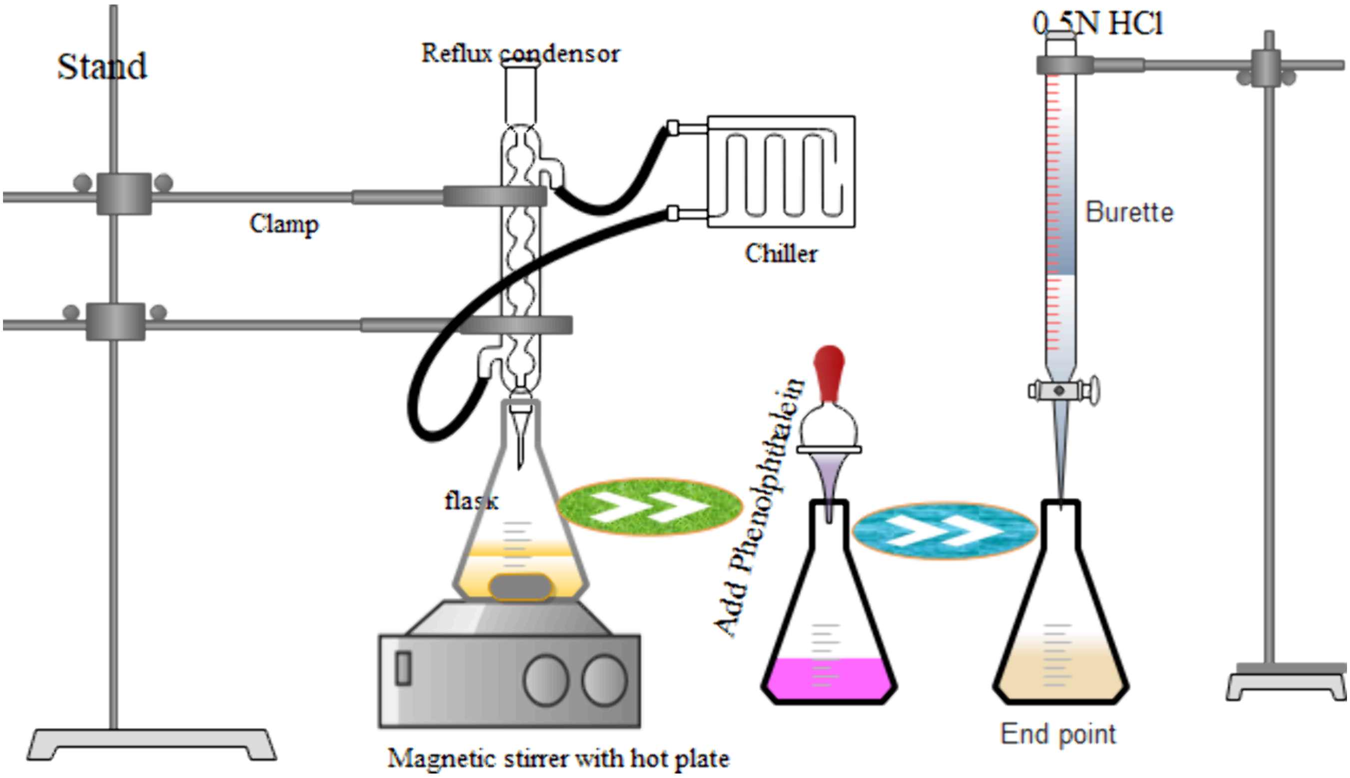

Figure 1. Titration method for saponification value measurement.

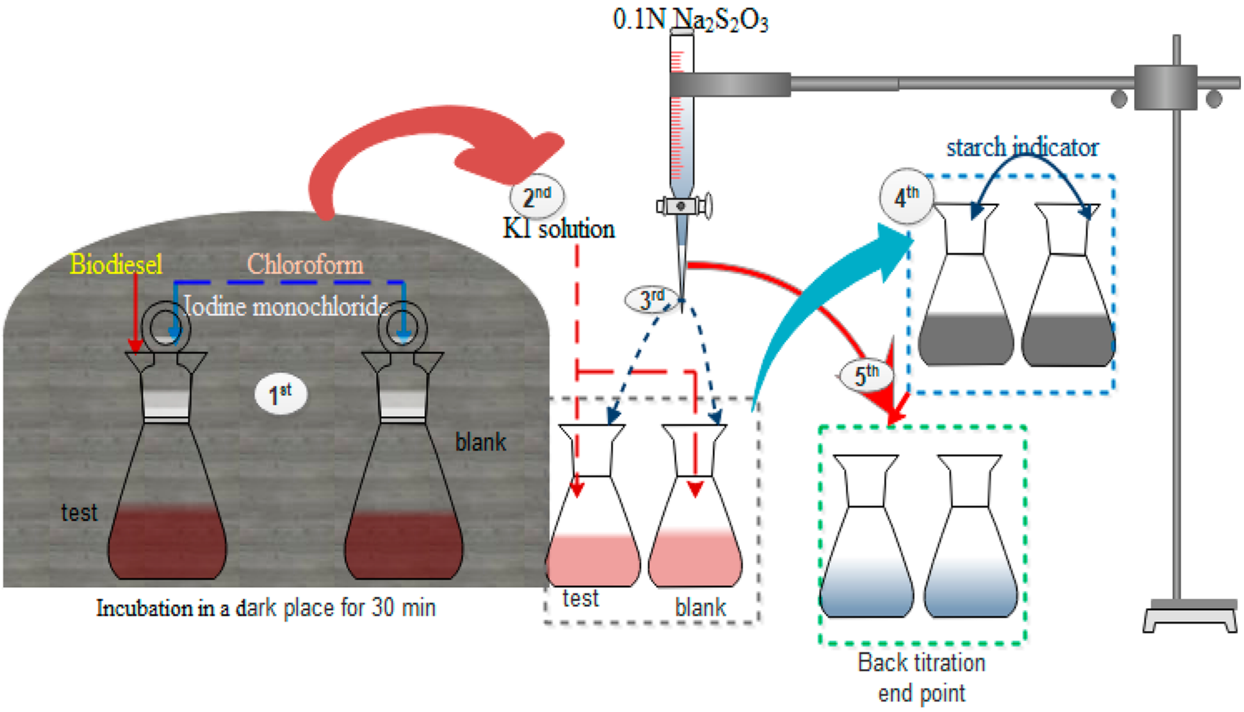

Figure 2. Titration method for iodine value measurement.

competition of human food intake. Therefore, nonedible oils could be promising for biodiesel production.[29,30] The main advantages of using biodiesels over fossil fuels include biodiesel being considered a green technology because of its low toxicity and high biodegradability, renewability (as it is made from vegetable oils and animal oils), and relative safety (its higher flash point), less CO emission, versatility (could be used in diesel engine without hardware modification), high cetane number, etc.[28,31−35] In addition to several advantages, the use of biodiesel has some drawbacks, such as lower spray speed plus inferior fuel atomization due to higher viscosity, lower calorific value, high corrosion in the copper strip, higher NOx emissions, and largely cold start problems in cold climate environment. Some of the proved systems used to reduce such demerits of

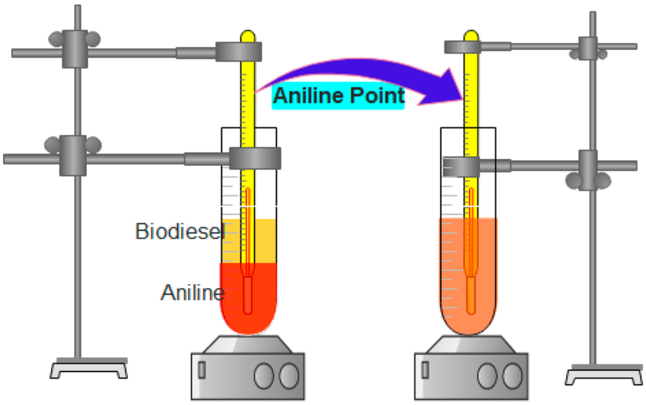

Figure 3. Aniline point measurement

biodiesel are blending it with petrol diesel at any ratio because of similar properties, and engine gas recirculation (EGR).[1,23,28,36−38]

Because of the extensive production and variety of feedstock used in transesterification, quality control of biodiesel with effective approaches is needed to determine its physicochemical properties.[27] One of the most important dimensionless quality parameters is the cetane number, which describes the auto ignition characteristics guaranteed for the selection of biodiesel.[39,40] The cetane number of biodiesel has been widely described in many literature reports. The standard test methods acceptable to assess cetane number of biodiesel are ASTM D613 and ISO 5165 procedures using a cetane engine,[41,42] where the delay of a mixture of cetane and alpha-methylnaphthalene with known cetane number is compared to the fuel ignition delay. But, theses standard experimental determinations need a large quantity of fuel samples for measurement (about 500 mL) and are quite difficult and time-consuming, and hence, numerous predictive models based on some fuel properties have been established.[43−49] Furthermore, certain fumes and gases present in the space where the test engine is situated might have a measurable impact on the result of the cetane number test.[41] Since the cetane number is one of the most typical properties of biodiesel ignition delay after adding it to the combustion chamber, many researchers are troubled by its value determination when the cetane engine is not accessible and smaller measuring samples are presented. The purpose of this paper is to provide a collection of some simple and significant alternative techniques convenient for evaluating the cetane number of biodiesel to simplify the aforementioned challenges associated with engine-based standard measurements.

1.1. Fundamentals of Cetane Number (CN).

Cetane number is one of the most important fuel properties tha measure the autoignition characteristics of the fuel in a power diesel engine, mostly critical during cold starting engine conditions.[38,49−51] This dimensionless number is highly responsible for ignition delay (the time interval between the start of fuel injection and the start of combustion), and it depends on the composition of the fuel. The higher the cetane number, the better the ignition quality and the shorter the ignition delay time. Thus, a fuel having a higher cetane number shows its higher combustibility, shorter ignition delay, and noiseless plus smoother fuel combustion, which determines the power as well as the economic performance of the engines. Conversely, lower cetane number leads to hard starting of the engine in cold environments, increased knocking, deposit formation caused by incomplete combustion, generation of pollutants from engine exhaust (hydrocarbons emissions) and affects the ignition delay.[28,38,43,52−54] In this approach, cetane number affects not only the exact rate of heat release but also responsible for pollutant emission as well as radiation of combustion noise.[43] As the fatty acids chain length and degree of saturation increased, the cetane number of biodiesel is also linearly improved. In this respect, alkanes possess higher cetane number than alkenes. Generally, biodiesel has greater cetane number relative to the pure conventional diesel fuels, due to the presence of saturated molecules, longer fatty acid carbon chains, and more oxygen contents associated with the carbonyl groups.[28,43,45] The amount of oxygen that compos biodiesel ranges in 10−11% which can be used to increase the combustion efficiency of engines and decrease the fuel's oxidation potential.[17] The minimum values of the cetane number for biodiesel dictated by ASTM D6751 and EN 14214 standards are 47 and 51, respectively.[43]

1.2. Alternative Techniques.

The proposed cetane number prediction methods based on the different physicochemical characteristics of biodiesel addressed in this paper include titration, aniline point, and fatty acid composition-based techniques. Their detailed information is explained below.

1.2.1. Titration Method from Saponification and Iodine Value.

Saponification Value (ASTM D1962). The saponification value of fuel represents the milligrams of KOH needed to saponify 1 g of sample.[28] It measures the chain length and average molecular weight of fatty acids. A lower saponification value corresponds with long chain fatty acids due to fewer carboxylic functional groups being present in the unit mass of fat.[54] One of the standard procedures for determining the saponification value of biodiesel is via the ASTM D1962 titration method. In this method, 1 g of sample is dissolved in 10 mL of ethanol followed by further addition of 25 mL of 0.5 N ethanolic KOH to the sample-solvent mixture and refluxing for 30 min, as shown in Figure 1 and which is termed as a test sample. Afterward, the sample is allowed to cool until it reaches room temperature. Finally, 2−3 drops of phenolphthalein indicator is added in to the cooled solution, and the excess KOH is titrated with 0.5 N HCl solution until the end point (disappeared of pink color) using a burette dropper as shown in Figure 1. The procedure for the blank titration (without sample) is similar to the sample test titration. Then, the saponification value is determined by eq 1.[8,55]

$$\text{Saponification value, mg of KOH/g} = \frac{56.1N(V_b - V_t)}{W_s} \tag{1}$$

Where N is the normality of KOH, mol/mL; V_t is the volume of HCl consumed for the test sample titration, mL; V_b is the volume of HCl consumed for blank sample titration, mL; and W_S is the weight of biodiesel sample, g.

Iodine Value (EN 14111). Iodine value is defined as grams of iodine absorbed by 100 g of sample. It measures the degree of unsaturation in the biodiesel. High levels of unsaturation results polymerization of glycerides because of epoxide formation as a result of addition of oxygen in double bonds. This can lead to the formation of deposits and thus decline the lubricating properties of the fuel.[8,54,56] However, the iodine value cannot be trusted for evaluating the unsaturation level since a composition with lots of fatty acids gives an equal iodine value. Thus, it is just a measure of the number of C=C bonds present and does not account for the position of the double bonds.[57] In general, biodiesel with a

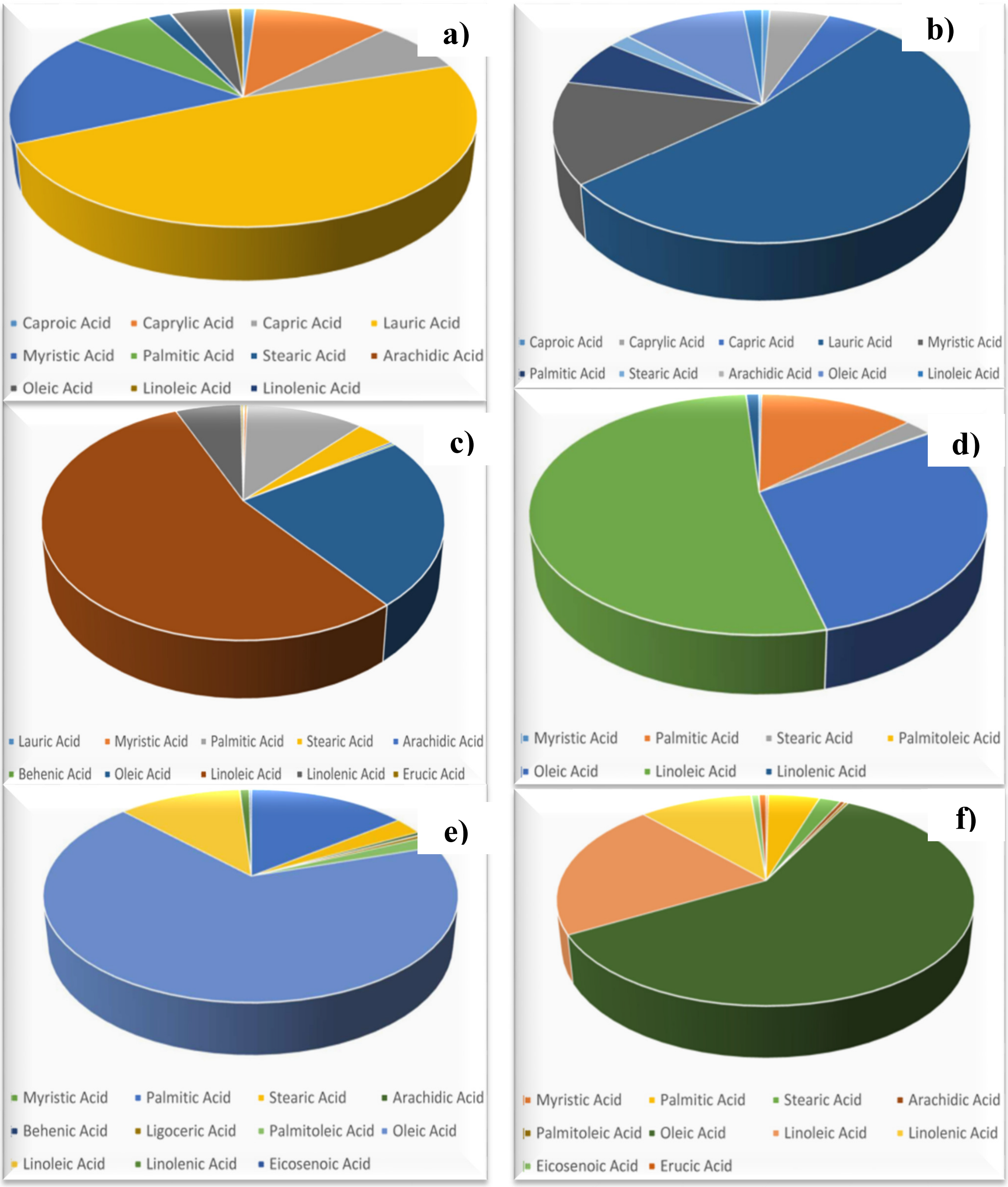

Figure 4. Contribution of fatty acid percentage to the saponification number of biodiesel derived from (a) coconut oil, (b) palm kernel oil, (c) soyabean oil, (d) corn oil, (e) olive oil, and (f) canola oil.

low iodine value is more efficient and combustible than fuel with a higher value, but it could possess poor cold-flow characteristics.[54] For biodiesel, the ASTM standard does not specify the iodine value;[28] however, European standards EN 14213 and EN 14214 state iodine values of 130 and 120, respectively. The standard procedure for iodine value determination is EN 14111. In this technique, 10 mL of chloroform solvent is poured into two separate flasks as presented in Figure 2. In one flask, 1 g of biodiesel sample is added to be dissolved as a test, and the second flask containing only solvent is used as a blank. In both flasks, 20 mL of iodine monochloride reagent was added and thoroughly mixed followed by setting in a dark place for incubation for about 30 min. Subsequently, 10 mL of KI solution was added into each test sample by taking care of complete mixing by rinsing the beaker sides with 50 mL of distilled water. Both the test and blank samples were then titrated with 0.1 N sodium thiosulfate ($Na_2S_2O_3$) aqueous solution until the color changed to pale straw. Next, 1 mL of starch indicator was added into both flasks and alteration of the color to purple could be observed. The solution was again back-titrated until the purple color changed to colorless. Finally, the iodine value can was determined by eq 2.[8,55,58,59]

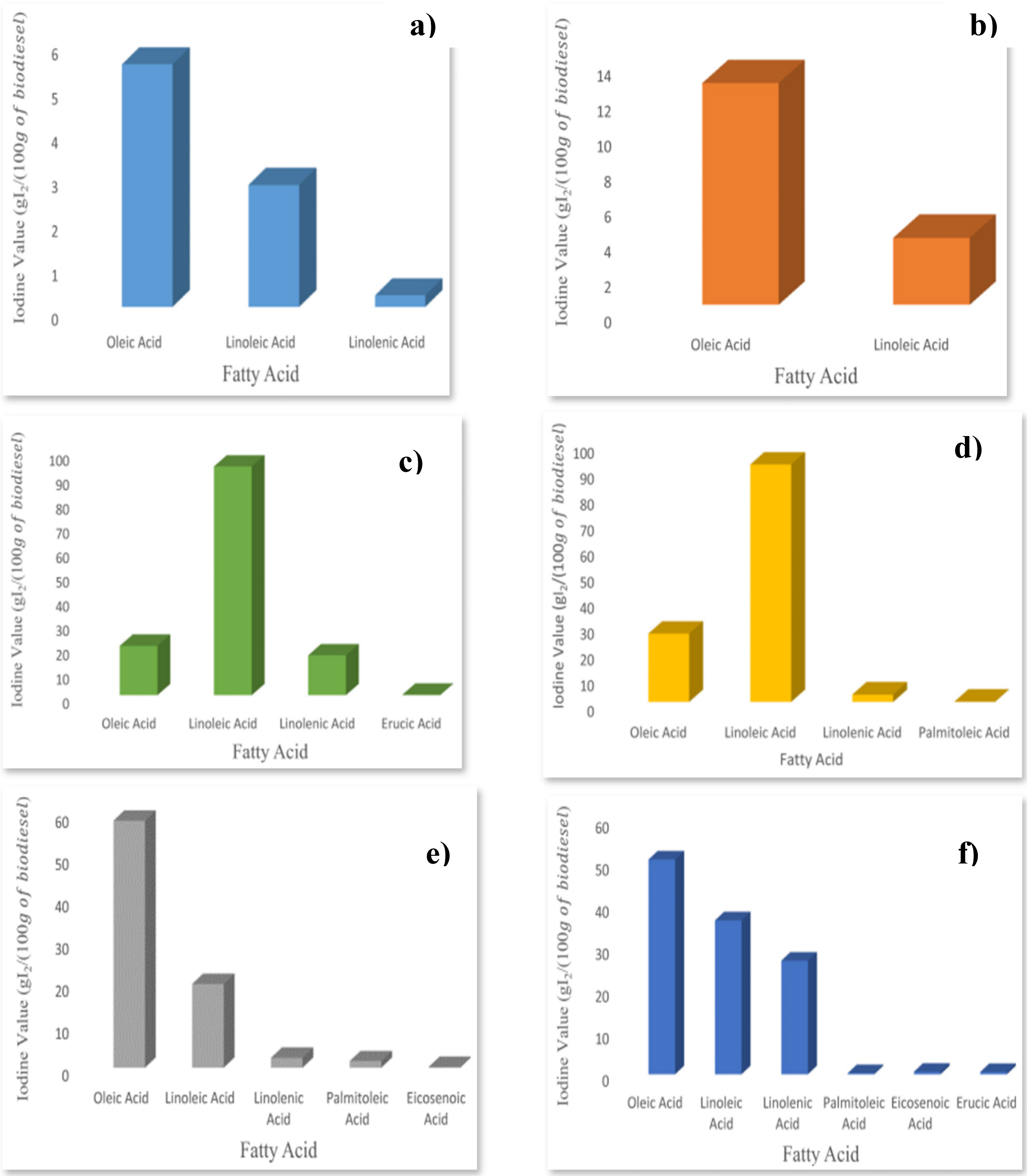

Figure 5. Graphical depiction of contribution of fatty acids to the iodine value of biodiesel derived from (a) coconut oil, (b) palm kernel oil , (c) soyabean oil, (d) corn oil, (e) olive oil, and (f) canola oil.

$$\text{Iodine value, g of iodine/100 g} = \frac{[12.69N(V_b - V_t)]}{W_s} \tag{2}$$

Where N is the normality of $Na_2S_2O_3$, mol/mL; V_b is the volume of $Na_2S_2O_3$ for the blank sample, mL; V_t is the volume of $Na_2S_2O_3$ for the test sample, mL; and W_S is the weight of sample, g.

Based on the saponification and iodine values, the cetane number of biodiesel was then predicted with eq 3.[8,53,60,61] For example, biodiesel derived from waste cooking oil,[8] castor bean,[59] Croton macrostachyus (Bisana) kernel oil,[62] and Aegle Marmelos Correa[55] shows cetane numbers of 57.11, 51.48, 50.78, and 58, respectively, which are acceptable and meet the ASTM D6751 specification.[8,55]

$$\text{CN} = 46.3 + \frac{5458}{\text{SV}} - 0.225\text{IV} \tag{3}$$

Table 1. Comparison of Biodiesel Cetane Number (CN) Determined with ASTM D613 and Analytical Methods[a,54]

Biodiesel	Measured CN by ASTM D613	Calculated CN with eq 9	Absolute error	Percent error (%)	ASTM D6751 limit
Coconut	66.30 ± 1.04	65.85 ± 0.99	0.45	0.6787	≥47
Palm kernel	62.50 ± 0.94	65.10 ± 0.98	2.60	4.1600	
Soyabean	47.00 ± 0.71	45.51 ± 0.68	1.49	3.1702	
Corn	48.20 ± 0.72	47.58 ± 0.71	0.62	1.2863	
Olive	58.60 ± 0.88	56.44 ± 0.85	2.16	3.6860	
Canola	48.50 ± 0.73	49.20 ± 0.74	0.7000	1.4433	

[a] Absolute error = |Measured − Calculated|; Percent error = $\left|\frac{\text{Measured} - \text{Calculated}}{\text{Measured}}\right| 100\%$

Where CN is the cetane number; IV is the iodine value; and SV is the saponification value.

1.2.2. Using Aniline Point (ASTM D4737). Aniline ($C_6H_5NH_2$) is an aromatic amine that can be used as a solvent selective to naphthenes and paraffins at higher temperatures, and aromatic molecules at low temperatures. Aniline is commonly used to evaluate the aromaticity of oil products via evaluating the aniline point. The aniline point is the lowest temperature at which equal volumes of oil based sample and aniline become completely miscible. When the aromaticity of the sample increases, the aniline point decreases because of the complete mixing at lower temperature. But the rise of the paraffinicity and the molecular weight of the oil product increase the aniline point. Olefins and naphthenes show values between aromatics and paraffins.[63,64] In conditions where the cetane number test cannot be done by the ASTM D613 standard method in a monocylindrical engine, it is possible to follow the ASTM D4737 procedure using the cetane index and the aniline point through eqs 4−6. In this method, equal volumes of aniline and biodiesel sample (about 10 mL of each) are initially added in to a U-shaped glass test tube in which their immiscibility is clearly seen. A temperature reading thermometer is placed in to the test tube as revealed in Figure 3. Then, the mixture is gradually heated until it becomes a completely mixed, homogeneous solution. The temperature reading where the homogenate mixture observed is noted as the aniline point and converted to Fahrenheit. The aniline point of a fuel and its ignitability are correlated by an intermediate empirical expression of diesel index (DI), which is calculated based on the density of fuel, and finally the cetane number can be determined from it.[25,38,63−65]

$$\text{API Gravity} = \frac{141.5}{\text{SG@60 °F}} - 131.5 \tag{4}$$

$$\text{DI} = \frac{\text{API gravity}\cdot\text{Aniline point in farenheit}}{100} \tag{5}$$

$$\text{cetane number, CN} = 0.72\text{DI} + 10 \tag{6}$$

Where SG is specific gravity of biodiesel at 60 °F (15.5 °C).

1.2.3. Using Fatty Acid Composition. The chemical composition of a feedstock determines the properties of the biodiesel being produced. Hence, the degree of unsaturation and fatty acid chain length are the important parameters in determining the physiochemical properties. The presence of saturation and long-chain fatty acids can increase the cetane number of biodiesel. Saturated fatty acids are fatty acids that do not contain a double bond in their structure, whereas unsaturated ones contain a double bond. The level of unsaturation is directly linked with the iodine value, and the molecular weight of fatty acids is connected with the saponification value.[53,66,67] Thus, the fatty acid structure and the level of unsaturation influence the ignition delay, which upsets the performance and causes an increase in exhaust emissions. Therefore, the cetane number of biodiesel can be predicted from its fatty acid composition using the empirical correlation with the saponification and iodine values, eqs 7−9.[40,53,54,68−75] The benefit of these equations is that the relative influence of each fatty acid component on the total iodine and saponification numbers can be simply evaluated.[52] The iodine value decreased with chain length while growing linearly with an increasing degree of unsaturation. As both the carbon length and molecular weight increased, the saponification number decreased because of their inverse relationship. Similarly, the saponification number decreases as the degree of unsaturation increases. Moreover, a comparative study of the cetane number determination of biodiesel derived from six different feedstocks, i.e., coconut, palm kernel, soybean, corn, olive, and canola, using the fatty acid composition and ASTM D613 standard techniques has been reported in the literature. The individual fatty acid profiles and contributions of all derived biodiesel products to the saponification and iodine numbers are depicted in Figures 4a−f and 5a−f, respectively. Based on these values, the calculated cetane number and the measured ones using the standard techniques, as well as the errors committed between the two methods, are clearly shown in Table 1.[54] The findings indicated that there is no noticeable difference between the calculated and measured cetane numbers, and hence, the suggested fatty acid composition method can reasonably predict the cetane number of biodiesel products.

$$\text{SV} = \sum \left[\frac{560A_i}{M_{wi}}\right] \tag{7}$$

$$\text{IV} = \sum \left[\frac{254DBA_i}{M_{wi}}\right] \tag{8}$$

$$\text{CN} = 46.3 + \left(\frac{5458}{\text{SV}}\right) - (0.225\text{IV}) \tag{9}$$

Where A_i is the percentage composition of each fatty acid in the oil or its ester; DB is the number of double bonds present in each unsaturated fatty acid or its ester; and M_{wi} is the molecular weight of each fatty acid or its ester component.

■ CONCLUSION

The article offers insightful details on the multiple approaches to biodiesel cetane number (ignition delay) valuation with their respective pictographic outlooks rather than adopting the extremely challenging and lengthy standard processes of ASTM D613 and ISO 5165. The aniline point, the color indication titration method based on saponification and iodine values, and the fatty acid composition-based approach with a committed average percentage error of 2.75% from the conventional monocylindrical cetane engine systems are some of the alternative schemes covered in this paper that exhibit minimal deviation. Moreover, the reported outcomes of these techniques meet the minimum cut point of the biodiesel cetane number specified in the ASTM D6751 requirement (≥47). However, the aforementioned alternative methods are not applicable for other biofuels, except biodiesel products, because they have a direct correlation with the fatty acid profiles of various oil precursors, like carbon chain length, degree of saturation or unsaturation, and aromaticity, which constitute monoalkyl esters. In general, the important alternative techniques to be applied when the cetane test engine is not possible are compiled and shared with researchers who are having trouble computing the biodiesel cetane number.

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Notes

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