Development of a test method for distillation of diesel-biodiesel-alcohols mixtures at reduced pressure

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Abstract. Increased environmental awareness and depletion of fossil petroleum resources are driving the automotive industry to seek out and use alternative fuels. For instance, the biofuel is a major renewable energy source to supplement declining fossil fuel resources. The addition of alcohols like methanol and ethanol is practical in biodiesel blends due to its miscibility with the pure biodiesel. Alcohols also improve physico-chemical properties of biodiesel blends, which lead to improved combustion efficiency. Proper volatility of fuels is critical to the operation of internal combustion engines with respect to both performance and emissions. Volatility may be characterised by various measurements, the most common of which are vapour pressure, distillation and the vapour/liquid ratio. The presence of ethanol or other oxygenates may affect these properties and, as a result, performance and emissions, as well. However, in the case of diesel-biodiesel-alcohols mixtures, the variance of component volatility makes difficult the analysis of the overall volatility.

Thus, the paper presents an experimental method of distilling diesel-biodiesel-alcohols mixtures by adjusting the boiler pressure of an i-Fischer Dist equipment.

1. Introduction

From the road transport standpoint, today, there are three major problems facing humanity: (1) concerns about greenhouse gas (GHG) emissions and global climate change; (2) a desire for renewable/sustainable energy sources; and (3) an interest in developing domestic and more secure fuel supplies, in other words, energy security. These issues have led to intense debate within international organizations and among political leaders on the impacts of the increased use of biofuel. [1]

The EU objective is to achieve an overall 80%–95% reduction in CO2 emissions by the year 2050, with respect to the 1990 level. Decarbonization of transport and the substitution of oil as transport fuel therefore have both the same time horizon of 2050. Thus, the European “Renewable Energy Directive” 2009/28/EC includes a binding target of 10 percent renewable fuels in transport in 2020 [2- 6].

S. K. Thangavelu et al. in [7] support the idea the most eminent alternative fuels for replacing fossil fuels in internal combustion engines are biofuels (biodiesel and bioethanol).

Also, Mohd Hafizil Mat Yasin et al. concluded in their study [8] that a lot of different researchers around the world revealed that biodiesel is proven as a substitute for mineral diesel and the use of additives such as alcohol is a solution to achieve objectives like reducing the emission significantly.
Ethanol and methanol has higher oxygen in basis (approximately 35% and 30%) as compared to mineral fuels that help internal combustion (IC) engine to achieve higher complete combustion. More oxygen in fuels means more complete combustion to be achieved [9 – 14].

The use of alcohol additives include methanol and ethanol are very practical in the biodiesel blends due to its miscibility with the pure biodiesel. Alcohol additives are very helpful to reduce the viscosity and density of the biodiesel which is higher compared to standard mineral diesel [9].

However, Obed M. Ali et al. say in [15] that these additives have a relatively low flash point and auto-ignition temperature compare to diesel and biodiesel fuel, and it may lead to noticeable increase in engine cycle-to-cycle variations when exceeds a certain limit. These variations might lead to both a reduction of engine output power and higher emissions; so, it is necessary to develop effective strategies to control the optimum additive ratio through gaining a better understanding of the different parameters that influence the overall combustion process.

An experiment was carried out by H.G. Howa et al. [16] on a common rail direct injection (DI) diesel engine with diesel, biodiesel and bioethanol fuel blends (B20E5) on engine performance, emissions and combustion characteristics at different engine load operations. They concluded that, the addition of higher oxygen content of ethanol as additive for biodiesel-diesel blend can be applied in compression ignition (CI) engine without any engine modifications.

Concerning the reduction of greenhouse gas emissions ethanol has proved to have a significant potential from 21% (in the case of using wheat as feedstock) to 75% (in case of sugar cane ethanol) or even to 87% (in case of wheat straw) [6].

Methanol is considered to be one of the favourable fuels for engines. Methanol has been studied in spark ignition (SI) engine application. Due to its physicochemical properties (lower cetane number, higher latent heat, and other) it is more difficult to fuel CI engines, sustain Wang L-J et al. in [11], citing a number of authors who investigated engine combustion characteristics using methanol/diesel blends. Methanol is also gaining interest as a renewable fuel, since its production derived from gasification of waste biomass followed by catalytic synthesis at high pressure provides both high yield (45–55% in weight) and energy efficiency (70–75%). In terms of cost and yield, Juan Hernandez et al. claim in this paper that methanol is better compared to ethanol. [6].

M. Mofijur et al. in their critically reviews show the benefits of using diesel-biodiesel-alcohols (ethanol) such as: reduce NOX and HC significantly, the peaks of smoke emissions were reduced, PM emission reduced, lowered particle number concentration and particulate mass emission. [4]

The massive use of alcohol-diesel blends is limited by the following factors highlighted by Juan Hernandez et al. in [6]: a) some key properties of the alcohol blends like blending stability, lubricity, viscosity and cold filter plugging point affect the injection system; b) the low cetane number that affects the combustion process is an important limiting factor; c) the fuel storage and handling difficulties derived from the high volatility of alcohols, in this respect, different technical modifications in the fuelling system are necessary, also, to avoid vapour locks there are necessary special systems for recovering of fuel vapours, when increase the alcohol amount.

It is very known that crude oil and the various petroleum fractions and products derived from it are consist of a complex mixture of various components, mostly hydrocarbons with various volatility characteristics. Volatility is defined as the tendency of a material to change from a liquid state to gaseous state. When dealing with petroleum products, the principal and significant volatility characteristics are distillation, vapor pressure, and flammability.

Evaporation characteristics influence the spray structure and affect the preparation of air–fuel mixture under different engine load conditions. Changes of the Reid vapor pressure and distillation properties do not show a linear correlation as a function of the alcohol blend ratio [17].

As we said before, transportation fuels consist of a large number of species that belong to different families of organic compounds. Thus, the usage of detailed evaporation models is restricted by these species and demands an alternative approach. Existing computational approaches introduce challenges in generalizing to realistic fuel blends due to assumptions that are introduced to describe evaporation and composition. Govindaraju et al. in their paper [18], present a review of the concerns of some
researchers regarding the modeling of evaporation characteristics of transport fuels mixtures. They also propose a model for assessing the evaporation characteristics of multicomponent fuels. Information about physical properties of each compound in the fuel is required to perform calculations using the evaporation model. It is important to note that these physical properties are functions of pressure and temperature and should be determined under standard conditions.

So, for the application of such models it takes complete research infrastructure and a lot of work. Considering the above, we assert, the determination of experimental volatility characteristics is an alternative option that requires less effort.

However, in the case of diesel-biodiesel-alcohols blends, the variance of component volatility makes it difficult to analyse overall volatility.

In this paper, we have conducted a study in order to develop a method for distillation of diesel-biodiesel-alcohols mixtures at reduced pressure.

2. Equipment description

The i-Fischer Dist D-1160 C distillation system used in our research (figure.1) is produced by Fischer Engineering, Germany and is a fully automatic vacuum distillation apparatus working according to ASTM D-1160. It can be used for the distillation of petroleum products, lubricating oils, palm oil, mixtures such as biodiesel-diesel, at atmospheric pressure and vacuum. It can also be used in an extended range - the system can be operated under atmospheric and vacuum conditions, e.g. pre-distillation at atmospheric pressure and further vacuum distillation, equally, according to ASTM D-1160. Table 1 shows the technical data of the equipment.

| Operating parameter       | Value                  |
|---------------------------|------------------------|
| Temperature               | max. 400 °C            |
| Boiling range             | max. 600 °C (equivalent to atmospheric pressure) |
| Pressure range            | 0.1 ... 760 mm Hg (Torr) |
| Standard work pressure    | 1 mmHg (Torr)          |
| Balloon size              | 500 ml distillation    |
| Sample                    | 200 ml                 |
| Ambient temperature       | max. 30 °C             |
| Power supply              | 230 V, 50 Hz           |

![Figure 1. Vacuum distillation unit - i-Fischer Dist D-1160 C](image)
3. D-1160 C Test Method description

For the first time, Distillation Measurement at Reduced Pressure has been described in 1938 by Fenske in a review of that time. This has evolved in a number of standard test methods to determine the distillation characteristics of petroleum products and fractions that would decompose if distilled at atmospheric pressure. ASTM D1160 was first published in 1951. The original version of this test method is similar to the current version, although this standard has undergone revision over time [19].

This boiling range, can be used in engineering calculations to design distillation equipment, to prepare appropriate blends for industrial purposes, to determine compliance with regulatory rules, to determine the suitability of the product as feed to a refining process, or for a host of other purposes. Many engineering design correlations have been developed on data by this test method [20].

ASTM D1160 “Standard Test Method for Distillation of Petroleum Products at Reduced Pressure” is a true analytical method the determination, at reduced pressures, of the range of boiling points for petroleum products that can be partially or completely vaporized at a maximum liquid temperature of 400 °C. D1160 is operated at a fixed distillation rate and for this, the rate of heating is adjusted, so that the distillate is recovered at a uniform rate of 6 to 8 mL/min. Moreover, interruption of the distillation is not permitted; the maximum end point is essentially dictated by the chosen operating pressure and the requirement that the liquid temperature shall not exceed 400 °C. As for any distillation method, the most important parameters having the largest impact on accuracy and precision are vapor temperature and operating pressure. For ASTM D1160, in addition, accurate volume measurement is also an important factor [21].

Temperature. The system must produce readings with an accuracy of ±0.5 °C over the range to 400 °C and have a response time of less than 200 s. The location of the vapour temperature sensor is extremely critical. The type of this sensor is very important, too. The sensor must to be able to respond sufficiently rapidly to changes in temperature such that no significant error due to lag is introduced in a rapidly rising temperature curve. This is very important when working with low pressures and the heat content of the vapours is minimal. However, in ASTM D1160, neither the mass, surface area, nor the heat transfer characteristics of the sensor is defined [21].

Distillation Pressure. Pressure is the second most important parameter to take care of, to assure reasonable reproducibility. D1160 test method is rather vague about the pressure to be applied. The sample is distilled at an accurately controlled pressure between 0.13 and 6.7 kPa (1 and 50 mm Hg). The pressure level also affects the temperature lag due to a rapidly rising vapor temperature in relation to sensor response time, thus introducing more “noise” in precision variance [21].

Volume Measurement. The boiling curve produced by ASTM D1160 is a plot of vapor temperature (AET atmospheric equivalent temperature) versus volume recovered. Volume is a function of temperature. Therefore, it is important that the intake volume measured (200 mL) has the same temperature as the distillate collected in the receiver. More, the accurate calibration and reading of the distillate receiver have a significant bearing on precision of ASTM D1160 results [21].

Accuracy. Distillation curves by D1160 will NOT match when they are obtained under different pressure conditions, or with different temperature measuring devices, or using D86 (atmospheric distillation method) [21].

4. Results and discussion

The mixtures for which a distillation method at reduced pressure was created are: B20M10 (diesel 70% + biodiesel 20% + methanol 10%) și B20E10 (diesel 70% + biodiesel 20% + ethanol 10%). The fuel samples were prepared as blended fuels: 70% diesel + 20% biodiesel + 10% ethanol / methanol, by volume, and strongly mixed manually for about 5 minutes before analyzing.

As mentioned in the previous paragraph, the distillation curves by D1160, obtained under different pressure conditions, will NOT match. So, to be able to compare the volatility of the two mixtures it is necessary that the determination is made in identical conditions. Table 2 presents these identical measurement parameters. As far as it was possible, we took care not to get out of the limits of the method. Figure 2 shows the notations of the set parameters.
Table 2. Set parameters of the method

| Set Parameters                                                                 | Symbol       | UM   | Value | Observation                                                                 |
|--------------------------------------------------------------------------------|--------------|------|-------|-----------------------------------------------------------------------------|
| Vacuum set point                                                              | Vacuum       | [torr]| 600   | T<sub>_min_</sub>                                                          |
| Minimum temperature for the thermostatic bath (TH1)                           | T<sub>_min_</sub> | °C   | 40    |                                                                             |
| Maximum temperature for the thermostatic bath (TH1)                           | T<sub>_max_</sub> | °C   | 90    |                                                                             |
| Differential temperature between the head temperature (ACT*) and the temperature of the thermostatic bath (TH1) | T<sub>_delta_</sub> | °C   | 120   | According to this preset the temperature of the thermostatic bath is controlled. |
| Distillation rate                                                              | -            | [ml/min]| 7     | According to this value the sump heater is controlled                       |
| Temperature of pre-heating the charge                                         | T<sub>_preheating_</sub> | °C   | 50    | After starting a run, the charge is pre-heated until this temperature is reached |
| Time to reach the vacuum                                                       | Vacuum       | [sec] | 1800  | If the vacuum is not reached within this time, distillation is stopped      |
| Maximum temperature difference between the sump heater T<sub>fl-heat</sub> and the charge T<sub>flask</sub> during heating up phase | dT<sub>_max</sub> | °C   | 200   | The sump heater will be automatically switched off if the value is above this limit |
| The heating constant power of the sump heater                                 | Start power  | %    | 20    | After the T<sub>_trigger_</sub> is reached the sump heater is heating with this power |
| Power after trigger                                                            | %            |      | 20    |                                                                             |
| The temperature of the receiver to have the exact volume and weight.           | T<sub>_receiver_</sub> | °C   | 60    | After entering the density and the corresponding temperature of the receiver the weight of the charge with 200 ml get to calculate.         |
| Gradient to detect T<sub>_trigger_</sub>                                      | T<sub>_trigger_</sub> | °C/sec | 0.2   | If the rise of the temperature of the vapours T<sub>_trigger_</sub> is more than this value per second the trigger is detected |

*ACT – Actual Temperature (vapor temperature measured at operating pressure)

To choose the parameters of the method, it was necessary to consider the very different volatility of the blends components. Applying the method with the determined parameters resulted in the distillation curves presented in fig. 3 and 4. As seen, three measurements were made for each type of fuel. The results are a clear indication of the repeatability of the experiment.

We wanted to verify the composition of the samples on Mid - FTIR Spectrometer, but difficulties were encountered. The equipment manufacturer's explanations were: “The alcohols interfere with the FAME determination for FAME concentrations higher than about 6.5 vol %. So, in this case, it seems
to be an interaction between FAME and alcohols such that this region is distorted, and then the normal calibration for FAME is not valid anymore. So, a new method is needed depending on the alcohol concentration, and that would have a completely new way to calculate the FAME concentration”.

We had to make this point because on the distillation curves obtained (fig. 3 and 4), the plateau believed to be the boiling area of the alcohol is more than 10 percent recovered. This could also be a cause of a volumetric measurement error.

Figure 2. Vacuum distillation parameters

There could be other causes we need to talk about. It is noticed that the stability of samples is affected by three factors mainly: temperature, water content and initial alcohol content.

Concerning the stability of the B20M10 mixture, the following remarks are given:
- biodiesel is relatively difficult to be fully described regarding its composition;
- it contains mainly a mixture of esters (obtained by a transesterification reaction);
- in non-polar compounds, it is relatively stable;
- with the addition of relatively polar compounds such as the methanol (with a molecular dipole moment of 1.7 Debye) or the ethanol (with a molecular dipole moment of 1.66 Debye) interactions and decomposition can occur;
- thus, mixtures of alcohols and fatty acids can be formed;
- the steady state of esters is adversely affected by methanol and by traces of water, (see the Le Chatelier principle);
- moreover, methanol and ethanol may have a relative reactivity in the presence of certain unsaturated component from biodiesel;
- in conclusion, the final composition of the mixture may be affected.

Concerning the stability of the B20E10 mixture, the following remarks are provided in the addition of what was given above:
- the main drawback is that ethanol is immiscible with diesel fuel over a wide range of temperatures, leading to phase separation [22];
- but, esters, biodiesel could be an additive that help to increase the stability of ethanol-diesel blends [9,23];
so, ethanol dissolves without problem in B20.

**Figure 3.** Distillation curves for B20E10
Figure 4. Distillation curves for B20M10
However, in fig. 3 it is observed that the curve B20E10-1 is slightly more distant than the other two. This could be explained by the fact that it was the first determination made with this method, and the device responded slower, given the conditions between different parameters (tab. 2).

Finally, it can be seen that, in both cases, the values obtained are very close. This justifies saying that the parameters set for determining the distillation curve are appropriate.

5. Summary and conclusions

General results concluded from this study can be summarized as follows:
- The significance of using alternative fuels can be attributed to the following aspects: (1) pursuing energy sustainability through the extended usage of those alternative fuels derived from renewable energy sources; (2) improving engine efficiency and engine-out emissions; (3) relieving the unbalanced usage of fossil fuels.
- The use of alcohol additives (e.g., methanol and ethanol) in the biodiesel blends is very practical due to its miscibility with the pure biodiesel. Equally, they reduce viscosity and density of the resulted blend.
- Biodiesel is relatively difficult to be fully described regarding its composition. With the addition of relatively polar compounds such as methanol or ethanol, interactions and decomposition can occur. Moreover, methanol and ethanol could have a relative reactivity in the presence of certain unsaturated component from biodiesel.
- Determination of physico-chemical properties of these mixtures could be difficult and new analyzing methods are required.
- Evaporation characteristics influence the spray structure and affect the preparation of air–fuel mixture. Therefore, the more accurate knowledge of the fuel volatility property is necessary.
- For the distillation of substances with high boiling temperatures at atmospheric pressure, like biodiesel, distillation at low pressures is needed because high temperatures may change its chemical structure.
- ASTM D1160 is a true analytical method of determination at reduced pressures of the range of boiling points for petroleum products that can be partially or completely vaporized at a maximum liquid temperature of 400°C.
- Distillation curves by D1160, obtained under different pressure conditions, will NOT match!
- To be able to compare the volatility of two or more different fuels it is necessary that the determination is made in identical conditions. This results in the need to find appropriate analysis parameters.
- The method presented in this paper can be applied successfully for the two blends: B20M10 (diesel 70% + biodiesel 20% + methanol 10%) și B20E10 (diesel 70% + biodiesel 20% + ethanol 10%).
- In the future, analyzes should be made in order to see if this method is applicable for higher percentage of methanol or ethanol, but also in case of other alcohols such as, butanol and isobutanol.

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Abbreviations

| ACT | Actual Temperature (vapor temperature measured at operating pressure) |
| AET | Atmospheric Equivalent Temperature |
| ASTM | American Society for Testing and Materials |
| B20 | Biodiesel20% + Diesel80% blend |
| B20E5 | Diesel 75% Biodiesel 20% Ethanol 5% Blend |
| B20E10 | Diesel 70% Biodiesel 20% Ethanol 10% Blend |
| B20M10 | Diesel 70% Biodiesel 20% Methanol 10% Blend |
| C1 | Compression Ignition |
| CO₂ | Carbon Dioxide |
| DI | Direct Injection |
| EC | European Commission |
| EU | European Union |
| FAME | Fatty Acid Methyl Ester |
| FTIR | Fourier Transform Infrared Spectroscopy |
| GHG | Green House Gas |
| HC | Hydrocarbons |
| IC | Internal Combustion |
| NOx | Nitric Oxides |
| PM | Particulate Matter |
| SI | Spark Ignition |
| T10/T50/T90(°C) | Temperatures at which 10/50/90 % of the fuel boiled |
| TH1 | Thermostatic Bath |
| Tfl | Sump Heater Temperature |
| Ti | Charge Temperature |
| Ttrigger | Temperature of The Vapours |
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