COLD FLOW PROPERTIES ANALYSIS OF WASTE COOKING OIL BIODIESEL BLENEDED WITH FOUR DIFFERENT PETRO-DIESEL USING COMPUTER-AIDED COOLING CURVE ANALYSIS

A THESIS SUBMITTED TO THE GRADUATE
SCHOOL OF APPLIED SCIENCES
OF
NEAR EAST UNIVERSITY

By
MOHAMED. I. A. SULIMAN

In Partial Fulfillment of the Requirements for
the Degree of Master of Science
in
Mechanical Engineering

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Approval of Director of Graduate School of
Applied Sciences

Prof. Dr. Nadire ÇAVUŞ

We certify this thesis is satisfactory for the award of the degree of Master of Science in Mechanical Engineering

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I hereby declare that, all the information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these rules and conduct, I have fully cited and referenced all materials and results that are not original to this work.

Name, Last Name:
Signature:
Date:

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To my parents and sibling…

ABSTRACT

Biodiesel is a renewable fuel from organic remain such as waste sunflower oil and used cooking oil. Biodiesel is acquired through the transesterification of fatty acid methyl esters (FAMEs) of waste sunflower oil. In this study, waste sunflower oil was used and it was blended with Benzene and Euro diesel which is an additive that reduces the viscosity and density of biodiesel, it was blended at different volume of fraction. The effect of thermal analysis on the waste sunflower biodiesel properties was experimentally determined. The cold flow properties was determined for each blend. The cold flow properties were determined according to ASTM D2500, ASTM D6371-05 and ASTM D97 for cloud point pour point and cold filter plugging point respectively. The cloud point (CP), the cold filter plugging point (CFPP) and the Pour point (PP) were noted as the slope changes on the cooling curve.

Keywords: Waste sunflower biodiesel; cloud point; pour point; thermal analysis;plugging point

ÖZET

Biyodizel, atık ayçiçek yağı ve kullanılmış pişirme yağı gibi organik kalıntılardan yenilenebilir bir yakıttır. Biyodizel, atık ayçiçek yağının yağ asidi metil esterlerinin (FAME) transesterifikasyonu yoluyla elde edilir. Bu çalışmada atık ayçiçek yağı kullanılmış ve biyodizelin viskozitesi ve yoğunluğunu azaltan bir katkı maddesi olan Benzene ve Euro dizel ile harmanlanmış, farklı fraksiyonlarda harmanlanmıştır. Termal analizlerin atık ayçiçeği biyodizel özellikleri üzerindeki etkisi deneysel olarak belirlenmiştir. Her harman için soğuk akış özellikleri belirlenmiştir. Soğuk akış özellikleri, sırasıyla bulut nokta akma noktası ve soğuk filtre tıkama noktası için ASTM D2500, ASTM D6371-05 ve ASTM D97'ye göre belirlenmiştir. Soğutma eğrisindeki eğim değiştikçe, bulut noktası (CP), soğuk filtre tıkama noktası (CFPP) ve Dökme noktası (PP) not edildi.

Anahtar Kelimeler: Atık ayçiçeği biyodizel; bulut noktası; akma noktası; ısı analizi;tıkama noktası

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A	Area (m^2)
$\boldsymbol{cc}$	Cooling Curve First Derivative
$\boldsymbol{C_p}$	Specific Heat (J/g°C)
g	Gravity (m/s^2)
$\boldsymbol{L}$	Latent heat (J/kg)
$\boldsymbol{M_{empty}}$	Mass of the empty pycnometer (g)
$\boldsymbol{M_{full}}$	Completely filled pycnometer mass (g)
$\boldsymbol{M}$	Mass (g)
$\boldsymbol{t}$	Time (sec)
$\boldsymbol{t_e}$	End of Solidification (sec)
$\boldsymbol{t_s}$	Start of Solidification (sec)
$\boldsymbol{T_o}$	Cooling Bath Temperature °C
$\boldsymbol{T}$	Thermocouple Temperature °C
$\boldsymbol{u}$	Velocity (m/s)
V	Volume (m^3)
$\boldsymbol{v_\theta}$	Velocity in Angular Direction (rad/s)
$\boldsymbol{v_r}$	Velocity in Radian Direction (rad/s)
$\boldsymbol{v_z}$	Velocity in Flow Direction (m/s)
$\boldsymbol{y}$	Kinetic energy correction
$\boldsymbol{z}$	Length in Flow Time (m)

Greek Symbols

$\boldsymbol{\mu}$	Dynamic viscosity ($N.s/m^2$)
$\boldsymbol{\nu}$	Kinematic viscosity (mm^2/s)
$\boldsymbol{\rho}$	Density (kg/m^3)
$\boldsymbol{\tau}$	Shear Stress (N/ m^2)

LIST OF ABBREVIATIONS

ASTM:	American Society for Testing Materials
B95K5:	95 Percent Biodiesel, 5 Percent kerosene
B90K10	90 Percent Biodiesel, 10 Percent kerosene
B85K15	85 Percent Biodiesel, 15 Percent kerosene
B80K20	80 Percent Biodiesel, 20 Percent kerosene
B100:	100 Percent Biodiesel
CCA	Cooling Curve Analysis
EN	European Standard
FAME:	Fatty Acid Methyl Ester Biodiesel
HC	Kinetic Energy Correction
ISO	International Standard Organization
WSFO	Waste sunflower oil
$\mathbf{Z_N}$	Newtonian Zero

CHAPTER 1

INTRODUCTION

1.1 Energy

Energy can be defined as that property which when an object is subjected to enables the object to perform work or heat the object. It is the capacity of an object to perform work.

Energy can be classified into two main categories which are renewable and non-renewable energy. Non-renewable energy is that category of energy which are limited in existence and are not replenishable(Boyle, 2004; Hornby, 1974).They are mostly found beneath the earth crust and advanced technological mining is require most times to enable one have access to them. They are formed from metamorphosis of organic such as dead animals, trees etc. remains over a long period of years. This type of energy sources includes natural gas, uranium, petrol diesel, coal etc(Kumar et al., 2010).

Renewable energy is a form of energy that is got from renewable sources which can be replenished over time, these includes biodiesel, biomass, solar energy, geothermal and wind (Showstack, 2016). As a result of increase in the world's population, globalization, increased technological equipment and industrialization has put pressure on the utilization of energy and its sources.

Hence, scientist and researches has predicted that most non-renewable fossil fuels will be depleted by 2040, therefore it has drawn attention to finding alternative to non-renewable form of energy such as solar, geothermal, biodiesel, hydropower, another challenge faced by researches towards non-renewable energy is the amount of pollutant emitted to the environment by its use, to ease these challenges biodiesel has been found to be an alternative to petrol diesel in the transportation sector and beyond(Lee, Park, & Daisho, 2004; Zou et al. 2016).

1.2 Biodiesel

Biodiesel can be defined as a sustainable mono alkyl esters derived from infinite oil bearing sources, such as used cooking oil, animal fat, vegetable oils sunflower oil etc through the process of transesterification (Joshi & Pegg, 2007). The transesterification process is achieved by reacting monohydric alcohols like methanol and ethanol in the presence of alkali catalyst(Narasimharao, Lee, & Wilson, 2007).

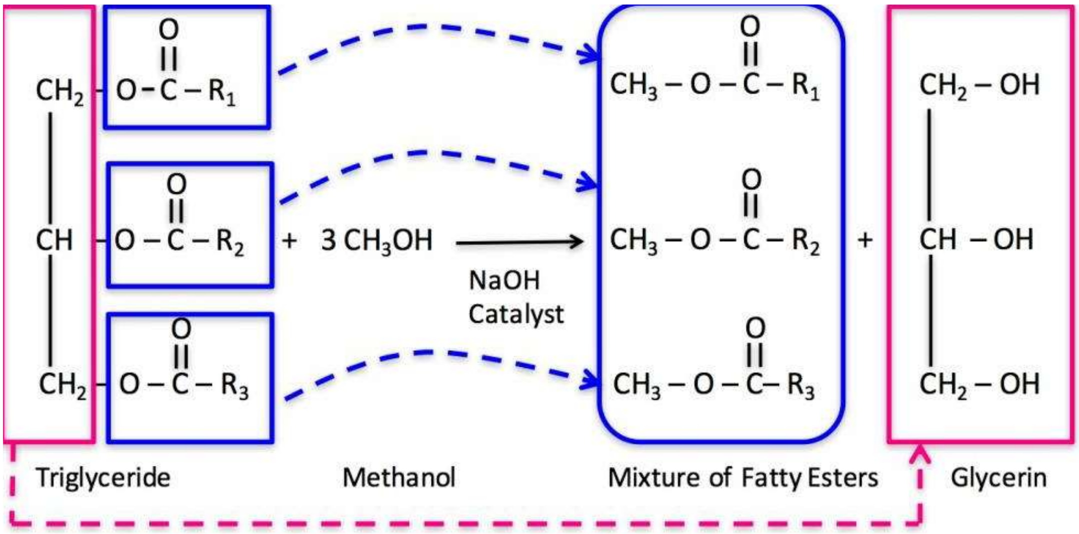


Figure 1.1: Biodiesel production process (Knothe & Steidley, 2005)

Considering the quest for high speed transportation system and agricultural equipment, biodiesel is expeditiously being used in cars, trucks and different kinds of agricultural equipments all over the world. For high performance and safety concerns, biodiesel can be blended with petrol-diesel at different percentage and can also be used alone in a diesel engine with little or no modification(Agarwal & Das, 2001).

When biodiesel is blended with petro-diesel or other fossil fuel a ‘B’ factor framework is used today to denote the concentration of the biodiesel in the blend. At different volume fraction when blended with petrol-diesel it is expressed as "BXX" with "XX" representing the content of biodiesel concentration in the blend(Blangino, Riveros, & Romano, 2008). Biodiesel can be blended in different proportion at different point, it can be blended at the point of production before delivery to fuel carrier trucks, it can be also blended by pouring specified percent (amount) of petrol diesel to biodiesel, and it can also be blended in the transportation pipeline, when transporting biodiesel. As regards the performance and quality of biodiesel it advisable that biodiesel should be used within six months from the date of production. This is as result of instability of biodiesel which can be attributed to exposure to light, heat and water that is instrumental in minimizing the quality of the biodiesel. However, the utilization of additives in its storage improves the shelf life (Simmons, Loqué, & Ralph, 2010).

In the study and analysis of biodiesel, there are some pertinent characteristic that must be put into consideration, these includes, kinematic viscosity, density, cold flow properties, acid number and oxidation stability, these parameters are greatly affected by the storage period of the biodiesel sample.

The use of biodiesel has been proved to be very advantageous and this is as a result of its environmental friendliness due to its clean way of burning, uses have expanded to heating and cooking.

1.3 Research Aim

This study aims to examine the cold flow behavior of biodiesel blend and measure the biodiesel properties including cold flow properties in terms of Cloud point (CP), Cold filter plugging point (CFPP) and Pour point (PP).
Moreover, in this study, examine the cold flow behavior of biodiesel blends during solidification by employing the computer-aided cooling curve analysis (CA-CCA) technique.

1.4 Thesis outline

Chapter one gives insight about the general aspect of energy, in particular the renewable energy in the form of biodiesel. It also explain the topic, the aims and objective are

explicitly outlined. Energy in its general form are outlined and the distribution in terms of utilization is also outlined. Chapter two gives the general information about past works done by other researches on related topic, its provides meticulous details about the literature review, the fundamental theorem as related to kinematic viscosity, density and the cold flow properties (cloud point PP, Cold fllter plugging point CFPP and pour point).
Chapter three provides us with information's regarding the materials and methods used in achieving the results obtained in the work. Chapter four focuses on the results obtained from the various analysis that was involved in the thesis and chapter five focuses on the conclusion and recommendations as regards further work that can be done to enhance this work.

CHAPTER 2
LITERATURE REVIEW OF BIOFUEL

2.1Biodiesel Properties

Biodiesel has some basic attributes which must be considered for it to replace fossil fuel, these attributes are the kinematic viscosity, density, cetane number, oxidation stability, acid number and the cold flow properties (cloud point, cold filter plugging point and pour point). Few other factors that needs to be looked into are the functionality of the biodiesel when blended with other fossil fuel blend as this will determine if the diesel engine will need little or no modification(Aydin, Bayindir, & Ilkilic, 2010) .

Table 2.1: Properties of Blend of biodiesel with kerosene (Hasan et al. 2016)

Property	**Blend Range**							**Limits**
	100:0	95:5	85:15	75:25	65:3	50:50	0.100	
Density Kgm^{-3}	875	868	861	855	849	837	807	815-870
Viscosity cSt	4.92	4.84	4.25	3.95	3.45	2.76	1.38	2-May
Flash Point ^{O}C	176	135	105	70	66	62	45	Min60diesel Min100 BD
Cloud Point ^{O}C	4	3	3	1	-2	-4	-	Max 18
Pour Point ^{O}C	2	0	-1	-2	-4	-5	-7	Max 18

2.2 Literature Review

For the biodiesel properties ie kinematic viscosity, cold flow properties and density a whole lots of approach have been used to predict its properties. Some of the attractive attributes are the renewability, clean combustion environmental friendliness and biodegradability(Isioma et al. 2013). However, biodiesel drawbacks which have limited its use are the cold flow properties, these are the properties which forecast its operability in cold region, these properties gives knowledge on the rate the biodiesel solidify (congeal) in a low temperature weather condition which in the long run clog the engine filter (Freire et al, 2012). Another drawback is the instability of the biodiesel over long period of storage(Castanheira et al. 2014; Shrestha et al. 2005).

The stability of liquid fuels is being affected by different factors, however scientist have carried out theoretical analysis and experiments to why fuel should be of high quality and remain stable even after prolong storage period(Biernat, 2015).

The essential compound of petroleum based fuel are likely to react with atmospheric oxygen and other compounds during storage. As a result of chain reaction this in turn causes contamination and corrosion to storage tanks and pipeline conveying the products and also causes a similar problem of filter plugging in the engine(Eneh, 2011).

According to Saltas et al., (2017)the impact of aging on the deposit of biodiesel produced inside the regular rail Fuel Injection Equipment (FIE). The FAME constituents was analysed, the major fuel properties and their rates of degradation. A reference test to assess the predisposition of diesel engine fuels to produce deposits was proposed.

According to Zhou, Xiong, Gong, & Liu (2017)other instrumental systems in collaboration with fourier transform spectroscopy (FTIR) was used to investigate the oxidation degradation of mixtures of biodiesel. It was found that the TD-DES method displayed outstanding forecasting operation for FTIR and TGA in the evaluation of oxidative degradation.

In the production of biodiesel by the transesterification process, the impact of impurities created in the biodiesel was investigated according to (Banga & Varshney, 2010)and how the impurities can be removed. The impact of elongated storage period on the performance of biodiesel is also underscored.

The result obtained displayed a substantial deviation from the values gotten from national metrological institute when pycnometer is used in measuring the density of biodiesel (Lima et al., 2010), however, they were still within the operating standards for commercial application in Brazil.

Blending petrodiesl with biodiesel can be possible to compliment the viscosity values so as to aid its use in motor engine. All fuels have the tendency to form crystal when subjected to certain low temperature, the increase in viscosity of the biodiesel and the viscosity of the blends varies among the biodiesel and petro-diesel depending on their blend proportion (Tat & Van Gerpen, 1999).

Properties of waste frying oil was analyzed for biodiesel got from it and the biodiesel yield of 99% was got from it. After the analysis it was observed that the range of standard was within the specification and conform to diesel engine operating(Kulkarni & Dalai, 2006).

2.3 Advantages of biodiesel

- Biodiesel can be utilized in various diesel engines, mostly newer ones, biodiesel present no difficulty in using it; free of Sulphur and aromatics and emits less greenhouse gases and air pollutant than nitrogen(Wilson, 2003).
- Utilization of biodiesel in our daily domestic equipment decreases dependence on finite fossil fuel reserve.
- When biodiesel is blended even as low as B2 to the ratio of 98% in proportion, it is observed that the amount of toxic carbon based emission is reduced significantly(How, Masjuki, Kalam, & Teoh, 2014).
- The effectiveness of biodiesel is the same as petro-diesel notwithstanding its lubricity benefits that non-renewable energy sources don't have.
- It has been confirmed scientifically that fumes from biodiesel exhaust is less harmful to human health as compared to that of petro-diesel. Hydrocarbons and nitrited compounds which causes cancer have a very low level of emission in biodiesel.
- Significant favorable position of using biodiesel is that it can be utilized in operating existing diesel engines without or less adjustments and can supplant fossil derivative fuel to become the most favored essential transport energy source.

2.4Disadvantages of biodiesel

- Palm oil is one of the best biofuel source in the world, however considering the environmental damage done by palm oil. People discovered that palm oil was a great material that can be utilized in the production of biofuel, not minding the environment issue and drawbacks of producing palm oil because forest was cleared and burnt to allow for palm oil plantation by so doing burning fossil fuel and thereby defeating the purpose of utilizing biodiesel.
- One of the major drawback of biodiesel is the cold flow properties, biodiesel gel and solidify when operated in cold weather condition, this clog the fuel filter of the engine in cold weather thereby making it difficult to pump into the engine, hence reducing the efficiency of the engine. Furthermore, this depend on the product the biodiesel is produced from and its blends(Monirul et al., 2015).
- Biodiesel on the average is cleaner than fossil fuel, however it tends to produce about 10% more of nitrogen oxide; this in turn contributes to acid rain and formation of smog which increase pollution around cities.
- To fully harness the potential of biofuels, the waste product from our food crops should be used for biofuel production or else there will be food shortage as result of utilizing consumable crops for biofuel production.

2.5 The Concept of Viscosity

In the course of transporting fluid through a pipeline the viscosity (μ) is widely utilized. Viscosity is the resistance to the flow of fluid experienced in flowabilty of a fluid. It is the basic characteristic of fluid which defines the way and manner in which the internal friction of integral of the intermolecular forces relates to each other. It is most times considered as the thickness of a fluid, that is to say that the thicker the fluid the higher the viscosity. Example biodiesel, SAE 40 engine oil, syrups etc. however thin fluid like water and acetone have lower viscosity.

The intermolecular particles of the fluid are held together by internal cohesive force, which makes them to be firm and slow in flow. However, when heat is applied the internal molecules gain energy and as a result disintegrate the cohesive forces, this action causes the molecular particles to slide over each other gradually and as the temperature increases, it becomes rapid making the fluid less viscous. The viscosity of a fluid is directly proportional to temperature. Some parameters that depends on viscosity for its determination are:

- Reynolds number
- Prandlt number

2.6 Types of Viscosity

There are two main types of viscosity, these are

- Dynamic Viscosity
- Kinematic Viscosity

2.6.1 Dynamic Viscosity

The dynamic viscosity or shear viscosity of a fluid demonstrate the fluid resistance to flow in such fluid the adjacent layer move parallel to each other at a non-identical speed. Consider a fluid flowing with a constant speed $u$that is confined in a layer between two horizontal plates, fixed at one end and free at the other end as shown below.

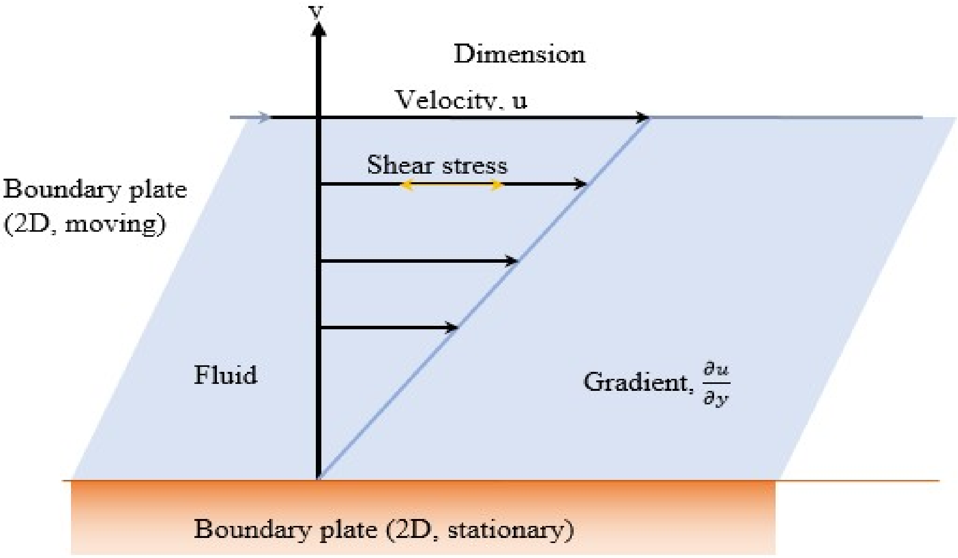


Figure 2.1: The flow of liquid between two plate in parallel

Let the movement of the top plate be infinitesimally small so as it moves parallel to each other, the speed of such particles varies linearly from zero at the bottom to u at the top. The fluid is made to move in layer and each layer moves faster than the layer below it, this generates frictional force between the layers which opposes each other. The movement of the top plate generate a force on it which creates an equal and opposite force on the down plate.

To maintain a constant speed on the plate an external force F is applied which is directly proportional to the speed u and area $A$of each plate,but inversely proportional to the separation $y$of the two plates. That is;

$$F=\mu A \frac{\partial u}{\partial y} \qquad (2.1)$$

Where

μ is a proportionality factor and is known as the dynamic viscosity.

$\frac{\partial u}{\partial y}$is the rate of shear deformation or shear velocity.

the viscous force can be expressed by differential equations, as seen in the illustration below.

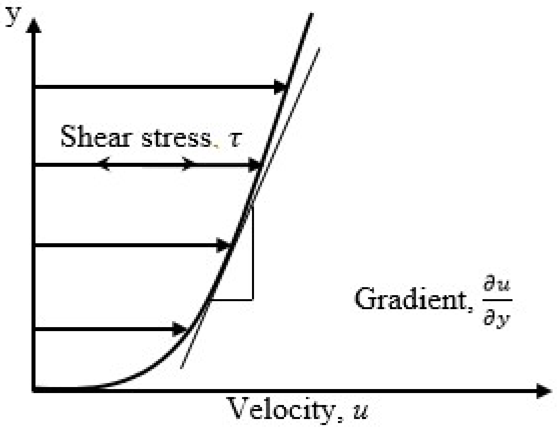


Figure 2.2: Illustration of viscous force differential expression

$$\tau = \frac{\partial U}{\partial y} . \mu \tag{2.2}$$

$$\mu = \frac{F}{A}$$

Consider:

$\tau = \frac{F}{A} \cdot \frac{\partial U}{\partial y}$ as thevelocity of shear

2.6.2 Kinematic Viscosity (*V*)

Kinematic Viscosity can be defined as the ratio of the dynamic viscosity to the density of a substance at the same temperature. Kinematic viscosity is measured in$\frac{mm^2}{s}$

$$\nu = \frac{\mu}{\rho} \tag{2.3}$$

Let:
$v$be the kinematic viscosity,
ρ is the fluid density
μdynamic viscosity.

2.6.3 Viscosity Measurement

In the measurement of viscosity different type of viscometer is used, a single measurement with a viscometer cannot be used to ascertain the accuracy of the value of the viscosity. Hence more parameter is required to accurately ascertain the value of a viscosity for such a fluid a rheometer is used to measure the kinematic viscosity (Sahin & Sumnu, 2006). The kinematic viscosity of the fluid is acquired by measuring with a stop watch the time taken for the fluid to flow freely under through a capillary viscometer from a specified mark to another while subtracting the energy correction factor from it and multiplying the value by the viscometer constant. There are different types of viscometers, these are:

- Rotational viscometer
- Capillary Viscometers (glass capillary being the most common)
- Orifice viscometer
- High temperature low shear rate viscometer
- Vibrational Viscometer
- Falling Sphere Viscometer
- Ultrasonic Viscometer
- Rheometers
- Bubble Viscometers

2.7 Theory of Capillary Viscometer

Hagen-Poiseuille law provides us with the pressure drop in an incompressible and Newtonian fluid flowing in a hollow pipe of regular cross section in a laminar flow pattern.

To ascertain the accuracy of Hagen-Poiseuille equation certain assumptions are made which includes:

- The capillary most is upright with regular cross section to allow for easy flow and the diameter shorter than the cross section of the pipe.
- It must be an incompressible fluid and also Newtonian fluid.

Consider a completely created laminar fluid flow along a straight vertical pipe of round cross section as shown in Figure 2.3. Rotational symmetry is considered to make the fluid two-dimensional axisymmetric and let the pivot in the tube of the liquid pariticles be taken as the Z-axis(Viswanath, Ghosh, Prasad, Dutt, & Rani, 2007.).

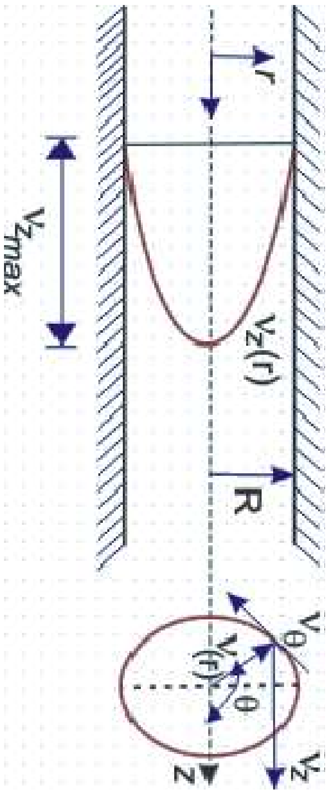


Figure 2.3: Diagrammatic illustration of Hagen -poiseuille fluidflow

Consider :

$$v_r = 0,\ v_\theta = 0,\ v_z \neq 0, \qquad (2.4)$$

From continuity equation in cylindrical coordinate

$$\underbrace{\frac{\partial v_r}{\partial r}}_{0} + \underbrace{\frac{\partial v_r}{r}}_{0} + \frac{\partial v_z}{\partial z} = 0 \qquad (2.5)$$

For rotational symmetry

$$\frac{1}{r} \cdot \frac{\partial v_\theta}{\partial \theta} = 0 \qquad (2.6)$$

$\frac{\partial v_z}{\partial v} = 0$Which means

$$v_z = v_z(r, t)$$

Introducing

$$\frac{\partial}{\partial \theta}(any\ quantity) = 0, v_\theta = 0,\ v_r = 0, \text{and} \frac{\partial v_z}{\partial z} = 0$$

In obtaining the cylindrical coordinate system in the Z direction according to Navier Stokes.

$$\frac{\partial \mathrm{v}_z}{\partial t} = -\frac{1}{\rho} \cdot \frac{\partial p}{\partial z} + v\left(\frac{\partial^2 v_z}{\partial r^2} + \frac{1}{r} \cdot \frac{\partial v_z}{\partial r}\right) \text{In Z direction} \qquad (2.7)$$

The governing equation for a continuous flow can thus be represented

$$\frac{1}{r} + \frac{\partial^2 v_z}{\partial r^2} \cdot \frac{\partial v_z}{\partial r} = \frac{\partial p}{\partial z} \cdot \frac{1}{\mu} \tag{2.8}$$

Solving equations with boundary conditions
At

$$r = 0;\ v_z\ is\ finite$$
$$r = R;\ v_z = 0$$

It can be obtained that

$$v_z = \frac{R^2}{4\mu}\left(-\frac{\partial p}{\partial z}\right)\left(1 - \frac{r^2}{R^2}\right) \tag{2.9}$$

Where

$$-\frac{\partial p}{\partial z} = \frac{\Delta p}{L} \tag{2.10}$$

Let the capillary be parabolic and considering the velocity distribution across it, the velocity flow rate (Q) is acquired from the expression below by integrating it.

$$Q = \int_0^R 2\pi\, v_z r \partial r \tag{2.11}$$

Equation 2.9 and 2.10 when substituted into 2.11 (Q) is obtained as

$$\mathrm{Q} = \pi \frac{\mathrm{R}^4}{8\mu}\left(\frac{\Delta \mathrm{p}}{\mathrm{L}}\right) \tag{2.12}$$

Equation 2.12 is called Poiseuille’s equation

$$Q = \frac{v}{t} \tag{2.13}$$

Let

$$the\ rate\ of\ flow\ \ be\ Q$$
$$v\ to\ be\ the\ volume\ of\ the\ liquid$$
$$t\ be\ the\ time\ taken$$

$$\text{v} = \frac{\mu}{\rho} \tag{2.14}$$

Consider the arrangement to be vertical, its height (h) depends on the hydrostatic pressure.

$$\Delta pgh$$

If,

$$v = \frac{\pi g H R^4}{8Lv}\ .t \tag{2.15}$$

The constant of the viscometer to K

$$K = \frac{\pi g H R^4}{8Lv} \tag{2.16}$$

Or

$$K = \frac{\pi g H D^4}{128Lv} \tag{2.17}$$

Therefore,

$$v = kt \tag{2.18}$$

Equation 2.18 has been the bases for the design of many viscometers, from Equation 2.19, a known density and viscosity of a liquid is obtained in the calibration of K-value. (Viswanath et al., 2007).

$$\mu = K\rho t \tag{2.19}$$

2.8 Kinetic Energy Correction (HC)

When carrying out the experiment, some errors are bound to occur in measuring the viscosity, to fix these error in the experiment the kinetic energy correction factor is subtracted from the time taken for the fluid to flow under gravity.

$$v = K(t - y) \qquad (2.20)$$

Where: K is theviscometer capillary number constant
y is the kinetic energy correction

2.9Density

The density of the biodiesel or any substance is the mass in grams per unit volume (m^3) of the biodiesel or substance.
It is expressed mathematically thus,

$$\rho = \frac{m}{v} \qquad (2.21)$$

Where:V is the biodieselvolume
m is the biodiesel mass
ρ is the density

2.10 Cold Flow Properties of Biodiesel

The cold flow properties of biodiesel are the properties of the biodiesel that determines its proper functionality or operability in a cold temperature condition or region. This includes

- Cloud point
- Pour point
- Cold filter plugging point

However, minimum temperature in which biodiesel can function effectively can be increased due to the presence of high level of saturation from animal to vegetable oil source. When the biodiesel is allowed in a cold temperature the constituent of the biodiesel develops crystal wax. These are some of the important drawback of biodiesel, these crystal wax cause the engine to experience start-up problem(Blanco Fonseca et al., 2010).

2.10.1 Cloud Point (CP)

The cloud point is one of the important properties of biodiesel that its knowledge needs to be sufficient in the use of biodiesel, the cloud point of a biodiesel is temperature at which the biodiesel indicates the first crystal like formation in its appearance when subjected to cold temperature condition(Ramadhas, 2016). The cloud point measurement was done as per (ASTM D2500) standards.

2.10.2 Pour Point (PP)

In the study of biodiesel it is observed that after the cloud point is reach, further subjection of the biodiesel to low temperature causes a gel formation to develop, at this temperature the where the biodiesel can no longer flow (it loses its flow ability) is considered as the pour point of the biodiesel. It is lower than the cloud point(Duffield, Shapouri, Graboski, McCormick, & Wilson, 1998). The pour point measurement was done as per (ASTM D97-2005, 2005).

2.10.3 Cold Filter Plugging Point (CFPP)

The cold filter plugging point is the minimum temperature at which a biodiesel volume flow rate passes through when cold within a specified conditions.It shows the lowest temperature that a biodiesel can be used and yield a trouble free flow in the system. After this temperature the biodiesel starts to clog the filter due to crystal formation. CFPP is often used to indicate the lowest operable temperature of a biodiesel. The cold filter plugging point measurement was done as per (ASTM-D6371-05, 2005).

2.11Thermal Analysis

Thermal analysis is a concept of materials science where the properties of materials are studied as they vary with temperature. Several methods are normally used, which are differentiated from one another by the property which is measured (Paulik, Paulik, & Erdey, 1966).

2.11.1 Newtonian thermal analysis

The heat flow produced in course of solidification of the biodiesel sample can be displayed by the heat of balance equation given below(Kierkus & Sokolowski, 1999).

$$\frac{dQ}{dt} - MC_p\frac{dT}{dt} = UA(T - T_0) \qquad (2.25)$$

Given that:

M is the mass of the sample,

C_p is the specific heat of the sample

$T$is the sample temperature,

The time is given by t,

The overall heat transfer coefficient is given by U,

And taking A to be the surface area of the sample

T_0 To be the temperature of the cooling bath

The heat produced during freezing is given by Q

Let us assume that in course of cooling no phase transformation took place $\frac{dQ}{dt} = 0$. The cooling rate of the biodiesel sample can be expressed mathematically thus,

$$\frac{dT}{dt} = -\frac{UA(T-T_0)}{MC_p} = Z_N \qquad (2.26)$$

In the above equation $Z_N$is considered as the Zero Curve or simply, the baseline

The total latent heat *L* is mathematically expressed thus,

$$L = \frac{Q}{M} = C_P \int_{t_s}^{t_e} \left[\left(\frac{dT}{dt} \right)_{cc} - Z_N \right] dt \qquad (2.27)$$

Considering:

t_e and t_s as the end and start time for solidification, the cooling curve first derivative can be taken to be *cc*

The heat produced during freezing of the sample is written thus,

$$L = C_p x \text{ (Area between derived cooling curve and zero curve)} \qquad (2.28)$$

Equation 2.28 is useful when the C_p of the biofuel is known. The total area inside the rate curve and the Newtonian baseline, as a fraction of total area between these two curves, the solid quantity at*t* during freezing is found(Evcil, Al-Shanableh, & Savas, 2018)

CHAPTER 3
MATERIALS AND METHODS

3.1 Materials

In this work, feedstock of Waste cooking oil was collected from restaurants and cafes and was utilized in the production of biodiesel sample called Waste Cooking Oil Methyl Ester (WCOME). In the preparation of the sample Benzene 95, Benzene 98, Euro diesel summer and Kerosene from 5-25 in percentage were blended together with WCOME, at a 5% interval for each blend making it 20 samples, they were poured in an identical bottle of equivalent amount for each sample.The concentrations of these petro-fuel in the blends were set as shown below on Table 3.1.

Table 3.1: The concentrations of these petro-fuel in the blends

No of Sample	Fuel	Denote	Meaning
1	**Pure Biodiesel**	B100	100% biodiesel
2	**Benzene 95**	BB510	90% biodiesel 10% Benzene 95
3		BB5 15	85% biodiesel 15% Benzene 95
4		BB5 20	80% biodiesel 20% Benzene 95
5		BB5 25	75% biodiesel 25% Benzene 95

3.2 Accessories

In achieving the ASTM standards for the biodiesel sample, the following instruments were utilized.

- Funnel
- Stirrer/Spatula.
- Measuring cylinder (1000ml)

- Pipette (10ml)
- Beaker (2000ml and 5000ml)
- Storage bottles (750ml)
- Viscometer holder
- Vacuum Syringe

Before the use of the instruments they were properly cleaned with locally prepared detergent, the detergent was prepared to contain 70% distilled water, 15% of hydrogen peroxide and 15% muriatic acid and the process was finished by rinsing with acetone and allowed to dry up.

Funnel

The funnel is used for conveying the biodiesel into the bottles and viscometer without spilling the product

Figure 3.1: Funnel

Measuring Cylinder (1000ml)

The measuring cylinder is used to measure the calculated volume of the biodiesel sample so it can be poured into the beaker or the bottles.

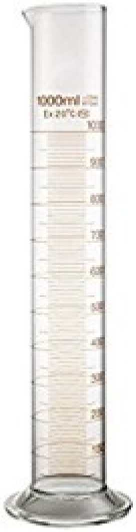


Figure 3.2: Measuring Cylinder

Beakers

Beaker is a bowl used for the measurement of the sample and also in most cases it serves as the heating bath for the ubbelode capillary viscometer, it also serves as a storage tank for the water and the various samples.

Figure 3.3: 2000ml Beaker

Glass Pipette

The glass pipette is a device used in measuring and conveying the biodiesel sample into the capillary viscometer.

Figure 3.4: Pipette

Viscometer Holder

The device holds theubbelohde capillary viscometer upright in the beaker and prevent it from falling in the cooling or heating bath.

Figure 3.5: Viscometer holder

Vacuum Syringe

This is a device use for supping (suction) the biodiesel sample into the viscometer bulb during the measurement of the kinematic viscosity.

Figure 3.6: Vacuum Syringe

3.2Temperature Measurement

Temperature plays a key role in acquiring the kinematic viscosity and density of the biodiesel products. Sometimes in this measurement a slight temperature change causes a significant change also in the kinematic viscosity and density of the sample.

In stabilizing the temperature effect, a thermostat is used in the cooling bath in conjunction with mercury thermometer.

The thermostat plays a key role by putting on and putting off the compressor automatically keeping the temperature of the cooling bath stable.

Figure 3.7: Thermostat reading

3.3 Kinematic Viscosity

The kinematic viscosity which is the internal resistance to flow of the biodiesel sample for this experiment was acquire using the Ubbelohde Viscometer ISO 3105/DIN51 562/Part1/BS188/NFT 60-100, Ref.No.501, 530, 532. The test was done according to ASTM D455.

The test was carried out in two steps

- Before the cooling of the sample
- After the cooling of the sample

3.3.1 Before Cooling

The test was carried out before the cooling of the sample at various temperatures, this was done to ascertain the effective functionality of the biodiesel at each temperature. The temperature are as follows; 0°C, 5°C, 10°C, 20°C 30°C and 40°C, the measurement was done with the accuracy of ±0.1°C.

The kinematic viscosity at 0°C, 5°C and 10°C was acquired by measuring 20ml of the biodiesel sample with a pipette and pouring it into the Ubbelohde capillary Viscometer and placed in a cooling bath for 15 to 20minutes for homogeneity in the temperature respectively for 0°C, 5°C and 10°C temperature. the sample was pressurized in the viscometer for it to move upward with the help of the suction syringe. The sample in the capillary viscometer was allowed to flow freely under gravity and the time taken for the sample to flow from mark M1 to M2 on the capillary viscometer was recorded. The process was carried out three times consecutive while taking the average time.

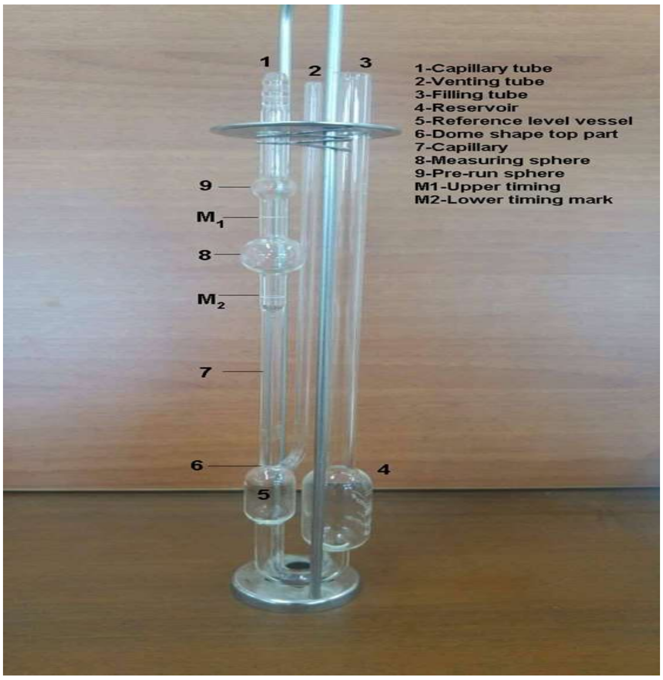


Figure 3.8: Ubbelohde type 525-10/I

After acquiring the average time the kinetic energy correction factor (y) was taken from the time and the viscosity constant (K) was used to multiply the remainder according to equation 3.1

$$v = K(t - y) \tag{3.1}$$

Where:

The kinematic viscosity (mm^2/s) be given as *v*
t is the average time (s)
y is the kinetic energy correction factor
K is the viscometer constant

For the kinematic viscosity measuring range of 6-30$\left({mm^2}/{s} \right)$,The capillary viscometer constant for this experiment was chosen according to the manufacturers catalog as stated in Table 3.1 where K = 0.02944. The ubbelohde capillary viscometer utilized in this experiment is type (Ic)

For the temperatures of 20°C 30°C and 40°C a heating bath was utilize in acquiring and maintaining the temperature, the heating bath was heated to 20°C and the previous procedures was followed, likewise for 30°C and 40°C.

Table 3.2: Datasheet for Viscometer Specification

Type No.	Capillary No	Capillary Dia. 1± 0.01[mm]	Constant K	Measuring range [mm^2/s]
525 03	0c	0.36	0.002856	0.6 … 3
525 10	I	0.58	0.00982	02 … 10
525 13	Ic	0.78	0.02944	06 … 30
525 20	II	1.03	0.08947	20… 100

525 23	IIc	1.36	0.2812	60… 300

Table 3.3: Datasheet forCorrection of Kinetic Energy for different Viscometers

Flow .time	**Capillary No 0**	**0c**	**0a**	**I**	**Ic**	**Ia**	**I**
40	. —^{B}	—.^{B}	—.^{B}	1.03	0.45	0.15	0.10
50	. —^{B}	—.^{B}	—.^{B}	3.96	0.66	0.29	0.07
60	—^{B}	—.^{B}	—^{B}	2.75	0.46	0.20	0.05
70	—.^{B}	—.^{B}	—.^{B}	2.02	0.34	0.15	0.04
80	—.^{B}	—.^{B}	4.78.^{B}	1.55	0.26	0.11	0.03
90	—.^{B}	—.^{B}	3.78^{B}	1.22	0.20	0.09	0.02
100	—.^{B}	7.07^{B}	3.06^{B}	0.99	0.17	0.07	0.02
110	—.^{B}	5.84^{B}	2.53	0.82	0.14	0.06	0.01
120	—.^{B}	4.91^{B}	2.13	0.69	0.12	0.05	0.01
130	—.^{B}	4.18^{B}	1.81	0.59	0.10	0.04	0.01
140	—.^{B}	3.61^{B}	1.56	0.51	0.08	0.04	0.01
150	—.^{B}	3.14^{B}	1.36	0.44	0.07	0.03	0.01
160	—.^{B}	2.76	1.20	0.39	0.06	0.03	0.01
170	—.^{B}	2.45	1.06	0.34	0.06	0.02	0.01
180	—.^{B}	2.18	0.94	0.30	0.05	0.02	0.01
190	—.^{B}	1.96	0.85	0.28	0.05	0.02	0.01
200	10.33^{B}	1.77	0.77	0.25	0.04	0.02	0.01
225	8.20	1.40	0.60	0.20	0.03	0.01	0.01
250	6.64	1.13	0.49	0.16	0.03	0.01	<0.01
275	5.47	0.93	0.40	0.13	0.02	0.01	<0.01
300	4.61	0.79	0.34	0.11	0.02	0.01	<0.01

Table 3.2: Continue

Flow	**.Capillary No**						
.time	**.0**	**.0c**	**.0a**	**.I**	**.Ic**	**.Ia**	**.I**
325	3.90	0.66	0.29	0.09	0.02	0.01	
350	3.39	0.58	0.25	0.08	0.01	0.01	
375	2.95	0.50	0.22	0.07	0.01	0.01	
400	2.59	0.44	0.19	0.06	0.01	<0.01	
425	2.30	0.66	0.29	0.09	0.01	<0.01	
450	2.05	0.58	0.25	0.08	0.01	<0.01	
475	1.84	0.50	0.22	0.07	0.01		
500	1.66	0.44	0.19	0.06	0.01		
550	1.37	0.23	0.1	0.03	0.01		
600	1.15	0.20	0.09	0.03	0.01		
650	0.98	0.17	0.07	0.03	<0.01		
700	0.85	0.14	0.06	0.02	<0.01		
750	0.74	0.13	0.05	0.02	<0.01		
800	0.65	0.11	0.05	0.01			
850	0.57	0.10	0.04	0.01			
900	0.51	0.09	0.04	0.01			
950	0.46	0.08	0.03	0.01			
1000	0.42	0.07	0.03	0.01			

[A] The correction seconds stated are related to the respective theoretical constant

[B] For precision measurement, these flow times should not be applied. Selection of a viscometer with a smaller capillary diameter is suggested

3.3.2Steps in measuring the kinematic viscosity

1. Clean all devices, the viscometer and all necessary equipment needed to carry out the experiment
2. The viscometer is then filled with sample at a volume of 20mL
3. After filling the viscometer, the assembly is then brought into the cooling bath and allowed to remain there for at least 15minutes for the sample to attain homogeneity.
4. When the sample have fully attain homogeneity the suction syringe is then used to sup the sample in the viscometer, the airlet (vent hole) is closed while pressure is applied from the syringe.
5. The biodiesel sample is then allowed to flow under gravity from mark M1 to M2 on the viscometer.
6. At mark M1 the stop watch is started and at mark M2 the stop watch is stopped and the time taken for the fluid to flow from M1 to M2 is recorded.
7. The process is repeated three consecutive times and the average result is recorded.

3.4Density Measurement

The density of the biodiesel sample was acquired using a pycnometer with a bulb of 99.693mL by volume. The density of the biodiesel sample was acquired and determined by the use of this device, the excess fluid and air bubbles is discharge from the glass stopper at the top of the pycnometer.

An electronic weighing balance that is very sensitive is used to measure the combine weight of the pycnometer and the fluid at a stipulated temperature before and after cooling of the biodiesel sample.

The density of the sample is obtained by considering the following, a cleaning solution was used to clean the pycnometer properly and rinsed with acetone and allowed to dry, and an electronic weighing balance was used to measure the weight of the empty pycnometer and recorded.

The pycnometer was then filled with the biodiesel sample and the glass stopper placed on top of it to allow for excessive fluid flow and air bubbles to be discharged, the entire assembly was then placed in the temperature controlled cooling bath at the respective temperature of 0℃, 5℃, 10℃ and 15℃ and allowed for homogeneity for about 20 minutes after which the electronic weighing balance was used to determine the mass of the sample by measuring it again.

To acquire the actual density of the biodiesel sample, the mass of the empty pycnometer was subtracted from the mass of the pycnometer when filled with the sample and the result is divided by the volume of the pycnometer as given by Equation 3.2

$$\rho = \frac{(M_{full} - M_{empty})}{V} \tag{3.2}$$

Where:

ρ (kg/m^3) is the density

M_{full} is the sample filled pycnometer mass (g)

M_{empty} is the mass of the empty pycnometer (g)

V be taken as volume of the pycnometer

Figure 3.9: Setup assembly of cooling bath and pycnometer

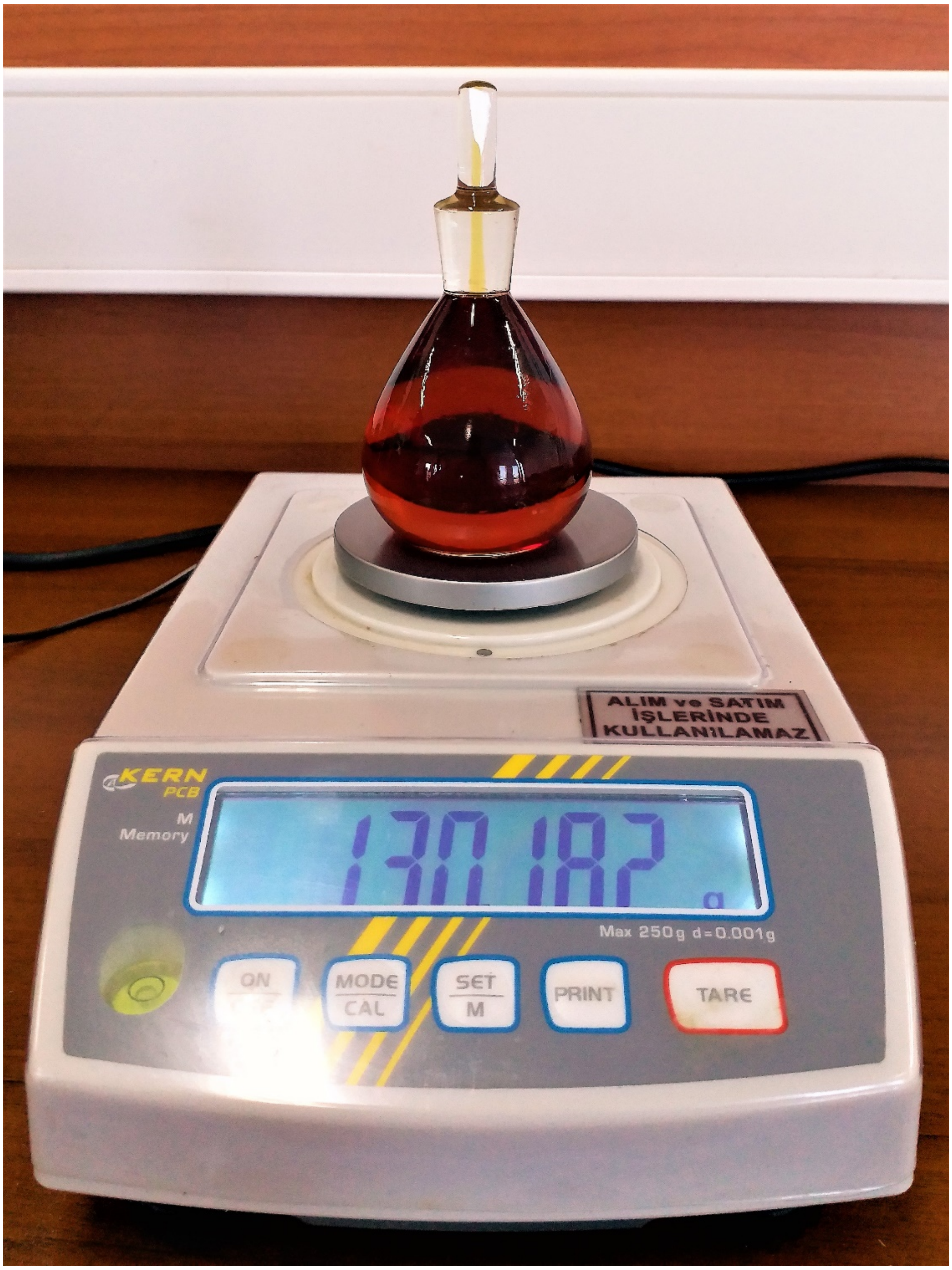


Figure 3.10: Density measurement setup

Figure 3.11: Empty pycnometer

3.4.1 Steps in measuring the density of biodiesel sample

1. The pycnometer and all devices to be used in the experiment are washed and rinsed with acetone before carrying out the experiment
2. The mass of the empty pycnometer is measured and recorded
3. The pycnometer is then filled with the sample
4. The temperature of the cooling bath is set to the required temperature
5. The complete assembly is then placed in the cooling bath and allowed there for about 15 to 20 minutes for it to attain homogeneity
6. After which, the mass of the sample is then measured using the electronic weighing balance
7. The process is repeated three times and the average value from the results obtained is recorded.

3.5 Cold Flow Properties Measurement

The cold flow properties were measured according to

1. American Standard Test Method, ASTM D2500 for cloud point,
2. ASTM D6371-05 for cold filter plugging point
3. ASTM D97-2005 for pour point.

The samples are as follows

Waste Cooking Oil Methyl Ester (WCOME) biodiesel blended with Benzene 95, Benzene 98, Euro diesel summer and Kerosene from 5-25 in percent, with a 5% interval for each blend making it 20 samples

The following component parts serves a critical purpose in the setup

- Cooling bath
- Data logger
- Cylinder jacket
- The test jar
- Insulators (Styrofoam)
- Refrigerator units
- Coolants (Alcohol)
- Thermostat
- Thermocouples
- Compressor system coil

To measure the cold flow properties the coolant (alcohol) was poured into the cooling bath and insulated properly to avoid heat transfer with the surrounding and also to maintain the internal temperature of the cooling bath.

The cooling bath was powered on and the temperature of the cooling bath was brought down by the compressor system coil to -17°C, this temperature was regulated by a thermostat attached to the refrigerator unit. The thermostat stabilizes the temperature so it does not fall below or go above the stipulated temperature. To maintain a homogenous temperature in the cooling bath, a stirrer is used to stir the coolant in the cooling bath.

A pipette was used to measure 45ml of the biodiesel sample and poured into the test jaw and the test jaw placed in a cylinder jacket that was clamped in the middle of the cooling bath. A T-type thermocouple was placed in the middle of the cooling bath so as to measure the temperature (T_o) of the coolant. Two thermocouples were further placed in the test jar to monitor the temperature of the biodiesel sample in the test jar. One of the thermocouple T_2 was placed 3mm just above the bottom of the test jaw and the second thermocouple T_1 was placed at a distance of 3.5mm just below the surface of the sample in the test jaw.

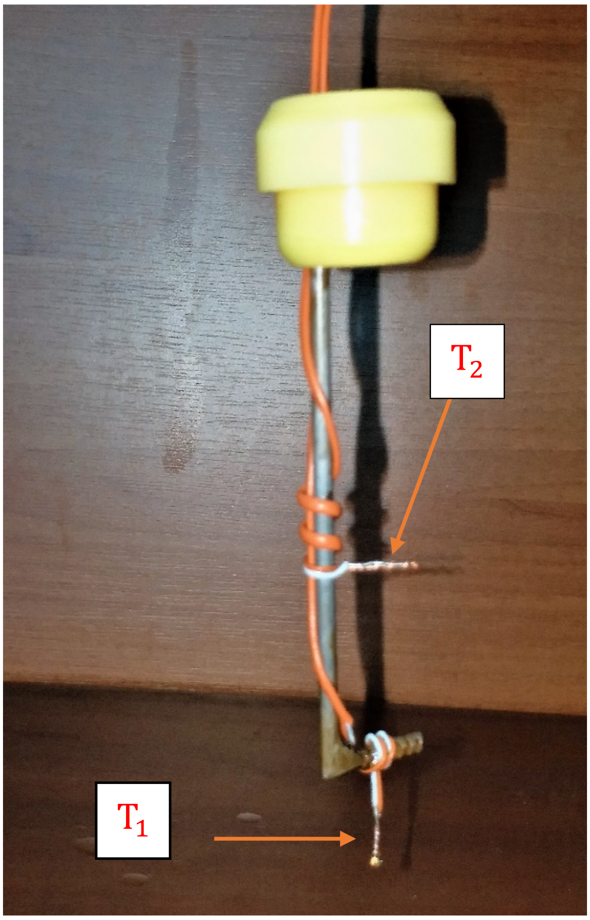


Figure 3.12: Thermocouple labelling for cold flow properties

From Figure 3.15, the thermocouple labelled T_1 is used to measure the temperature at which the cloud point (CP) occur when the temperature of the biodiesel sample start dropping and the second thermocouple T_2 keeps record of the temperature when the sample can no longer be poured, that is the pour point (PP).

This process is achieved by periodically inspecting the sample as the temperature decreases until for the cloud point, a first crystal wax formation is seen at the bottom of the test jar the cooling bath was allowed to continue to the point the biodiesel sample became solid wax and could no longer be poured as a result of gel formation, the temperature at this point is the pour point and the thermocouple T_2displays it on the computer and was recorded to be the pour point of the biodiesel.

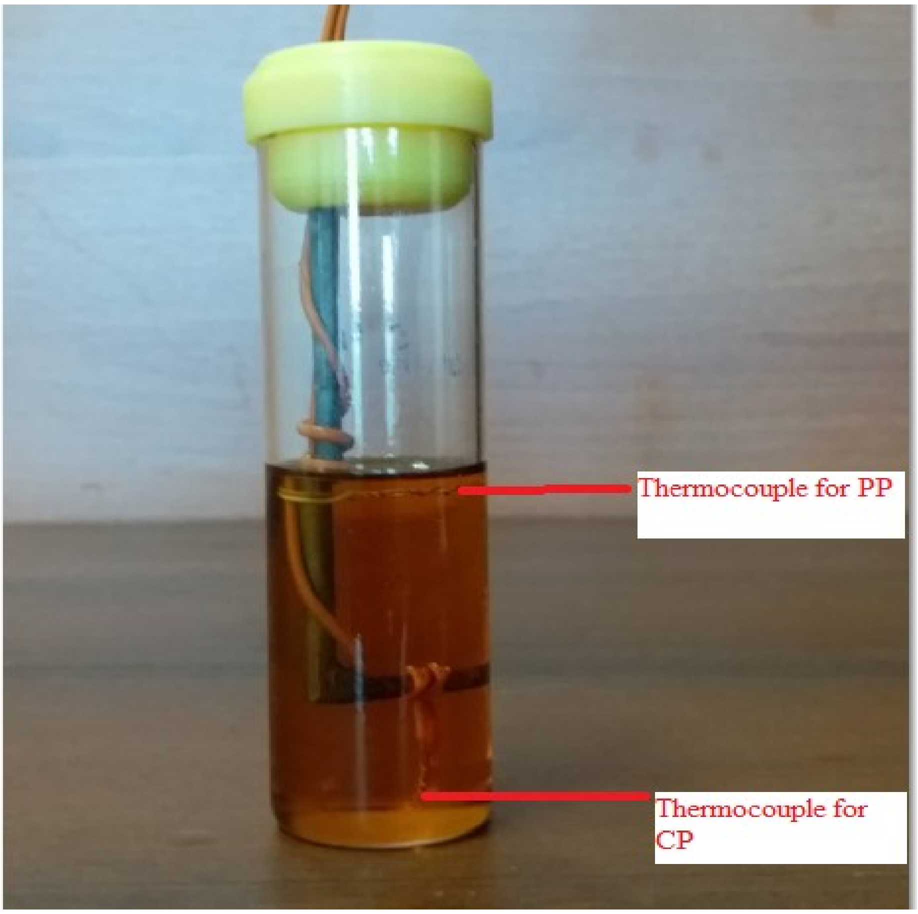


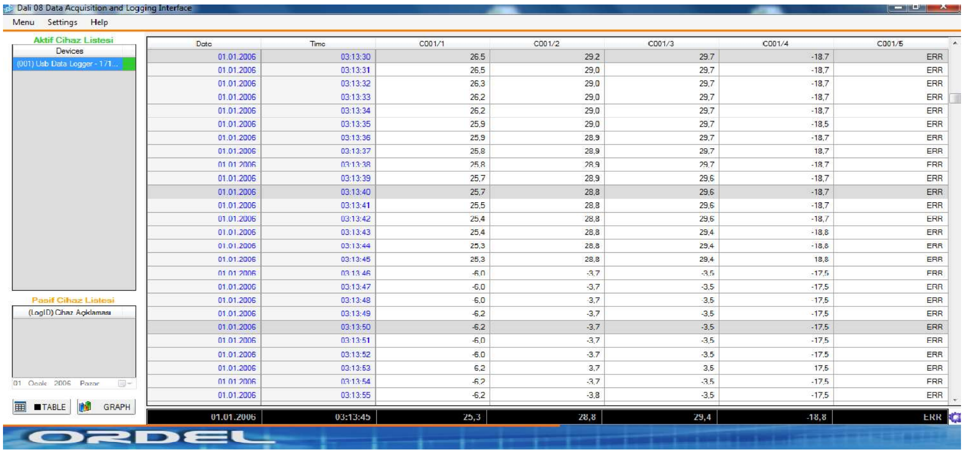
Figure 3.13: Glass of test

3.6Cooling Curve

Considering the cooling curve analysis, -20^{O}C was set on the digital control unit and allowed so as to bring down the temperature of the cooling bath to the required level. While waiting for the cooling bath to attain the desired temperature, 45ml of the sample was measured into the test jaw and the kinematic viscosity was measured at 0, 5, 10, 20, 30, 40°C and density at 0, 5, 10, 15°C was measured, this was done to ascertain the cooling effects on the biodiesel sample. In measuring the density for this, a smaller pycnometer of 25ml by volume was used.

After some hours when the cooling bath have attain the required temperature of -17°C, the number of thermocouple utilized in the measurement of the temperature of the biodiesel sample in the test jar summed up to threeT-type thermocouples as shown in Figure 3.19. The thermocouplesT_1, T_2 and T_3were placed at an equivalent distance from each other at the midpoint of the sample in the test jar.

However, before the collection of the data the 45ml of the biodiesel sample was heated to about 67°C keeping the temperature of the cooling bath constant at -17°C, the entire assembly of the heated biodiesel sample in the test jaw was then inserted into the cylinder jacket and kept in the cooling bath. From the software user interface (UI) the data logger displayed the temperature of the thermocouples. the computer was used in storing the various reading at an interval of one second using the data logger as shown below in Figure 3.16.



Date	Time	C001/1	C001/2	C001/3	C001/4	C001/5
01.01.2006	03:13:30	26,5	29.2	29.7	-18.7	ERR
01.01.2006	03:13:31	26,5	29,0	29,7	-18,7	ERR
01.01.2006	03:13:32	26,3	29,0	29,7	-18,7	ERR
01.01.2006	03:13:33	26,2	29,0	29,7	-18,7	ERR
01.01.2006	03:13:34	26,2	29,0	29,7	-18,7	ERR
01.01.2006	03:13:35	25,9	29,0	29,7	-18,5	ERR
01.01.2006	03:13:36	25,9	28,9	29,7	-18,7	ERR
01.01.2006	03:13:37	25,8	28,9	29,7	18,7	ERR
01.01.2006	03:13:38	25,8	28,9	29,7	-18,7	FRR
01.01.2006	03:13:39	25,7	28,9	29,6	-18,7	ERR
01.01.2006	03:13:40	25,7	28,8	29,6	-18,7	ERR
01.01.2006	03:13:41	25,5	28,8	29,6	-18,7	ERR
01.01.2006	03:13:42	25,4	28,8	29,6	-18,7	ERR
01.01.2006	03:13:43	25,4	28,8	29,4	-18,8	ERR
01.01.2006	03:13:44	25,3	28,8	29,4	-18,8	ERR
01.01.2006	03:13:45	25,3	28,8	29,4	18,8	ERR
01.01.2006	03:13:46	-6,0	-3,7	-3,5	-17,5	FRR
01.01.2006	03:13:47	-6,0	-3,7	-3,5	-17,5	ERR
01.01.2006	03:13:48	-6,0	-3,7	-3,5	-17,5	ERR
01.01.2006	03:13:49	-6,2	-3,7	-3,5	-17,5	ERR
01.01.2006	03:13:50	-6,2	-3,7	-3,5	-17,5	ERR
01.01.2006	03:13:51	-6,0	-3,7	-3,5	-17,5	ERR
01.01.2006	03:13:52	-6.0	-3.7	-3.5	-17.5	ERR
01.01.2006	03:13:53	6,2	3,7	3,5	17,5	ERR
01.01.2006	03:13:54	-6,2	-3,7	-3,5	-17,5	FRR
01.01.2006	03:13:55	-6,2	-3,8	-3,5	-17,5	ERR
01.01.2006	03:13:45	25,3	28,8	29,4	-18,8	ERR

Figure 3.14: Data logger reading

Thesample was allowed in the cooling bath of -17°C for four hours, the setup was allowed without interruption and the data collected was taken for analysis using the Newtonian thermal analysis theorem. This analysis could be achieved using MATLAB or MICROSOFT EXCEL software.

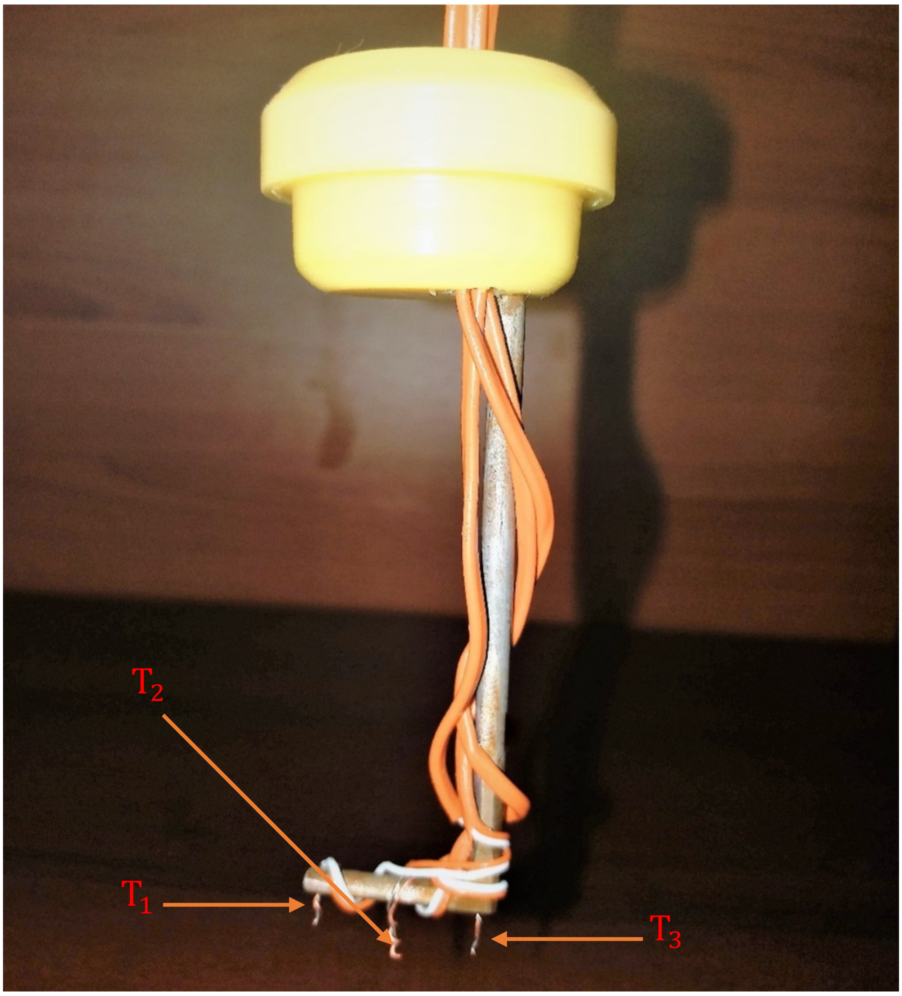


Figure 3.15: Thermocouple labelling for cooling curve

CHAPTER 4
RESULTS AND DISCUSSIONS

4.1 Kinematic viscosity of Biodiesel-Fuel Blends

The results of kinematic viscosity for the studied mixtures in this work are shown in Figures 4.1 and 4.2. The experimental data (kinematic viscosity data)of biodiesel blends are compared with (ASTM D455-69 1995.)values. It is found that all experimental data of Eurodiesel-biodiesel blends are within the range of MATM D 455 (1.9 - 6 mm^2/s at 40°C) as shown in Figure 4.1. Moreover, It is noticed that kinematic viscosity of Benzene 95 with biodiesel blends content higher than 60 vol% fulfill biodiesel standard requirement. In addition, it is noticed that the kinematic visocsity increases with the increase of volume fraction of biosdiesel.

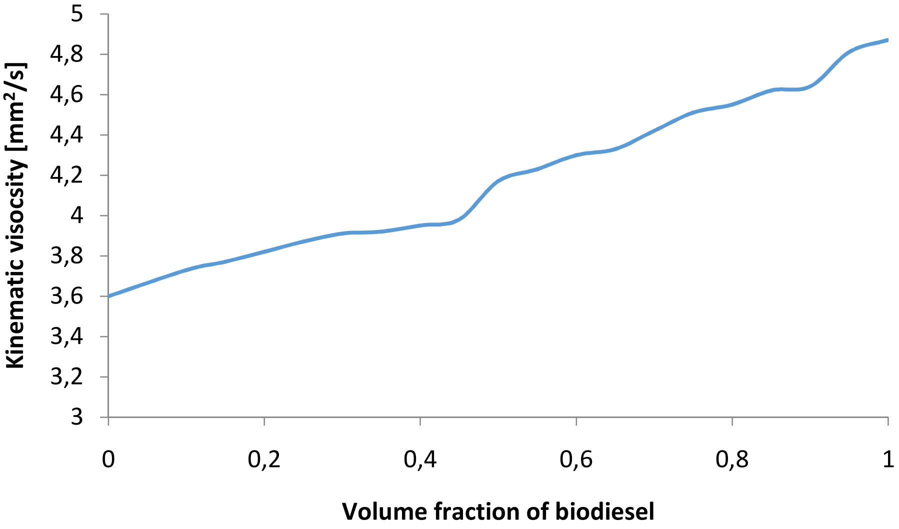


Figure 4.1: Measured kinematic viscosity values of Euro diesel-biodiesel blends at 40°C

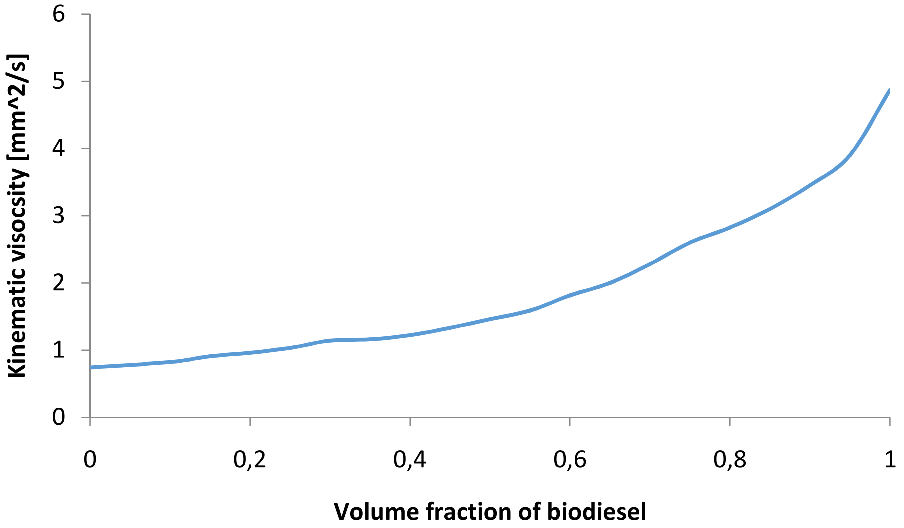


Figure 4.2: Measured kinematic viscosity values of Benzene 95-biodiesel blends at 40°C

4.2 Density of Biodiesel-Fuel Blends

Figures 4.3 and 4.4 show the density values at 15°C for different biodiesel blends with two different fuels at different percentage of volume factions of biodiesel. It is observed that if the experimental results of biodiesel fuel blends with Euro diesel compared with specification diesel fuel standard range between 800 and 880 kg/m^3 overall density results meet the minimum and maximum standard or specification requirements of diesel fuel. Moreover, it is noticed that when volume fraction of Benzene 95 increased in the mixture of blend, the density of the mixture decreases. . Moreover, the density of Benzene 95-Biodiesel with biodiesel content higher than 70 vol% fulfill biodiesel standard requirements.

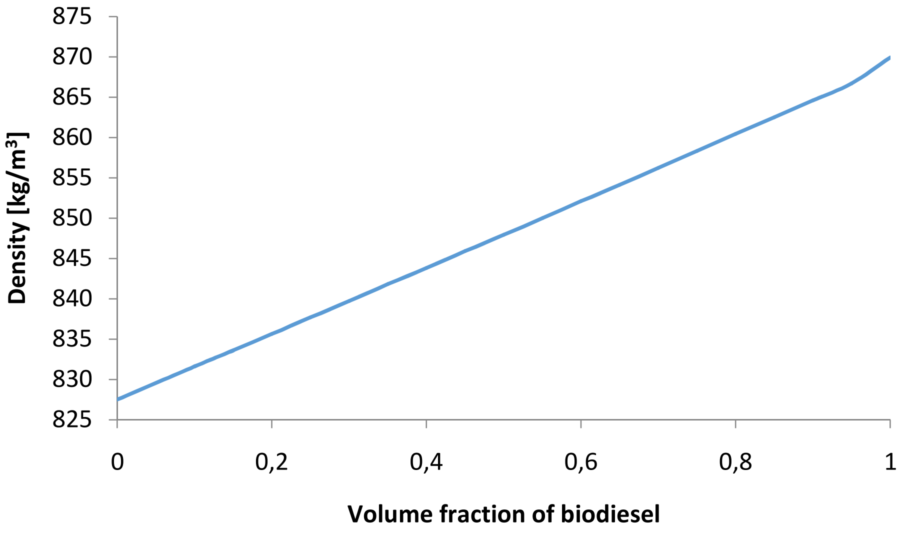


Figure 4.3: Measured density values of Euro diesel-biodiesel blends at 15^{o}C

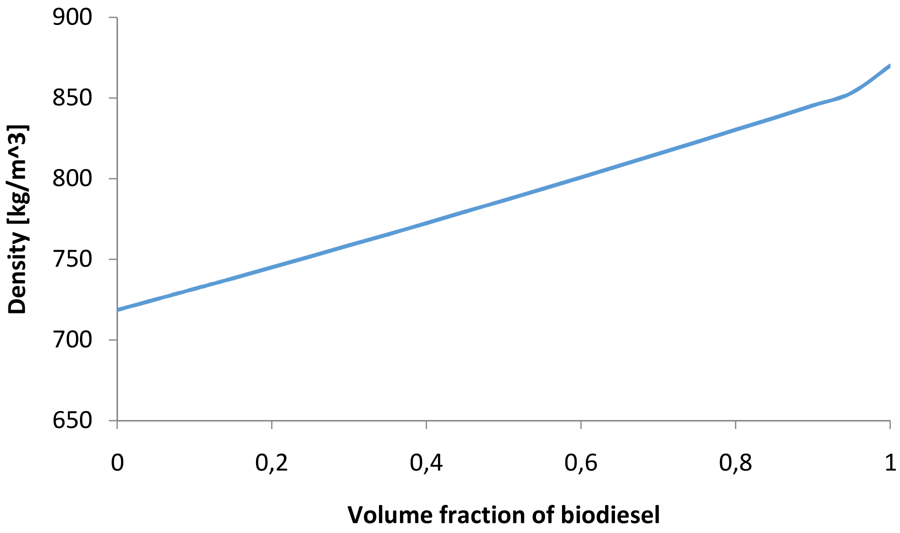


Figure 4.4: Measured density values of Benzene 95-biodiesel blends at 15^{o}C

4.3 Cold flow Properties of Biodiesel-Fuel Blends

Based on the results of kinematic viscosity and density of biodiesel-fuel blends, the cold flow properties in terms of CP (Cloud Point) and PP (Pour Point) of blends were measured manually for some selected specific volume as shown in Table 4.1. It is observed that CP and PP get regularly increase as increasing volume fraction of biodiesel. Also, it is noticed that addition of Benzene 95 can reduce the cold flow properties of biodiesel.

Table 4.1: Cloud Point and Pour Point of biodiesel blends

	Biodiesel-Euro diesel blends		
No:	**Volume fraction of blend**	**CP**	**PP**
1	20% Biodiesel + 80% Euro diesel	-1.675	-3.159
2	50% Biodiesel +50% Euro diesel	2.66	-1.34
3	80% Biodiesel +20% Euro diesel	7.7	3.08
4	90% Biodiesel +10% Euro diesel	9.9	4.84
5	100% Biodiesel + 0% Euro diesel	11	7
	Biodiesel-Benzene 95 blends		
No:	**Volume fraction of blend**	**CP**	**PP**
1	80% Biodiesel +20% Benzene 95	2	-4.50
2	85% Biodiesel +15% Benzene 95	2.75	-2.45
3	90% Biodiesel +10% Benzene 95	3.5	0
4	95% Biodiesel +5% Benzene 95	7	4.65
5	100% Biodiesel +0% Benzene 95	11	7

4.4 Thermal analysis of Biodiesel-Euro Diesel Blends

The cooling curve of biodiesel-Euro diesel blends , T vs t, and dT/dt vs t curves were plotted and are shown in Figures 4.1-4.5. The cooling bath temperature is also shown in the same figures. The average cooling bath temperature was fixed at −20°C with a minimum of −17 °C and a maximum of −19.5 °C.It is observed that, the value of CP and PP from cooling curve and observation data (Table 4.1) are almost equals.

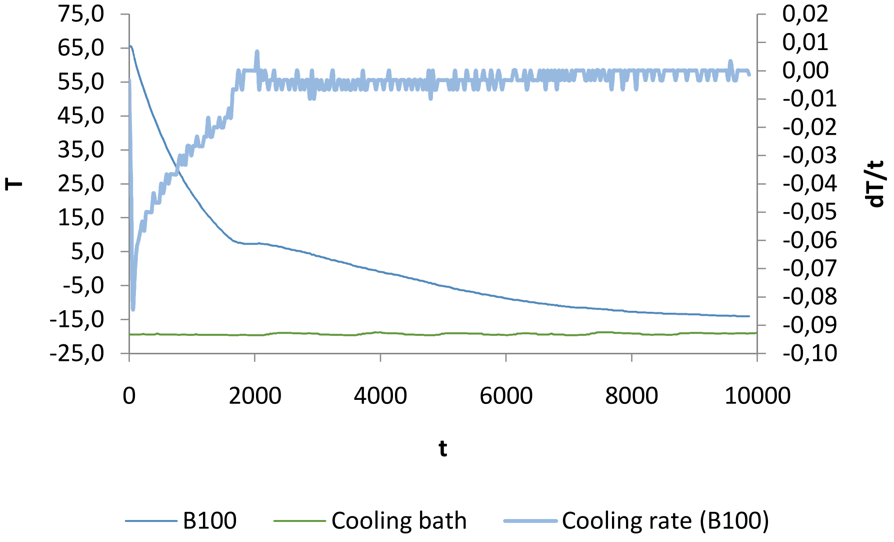


Figure 4.5: Cooling curve analysis of B100

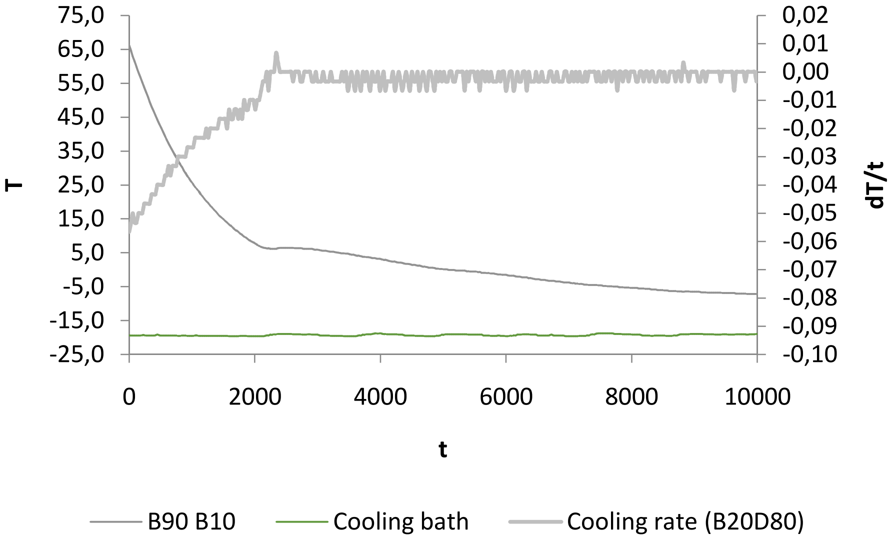


Figure 4.6: Cooling curve analysis of 90B10

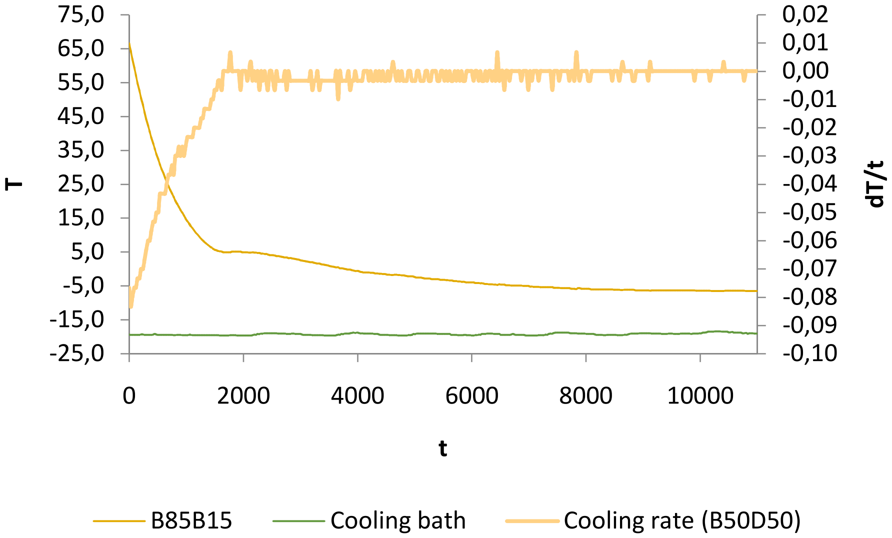


Figure 4.7: Cooling curve analysis of B85B15

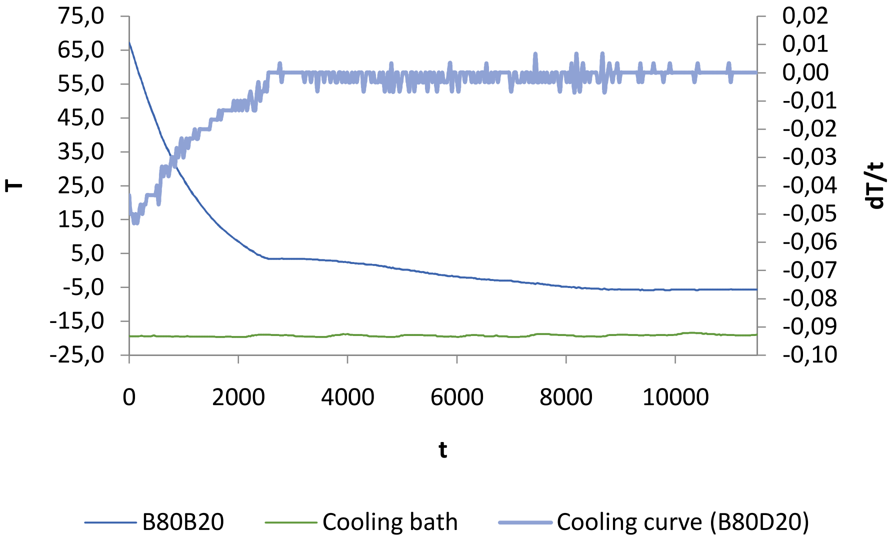


Figure 4.8: Cooling curve analysis of B80B20

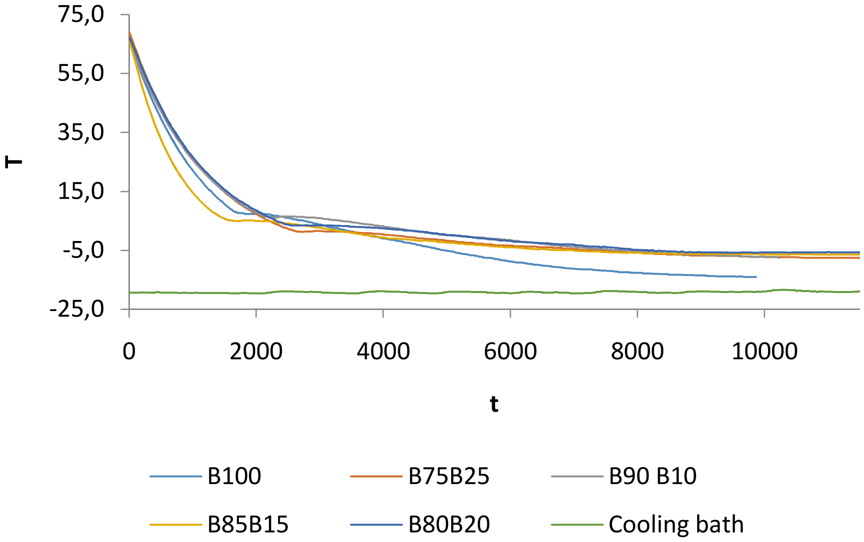


Figure 4.9: Cooling curve analysis of all biodiesel-Euro diesel blends

4.5 Thermal analysis of Biodiesel-Benzene 95 Blends

The cooling curve of biodiesel-Benzene 95 blends , T vs t, and dT/dt vs t curves were plotted and are shown in Figures 4.6-4.9. The cooling bath temperature is also shown in the same figures. The average cooling bath temperature was fixed at −20°C with a minimum of −17 °C and a maximum of −19.5 °C.It is observed that, the value of CP and PP from cooling curve and observation data (Table 4.1) are almost equals.

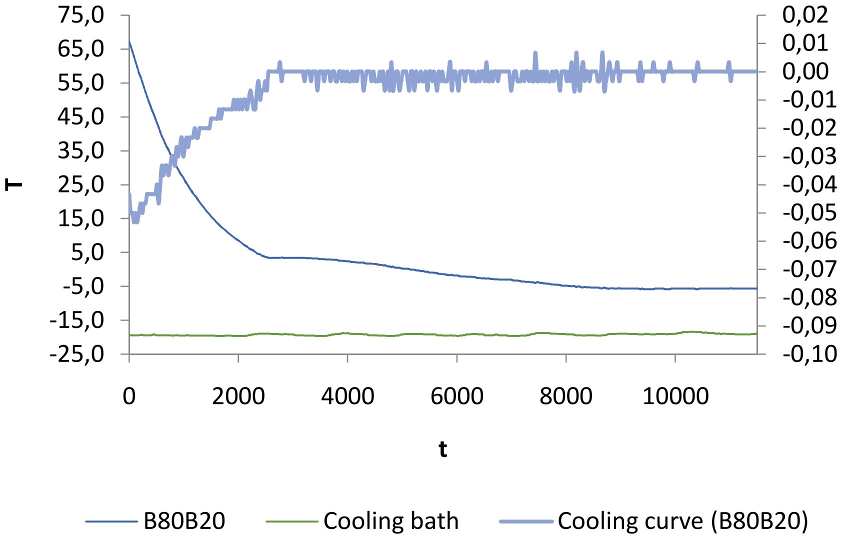


Figure 4.10: Cooling curve analysis of B80B20

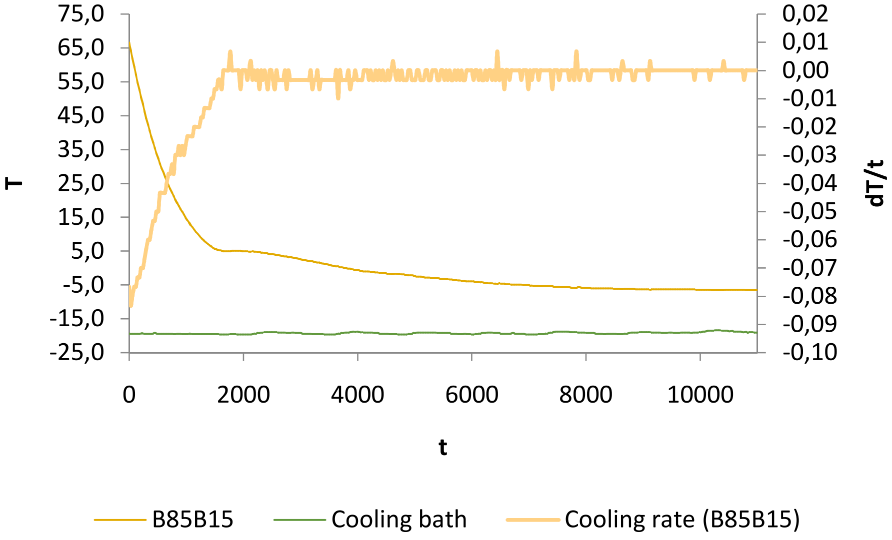


Figure 4.11: Cooling curve analysis of B85B15

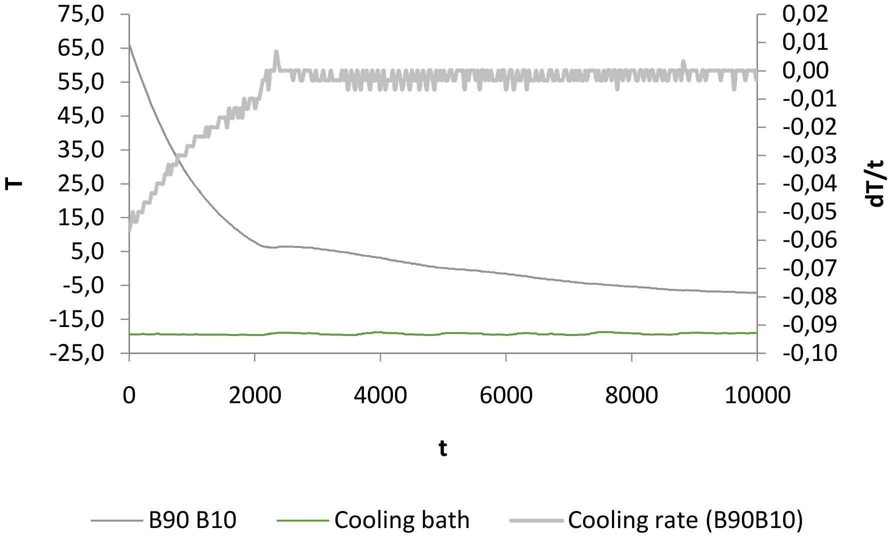


Figure 4.12: Cooling curve analysis of B90B10

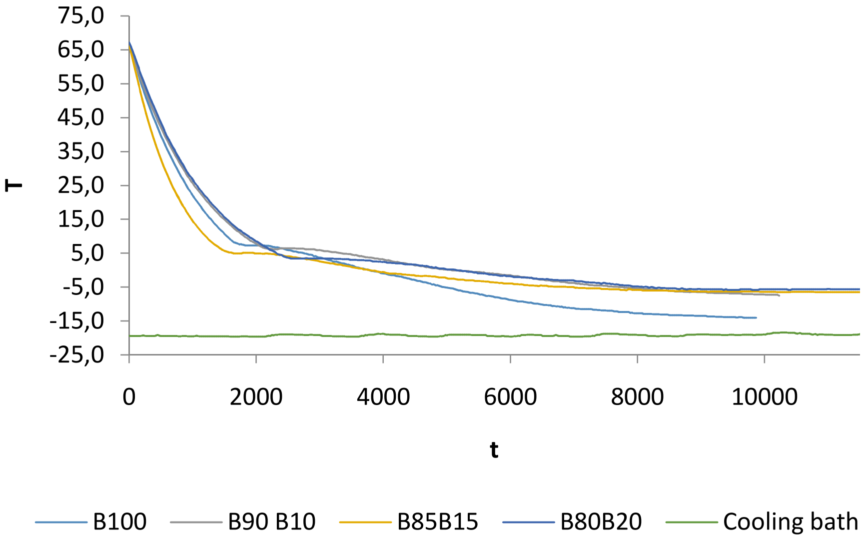


Figure 4.13: Cooling curve analysis of all biodiesel-Benzene blends

CHAPTER 5
CONCLUSIONS AND RECOMMENDATION

Biodiesel is increasingly becoming an alternative fuel for diesel engines because biodiesel use reduces the consumption of petroleum; thus, engine gas emissions are environmentally safer, also ,biodiesel is a renewable fuel from organic remain such as waste sunflower oil and used cooking oil.

This research aimed to critically to examine the cold flow behavior of biodiesel blend and measure the biodiesel properties including cold flow properties in terms of Cloud point (CP), Cold filter plugging point (CFPP) and Pour point (PP). Moreover, in this study, examine the cold flow behavior of biodiesel blends during solidification by employing the computer-aided cooling curve analysis (CA-CCA) technique.

5.1 Conclusion

The effect of thermal analysis on the waste sunflower biodiesel properties was experimentally determined. The cold flow properties were determined for each blend. The cold flow properties were determined according to (ASTM D2500, 2014), (ASTM D6371, 2005) and (ASTM D97-2005, 2005) for cloud point pour point and cold filter plugging point respectively. The cloud point (CP), the cold filter plugging point (CFPP) and the Pour point (PP) were noted as the slope changes on the cooling curve.

The researcher concluded that:

- All experimental data of Eurodiesel-biodiesel blends are within the range of MATM D 455 (1.9 - 6 mm^2/s at 40C°), moreover, It is noticed that kinematic viscosity of Benzene 95 with biodiesel blends content higher than 60 vol% fulfill biodiesel standard requirement and the kinematic visocsity increases with the increase of volume fraction of biosdiesel.
- Regarding the density of biodiesel-fuel blends, It is observed that if the experimental results of biodiesel fuel blends with Euro diesel compared with

specification diesel fuel standard range between 800 and 880 kg/m^3 overall density results meet the minimum and maximum standard or specification requirements of diesel fuel. Moreover, it is noticed that when volume fraction of Benzene 95 increased in the mixture of blend, the density of the mixture decreases.

- The research has also shown that cloud point (CP) and Pour point (PP) get regularly increase as increasing volume fraction of biodiesel. Also, it is noticed that addition of Benzene 95 can reduce the cold flow properties of biodiesel.
- The investigation of thermal analysis of biodiesel-euro diesel blends has shown that the average cooling bath temperature was fixed at −20°C with a minimum of −17 °C and a maximum of −19.5 °C.

5.2 Recommendations

The success of the work has led to more questions than not. The researcher deemed it necessary to recommend the following

- To store the same samples in a constant temperature of 10 C° to study how it will perform in regions with similar conditions.
- To study further mixtures of the blend, up to B50 K50 at the same conditions.
- This is because of the successful use of kerosene as a blending additive.
- To study the Fourier Thermal Analysis for the same samples.

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APPENDICES

APPENDIX 1

ASTM D2500

An American National Standard
British Standard 4458

Designation: D 2500 – 09

Designation: 219/82

Standard Test Method for Cloud Point of Petroleum Products[1]

This standard is issued under the fixed designation D 2500; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This test method covers only petroleum products and biodiesel fuels that are transparent in layers 40 mm in thickness, and with a cloud point below 49°C.

NOTE 1—The interlaboratory program consisted of petroleum products of Test Method D 1500 color of 3.5 and lower. The precisions stated in this test method may not apply to samples with ASTM color higher than 3.5.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D 1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)

E 1 Specification for ASTM Liquid-in-Glass Thermometers

2.2 *Energy Institute Standard:*[3]

Specifications for IP Standard Thermometers

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *biodiesel, n*—a fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats, designated B100.

3.1.1.1 *Discussion*—Biodiesel is typically produced by a reaction of vegetable oil or animal fat with an alcohol such as methanol or ethanol in the presence of a catalyst to yield mono-esters and glycerin. The fuel typically may contain up to 14 different types of fatty acids that are chemically transformed into fatty acid methyl esters (FAME).

3.1.2 *biodiesel blend, n*—a blend of biodiesel fuel with petroleum-based diesel fuel designated BXX, where XX is the volume % of biodiesel.

3.1.3 *cloud point, n—in petroleum products and biodiesel fuels,* the temperature of a liquid specimen when the smallest observable cluster of hydrocarbon crystals first occurs upon cooling under prescribed conditions.

3.1.3.1 *Discussion*—To many observers, the cluster of wax crystals looks like a patch of whitish or milky cloud, hence the name of the test method. The cloud appears when the temperature of the specimen is low enough to cause wax crystals to form. For many specimens, the crystals first form at the lower circumferential wall of the test jar where the temperature is lowest. The size and position of the cloud or cluster at the cloud point varies depending on the nature of the specimen. Some samples will form large, easily observable, clusters, while others are barely perceptible.

3.1.3.2 *Discussion*—Upon cooling to temperatures lower than the cloud point, clusters of crystals will grow in multiple directions; for example, around the lower circumference of the test jar, towards the center of the jar, or vertically upwards. The crystals can develop into a ring of cloud along the bottom circumference, followed by extensive crystallization across the bottom of the test jar as temperature decreases. Nevertheless, the cloud point is defined as the temperature at which the crystals first appear, not when an entire ring or full layer of wax has been formed at the bottom of the test jar.

3.1.3.3 *Discussion*—In general, it is easier to detect the cloud point of samples with large clusters that form quickly, such as paraffinic samples. The contrast between the opacity of the cluster and the liquid is also sharper. In addition, small brightly-reflective spots can sometimes be observed inside the cluster when the specimen is well illuminated. For other more difficult samples, such as naphthenic, hydrocracked, and those samples whose cold flow behavior have been chemically

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved April 15, 2009. Published April 2009. Originally approved in 1966. Last previous edition approved in 2005 as D 2500–05.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., http://www.energyinst.org.uk.

***A Summary of Changes section appears at the end of this standard.**

altered, the appearance of the first cloud can be less distinct. The rate of crystal growth is slow, the opacity contrast is weak, and the boundary of the cluster is more diffuse. As the temperature of these specimens decrease below the cloud point, the diffuse cluster will increase in size and can form a general haze throughout. A slight haze throughout the entire sample, which slowly becomes more apparent as the temperature of the specimen decreases, can also be caused by traces of water in the specimen instead of crystal formation (see Note 3). With these difficult samples, drying the sample prior to testing can eliminate this type of interference.

3.1.3.4 *Discussion*—The purpose of the cloud point method is to detect the presence of the wax crystals in the specimen; however trace amounts of water and inorganic compounds may also be present. The intent of the cloud point method is to capture the temperature at which the liquids in the specimen begin to change from a single liquid phase to a two-phase system containing solid and liquid. It is not the intent of this test method to monitor the phase transition of the trace components, such as water.

4. Summary of Test Method

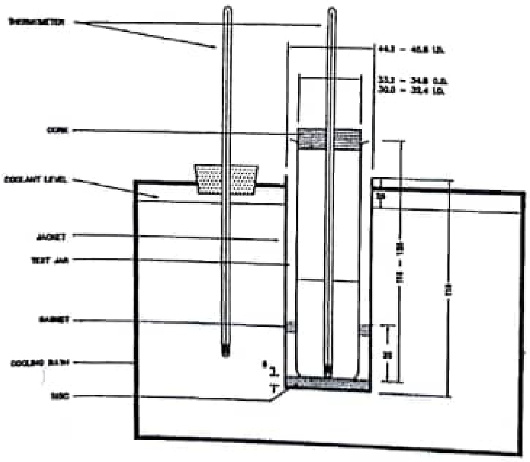
4.1 The specimen is cooled at a specified rate and examined periodically. The temperature at which a cloud is first observed at the bottom of the test jar is recorded as the cloud point.

5. Significance and Use

5.1 For petroleum products and biodiesel fuels, cloud point of a petroleum product is an index of the lowest temperature of their utility for certain applications.

6. Apparatus (see Fig. 1)

6.1 *Test Jar*, clear, cylindrical glass, flat bottom, 33.2 to 34.8-mm outside diameter and 115 and 125-mm height. The inside diameter of the jar may range from 30 to 32.4 mm within the constraint that the wall thickness be no greater than 1.6 mm. The jar should be marked with a line to indicate sample height 54 ± 3 mm above the inside bottom.



NOTE—All dimensions are in millimetres.

FIG. 1 Apparatus for Cloud Point Test

6.2 *Thermometers*, having ranges shown below and conforming to the requirements as prescribed in Specification E 1 or Specifications for IP Standard Thermometers.

Thermometer	Temperature Range	Thermometer Number ASTM	 IP
High cloud and pour	−38 to +50°C	5C	1C
Low cloud and pour	−80 to +20°C	6C	2C

6.3 *Cork*, to fit the test jar, bored centrally for the test thermometer.

6.4 *Jacket*, metal or glass, watertight, cylindrical, flat bottom, about 115 mm in depth, with an inside diameter of 44.2 to 45.8 mm. It shall be supported free of excessive vibration and firmly in a vertical position in the cooling bath of 6.7 so that not more than 25 mm projects out of the cooling medium.

6.5 *Disk*, cork or felt, 6-mm thick to fit loosely inside the jacket.

6.6 *Gasket*, ring form, about 5 mm in thickness, to fit snugly around the outside of the test jar and loosely inside the jacket. The gasket may be made of rubber, leather, or other material that is elastic enough to cling to the test jar and hard enough to hold its shape. Its purpose is to prevent the test jar from touching the jacket.

6.7 *Bath or Baths*, maintained at prescribed temperatures with a firm support to hold the jacket vertical. The required bath temperatures may be maintained by refrigeration if available, otherwise by suitable cooling mixtures. Cooling mixtures commonly used for bath temperatures shown are in Table 1.

7. Reagents and Materials

7.1 *Acetone*—Technical grade acetone is suitable for the cooling bath, provided it does not leave a residue on drying. (**Warning**—Extremely flammable.)

7.2 *Carbon Dioxide (Solid) or Dry Ice*—A commercial grade of dry ice is suitable for use in the cooling bath.

7.3 *Petroleum Naphtha*—A commercial or technical grade of petroleum naphtha is suitable for the cooling bath. (**Warning**—Combustible. Vapor harmful.)

7.4 *Sodium Chloride Crystals*—Commercial or technical grade sodium chloride is suitable.

7.5 *Sodium Sulfate*—A reagent grade of anhydrous sodium sulfate should be used when required (see Note 3).

TABLE 1 Cooling Mixtures and Bath Temperatures

	Bath Temperature
Ice and water	0 ± 1.5°C
Crushed ice and sodium chloride crystals, or acetone or petroleum naptha (see 7) with solid carbon dioxide added to give the desired temperature	−18 ± 1.5°C
Acetone or petroleum naptha (see 7) with solid carbon dioxide added to give the desired temperature	−33 ± 1.5°C
Acetone or petroleum naptha (see 7) with solid carbon dioxide added to give the desired temperature	−51 ± 1.5°C
Acetone or petroleum naptha (see 7) with solid carbon dioxide added to give the desired temperature	−69 ± 1. 5°C

8. Procedure

8.1 Bring the sample to be tested to a temperature at least 14°C above the expected cloud point. Remove any moisture present by a method such as filtration through dry lintless filter paper until the oil is perfectly clear, but make such filtration at a temperature of at least 14°C above the approximate cloud point.

8.2 Pour the sample into the test jar to the level mark.

8.3 Close the test jar tightly by the cork carrying the test thermometer. Use the high cloud and pour thermometer if the expected cloud point is above −36°C and the low cloud and pour thermometer if the expected cloud point is below −36°C. Adjust the position of the cork and the thermometer so that the cork fits tightly, the thermometer and the jar are coaxial, and the thermometer bulb is resting on the bottom of the jar.

NOTE 2—Liquid column separation of thermometers occasionally occurs and may escape detection. Thermometers should be checked periodically and used only if their ice points are 0 ± 1°C, when the thermometer is immersed to the immersion line in an ice bath, and when the emergent column temperature does not differ significantly from 21°C. Alternatively, immerse the thermometer to a reading and correct for the resultant cooler stem temperature.

8.4 See that the disk, gasket, and the inside of the jacket are clean and dry. Place the disk in the bottom of the jacket. The disk and jacket shall have been placed in the cooling medium a minimum of 10 min before the test jar is inserted. The use of a jacket cover while the empty jacket is cooling is permitted. Place the gasket around the test jar, 25 mm from the bottom. Insert the test jar in the jacket. Never place a jar directly into the cooling medium.

NOTE 3—Failure to keep the disk, gasket, and the inside of the jacket clean and dry may lead to frost formation, which may cause erroneous results.

8.5 Maintain the temperature of the cooling bath at 0 ± 1.5°C.

8.6 At each test thermometer reading that is a multiple of 1°C, remove the test jar from the jacket quickly but without disturbing the specimen, inspect for cloud, and replace in the jacket. This complete operation shall require not more than 3 s. If the oil does not show a cloud when it has been cooled to 9°C, transfer the test jar to a jacket in a second bath maintained at a temperature of −18 ± 1.5°C (see Table 2). Do not transfer the jacket. If the specimen does not show a cloud when it has been cooled to −6°C, transfer the test jar to a jacket in a third bath maintained at a temperature of −33± 1.5°C. For the determination of very low cloud points, additional baths are required, each bath to be maintained in accordance with Table 2. In each case, transfer the jar to the next bath, if the specimen does not exhibit cloud point and the temperature of the specimen reaches the lowest specimen temperature in the range identified for the current bath in use, based on the ranges stated in Table 2.

TABLE 2 Bath and Sample Temperature Ranges

Bath	Bath Temperature Setting, °C	Sample Temperature Range, °C
1	0 ± 1.5	Start to 9
2	−18 ± 1.5	9 to −6
3	−33 ± 1.5	−6 to −24
4	−51 ± 1.5	−24 to −42
5	−69 ± 1.5	−42 to −60

8.7 Report the cloud point, to the nearest 1°C, at which any cloud is observed at the bottom of the test jar, which is confirmed by continued cooling.

NOTE 4—A wax cloud or haze is always noted first at the bottom of the test jar where the temperature is lowest. A slight haze throughout the entire sample, which slowly becomes more apparent as the temperature is lowered, is usually due to traces of water in the oil. Generally this water haze will not interfere with the determination of the wax cloud point. In most cases of interference, filtration through dry lintless filter papers, such as described in 8.1, is sufficient. In the case of diesel fuels, however, if the haze is very dense, a fresh portion of the sample should be dried by shaking 100 mL with 5 g of anhydrous sodium sulfate for at least 5 min and then filtering through dry lintless filter paper. Given sufficient contact time, this procedure will remove or sufficiently reduce the water haze so that the wax cloud can be readily discerned. Drying and filtering should be done always at a temperature at least 14°C above the approximate cloud point but otherwise not in excess of 49°C.

9. Report

9.1 Report the temperature recorded in 8.7 as the cloud point, Test Method D 2500.

10. Precision and Bias

10.1 The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

10.1.1 *Repeatability*—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of this test method, exceed 2°C only in 1 case in 20.

10.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of this test method, exceed 4°C only in 1 case in 20.

10.1.3 The precision statements were derived from a 1990 interlaboratory cooperative test program.[4] Participants analyzed 13 sample sets comprised of various distillate fuels and lubricating oils with temperature range from -1 to -37°C. Eight laboratories participated with the manual D 2500/IP219 test method. Information on the type of samples and their average cloud points are in the research report.

10.2 *Bias*—The procedure in this test method has no bias, because the value of cloud point can be defined only in terms of a test method.

10.3 *Precision for Biodiesel Products*[5]—The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

10.3.1 *Repeatability for Blends of Biodiesel in Diesel*—The difference between successive test results obtained by the same operator, using the same apparatus, under constant operating

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1444.

[5] Supporting data (the results of the 2001 interlaboratory cooperative test program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02–1524.

conditions, on identical test material would, in the long run, in the normal and correct operation of this test method, exceed 2°C only in 1 case in 20.

10.3.2 *Reproducibility for Blends of Biodiesel in Diesel*—The difference between two single and independent test results obtained by different operators, working in different laboratories, on identical test material would, in the long run, in the normal and correct operation of this test method, exceed 3°C only in 1 case in 20.

NOTE 5—The precision for blends of biodiesel in diesel samples comprised cloud points from about −2 to +10°C.

10.3.3 The precision statements were derived from a 2001 interlaboratory cooperative test program.[5] Participants analyzed eleven sample sets comprised of different blends of two petroleum distillate fuels, diesel and kerosene, with various biodiesel fuels with temperature range from +10 to −45°C. Ten laboratories participated with the manual D 2500/IP219 test method. Information on the type of samples and their average cloud points are in the research report.

NOTE 6—One of the outcomes of the interlaboratory study was the selection of the sample types, since the ones used in the study contributed to a difficulty in determining the precision statement. Kerosene is a sufficiently different fuel type from biodiesel to cause some slight separation of phases upon cooling when in B20 blends. Also, the particular kerosene sample used was atypical, which complicated the study further. Therefore, data from the blends of kerosene in biodiesel were not used in the precision statement. In addition, the diesel fuel used in the research report was high cloud point material. Due to the cloud point of the base diesel material, this temperature range in the precision statement was limited.

NOTE 7—A future interlaboratory cloud study will be done to include a wider range of base biodiesel fuels with various distillate blend stocks.

10.4 *Bias for Biodiesel Products*[5]—The procedure in this test method has no bias, because the value of cloud point can be defined only in terms of a test method.

11. Keywords

11.1 cloud point; petroleum products; wax crystals

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D 2500–05) that may impact the use of this standard.

(*1*) Revised 6.7.
(2) Revised 7.
(*3*) Added Table 1.
(*4*) Deleted original Note 2.

APPENDIX 2

ASTM D97-2500

Designation: D 97 – 05

An American National Standard

Designation: 15/95

Standard Test Method for Pour Point of Petroleum Products[1]

This standard is issued under the fixed designation D 97; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This test method is intended for use on any petroleum product.[2] A procedure suitable for black specimens, cylinder stock, and nondistillate fuel oil is described in 8.8. A procedure for testing the fluidity of a residual fuel oil at a specified temperature is described in Appendix X1.

1.2 Several ASTM test methods offering alternative procedures for determining pour points using automatic apparatus are available. None of them share the same designation number as Test Method D 97. When an automatic instrument is used, the ASTM test method designation number specific to the technique shall be reported with the results. A procedure for testing the pour point of crude oils is described in Test Method D 5853.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [3]

D 117 Guide for Sampling, Test Methods, and Specifications for Electrical Insulating Oils of Petroleum Origin
D 396 Specification for Fuel Oils
D 1659 Test Method for Maximum Fluidity Temperature of Residual Fuel Oil[4]
D 2500 Test Method for Cloud Point of Petroleum Products
D 3245 Test Method for Pumpability of Industrial Fuel Oils
D 5853 Test Method for Pour Point of Crude Oils
E 1 Specification for ASTM Liquid-in-Glass Thermometers

2.2 *Energy Institute Standards:*

Specifications for IP Standard Thermometers [5]

3. Terminology

3.1 *Definitions:*

3.1.1 *black oil, n*—lubricant containing asphaltic materials. Black oils are used in heavy-duty equipment applications, such as mining and quarrying, where extra adhesiveness is desired.

3.1.2 *cylinder stock, n*—lubricant for independently lubricated engine cylinders, such as those of steam engines and air compressors. Cylinder stock are also used for lubrication of valves and other elements in the cylinder area.

3.1.3 *pour point, n—in petroleum products,* the lowest temperature at which movement of the test specimen is observed under prescribed conditions of test.

3.1.4 *residual fuel, n*—a liquid fuel containing bottoms remaining from crude distillation or thermal cracking; sometimes referred to as heavy fuel oil.

3.1.4.1 *Discussion*—Residual fuels comprise Grades 4, 5, and 6 fuel oils, as defined in Specification D 396.

4. Summary of Test Method

4.1 After preliminary heating, the sample is cooled at a specified rate and examined at intervals of 3°C for flow characteristics. The lowest temperature at which movement of the specimen is observed is recorded as the pour point.

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved June 1, 2005. Published July 2005. Originally approved in 1927, replacing D 47. Last previous edition approved in 2004 as D 97–04.

In the IP, this test method is under the jurisdiction of the Standardization Committee. This test method was adopted as a joint ASTM-IP Standard in 1965.

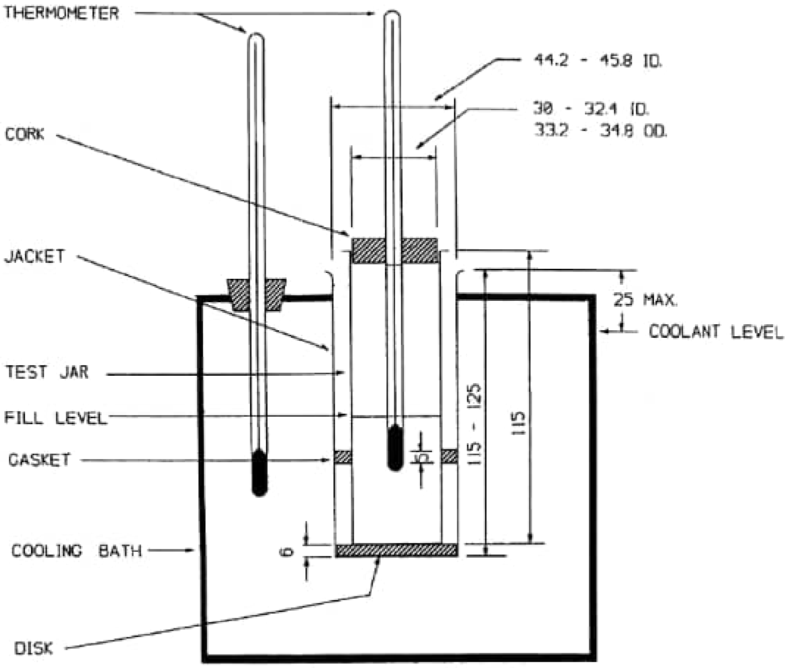
[2] Statements defining this test and its significance when applied to electrical insulating oils of mineral origin will be found in Guide D 117.

[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[4] Withdrawn.

[5] Methods for Analysis and Testing, *IP Standards for Petroleum and its Products*, Part I, Vol 2.

***A Summary of Changes section appears at the end of this standard.**



NOTE—Dimensions are in millimetres (not to scale).
FIG. 1 Apparatus for Pour Point Test

5. Significance and Use

5.1 The pour point of a petroleum specimen is an index of the lowest temperature of its utility for certain applications.

6. Apparatus

6.1 *Test Jar*, cylindrical, of clear glass, flat bottom, 33.2 to 34.8-mm outside diameter, and 115 to 125 mm in height. The inside diameter of the jar can range from 30.0 to 32.4 mm, within the constraint that the wall thickness be no greater than 1.6 mm. The jar shall have a line to indicate a sample height 54 ± 3 mm above the inside bottom. See Fig. 1.

6.2 *Thermometers*, having the following ranges and conforming to the requirements prescribed in Specification E 1 for thermometers:

	Temperature	Thermometer Number	
Thermometer	Range	ASTM	IP
High cloud and pour	−38 to +50°C	5C	1C
Low cloud and pour	−80 to +20°C	6C	2C
Melting point	+32 to +127°C	61C	63C

6.2.1 Since separation of liquid column thermometers occasionally occurs and may escape detection, thermometers should be checked immediately prior to the test and used only if they prove accurate within ±1°C (for example ice point).

6.3 *Cork*, to fit the test jar, bored centrally for the test thermometer.

6.4 *Jacket*, watertight, cylindrical, metal, flat-bottomed, 115 ± 3-mm depth, with inside diameter of 44.2 to 45.8 mm. It shall be supported in a vertical position in the cooling bath (see 6.7) so that not more than 25 mm projects out of the cooling medium, and shall be capable of being cleaned.

6.5 *Disk*, cork or felt, 6 mm thick to fit loosely inside the jacket.

6.6 *Gasket*, to fit snugly around the outside of the test jar and loosely inside the jacket. The gasket may be made of rubber, leather, or other material that is elastic enough to cling to the test jar and hard enough to hold its shape. Its purpose is to prevent the test jar from touching the jacket.

6.7 *Bath or Baths*, maintained at prescribed temperatures with a firm support to hold the jacket vertical. The required bath temperatures may be obtained by refrigeration if available, otherwise by suitable freezing mixtures. Freezing mixtures commonly used for temperatures down to those shown are as follows:

	For Temperatures Down
Ice and water	9°C
Crushed ice and sodium chloride crystals	−12°C
Crushed ice and calcium chloride crystals	−27°C
Acetone or petroleum naphtha (see Section 6) chilled in a covered metal beaker with an ice-salt mixture to −12°C then with enough solid carbon dioxide to give the desired temperature.	−57°C

7. Reagents and Materials

7.1 The following solvents of technical grade are appropriate for low-temperature bath media.

7.1.1 *Acetone*, (**Warning**—Extremely flammable).

7.1.2 *Alcohol, Ethanol* (**Warning**—Flammable).

7.1.3 *Alcohol, Methanol* (**Warning**—Flammable. Vapor harmful).

7.1.4 *Petroleum Naphtha*. (**Warning**—Combustible. Vapor harmful).

7.1.5 *Solid Carbon Dioxide*, (**Warning**—Extremely cold −78.5°C).

8. Procedure

8.1 Pour the specimen into the test jar to the level mark. When necessary, heat the specimen in a water bath until it is just sufficiently fluid to pour into the test jar.

NOTE 1—It is known that some materials, when heated to a temperature higher than 45°C during the preceding 24 h, do not yield the same pour point results as when they are kept at room temperature for 24 h prior to testing. Examples of materials which are known to show sensitivity to thermal history are residual fuels, black oils, and cylinder stocks.

8.1.1 Samples of residual fuels, black oils, and cylinder stocks which have been heated to a temperature higher than 45°C during the preceding 24 h, or when the thermal history of these sample types is not known, shall be kept at room temperature for 24 h before testing. Samples which are known by the operator not to be sensitive to thermal history need not be kept at room temperature for 24 h before testing.

8.1.2 Experimental evidence supporting elimination of the 24-h waiting period for some sample types is contained in a research report.[6]

8.2 Close the test jar with the cork carrying the high-pour thermometer (5.2). In the case of pour points above 36°C, use a higher range thermometer such as IP 63C or ASTM 61C. Adjust the position of the cork and thermometer so the cork fits tightly, the thermometer and the jar are coaxial, and the thermometer bulb is immersed so the beginning of the capillary is 3 mm below the surface of the specimen.

8.3 For the measurement of pour point, subject the specimen in the test jar to the following preliminary treatment:

8.3.1 *Specimens Having Pour Points Above −33°C*—Heat the specimen without stirring to 9°C above the expected pour point, but to at least 45°C, in a bath maintained at 12°C above the expected pour point, but at least 48°C. Transfer the test jar to a water bath maintained at 24°C and commence observations for pour point.

8.3.2 *Specimens Having Pour Points of −33°C and Below*—Heat the specimen without stirring to 45°C in a bath maintained at 48°C and cool to 15°C in a water bath maintained at 6°C. Remove the high cloud and pour thermometer, and place the low cloud and pour thermometer in position.

8.4 See that the disk, gasket, and the inside of the jacket are clean and dry. Place the disk in the bottom of the jacket. Place the gasket around the test jar, 25 mm from the bottom. Insert the test jar in the jacket. Never place a jar directly into the cooling medium.

8.5 After the specimen has cooled to allow the formation of paraffin wax crystals, take great care not to disturb the mass of specimen nor permit the thermometer to shift in the specimen; any disturbance of the spongy network of wax crystals will lead to low and erroneous results.

8.6 Pour points are expressed in integers that are positive or negative multiples of 3°C. Begin to examine the appearance of the specimen when the temperature of the specimen is 9°C above the expected pour point (estimated as a multiple of 3°C). At each test thermometer reading that is a multiple of 3°C below the starting temperature remove the test jar from the jacket. To remove condensed moisture that limits visibility wipe the surface with a clean cloth moistened in alcohol (ethanol or methanol). Tilt the jar just enough to ascertain whether there is a movement of the specimen in the test jar. The complete operation of removal, wiping, and replacement shall require not more than 3 s.

8.6.1 If the specimen has not ceased to flow when its temperature has reached 27°C, transfer the test jar to the next lower temperature bath in accordance with the following schedule:

Specimen is at +27°C, move to 0°C bath
Specimen is at +9°C, move to −18°C bath
Specimen is at −6°C, move to −33°C bath
Specimen is at −24°C, move to −51°C bath
Specimen is at −42°C, move to −69°C bath

8.6.2 As soon as the specimen in the jar does not flow when tilted, hold the jar in a horizontal position for 5 s, as noted by an accurate timing device and observe carefully. If the specimen shows any movement, replace the test jar immediately in the jacket and repeat a test for flow at the next temperature, 3°C lower.

8.7 Continue in this manner until a point is reached at which the specimen shows no movement when the test jar is held in a horizontal position for 5 s. Record the observed reading of the test thermometer.

8.8 For black specimen, cylinder stock, and nondistillate fuel specimen, the result obtained by the procedure described in 8.1 through 8.7 is the upper (maximum) pour point. If required, determine the lower (minimum) pour point by heating the sample while stirring, to 105°C, pouring it into the jar, and determining the pour point as described in 8.4 through 8.7.

8.9 Some specifications allow for a pass/fail test or have pour point limits at temperatures not divisible by 3°C. In these cases, it is acceptable practice to conduct the pour point measurement according to the following schedule: Begin to examine the appearance of the specimen when the temperature of the specimen is 9°C above the specification pour point. Continue observations at 3°C intervals as described in 8.6 and 8.7 until the specification temperature is reached. Report the sample as passing or failing the specification limit.

9. Calculation and Report

9.1 Add 3°C to the temperature recorded in 8.7 and report the result as the Pour Point, ASTM D 97. For black oil, and so forth, add 3°C to the temperature recorded in 8.7 and report the

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1377.

result as Upper Pour Point, ASTM D 97, or Lower Pour Point, ASTM D 97, as required.

10. Precision and Bias

10.1 *Lubricating Oil and Distillate and Residual Fuel Oil.* [7]

10.1.1 *Repeatability*—The difference between successive test results, obtained by the same operator using the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of this test method, exceed 3°C only in one case in twenty. Differences greater than this should be considered suspect.

10.1.2 *Reproducibility*—The difference between two single and independent test results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of this test method, exceed 6°C only in one case in twenty. Differences greater than this should be considered suspect.

10.2 *Bias*—There being no criteria for measuring bias in these test-product combinations, no statement of bias can be made.

10.3 The precision statements were prepared with data on ten new (unused) mineral oil-based lubricants and sixteen assorted fuel oils tested by twelve cooperators. The mineral oil-based lubricants had pour points ranging from −48 to −6°C while the fuel oils had pour points ranging from −33 to +51°C. The following precision data were obtained:

	Mineral Oil Lubricants	Fuel Oils
95 % Confidence		
Repeatability, °C	2.87	2.52
Reproducibility, °C	6.43	6.59

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[7] The cloud point procedure formerly part of this test method now appears as Test Method D 2500.

APPENDIX

(Nonmandatory Information)

X1. TEST FOR FLUIDITY OF A RESIDUAL FUEL OIL AT A SPECIFIED TEMPERATURE

X1.1 General

X1.1.1 The low-temperature flow properties of a waxy fuel oil depend on handling and storage conditions. Thus, they may not be truly indicated by pour point. The pour point test does not indicate what happens when an oil has a considerable head of pressure behind it, such as when gravitating from a storage tank or being pumped along a pipeline. Failure to flow at the pour point is normally attributed to the separation of wax from the fuel; however, it can also be due to the effect of viscosity in the case of very viscous fuel oils. In addition pour points of residual fuels are influenced by the previous thermal history of the specimens. A loosely knit wax structure built up on cooling of the oil can be normally broken by the application of relatively little pressure.

X1.1.2 The usefulness of the pour point test in relation to residual fuel oils is open to question, and the tendency to regard the pour point as the limiting temperature at which a fuel will flow can be misleading. The problem of accurately specifying the handling behavior of fuel oil is important, and because of the technical limitations of the pour point test, various pumpability tests have been devised to assess the low-temperature flow characteristics of heavy residual fuel oils. Test Method D 3245 is one such method. However, most alternative methods tend to be time-consuming and as such do not find ready acceptance as routine control tests for determining low-temperature flow properties. One method which is relatively quick and easy to perform and has found limited acceptance as a "go-no-go" method is based on the appendix method to the former Test Method D 1659–65. The method is described as follows.

X1.2 Scope

X1.2.1 This method covers the determination of the fluidity of a residual fuel oil at a specified temperature in an as-received condition.

X1.3 Definition

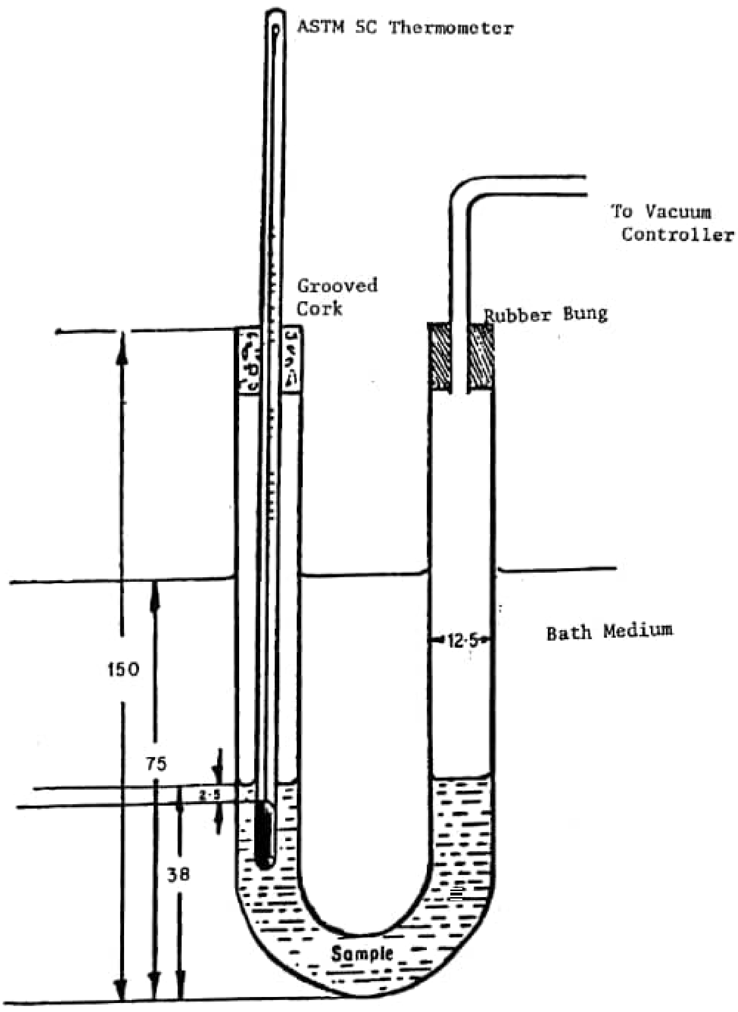
X1.3.1 *fluidity temperature*—the sample when tested in an as-received condition is considered "fluid at the temperature of the test" if it will flow 2 mm in 1 min in a 12.5 mm U-tube under a maximum pressure of 152 mm of mercury.

X1.4 Summary of Test Method

X1.4.1 A sample of fuel in its as-received condition is cooled at the specified temperature for 30 min in the standard U-tube and is tested for movement under prescribed pressure conditions.

X1.5 Significance and Use

X1.5.1 This method may be used as a "go-no-go" procedure for operational situations where it is necessary to ascertain the fluidity of a residual oil under prescribed conditions in an as-received condition. The conditions of this method simulate those of a pumping situation where the oil is expected to flow through a 12-mm pipe under slight pressure at a specified temperature. Fluidity, like Test Method D 97, is used to define cold flow properties. It differs from D 97, however, in that (*1*) it is restricted to residual fuel oil and (*2*) a prescribed pressure is applied to the sample. The latter represents an attempt to overcome the technical limitations of the Pour Point Method where gravity-induced flow is the criterion. Test Method



NOTE—All dimensions are in millimetres

FIG. X1.1 Disposition of U-tube in Fluidity Temperature Test Bath

D 3245, represents another method for predicting field performance in cold flow conditions. Test Method D 3245, however, does have limitations and may not be suitable for use with very waxy fuel oils which solidify so rapidly in the chilling bath that a reading cannot be obtained under the conditions of the test. It is also a time-consuming test and therefore not suitable for routine control testing.

X1.6 Apparatus

X1.6.1 *Glass U-Tubes*, 150 mm high, having a uniform internal diameter of 12.5 ± 1 mm and a radius of curvature, measured to the outside curve of the tube of 35 mm (Fig. X1.1).

X1.6.2 *Thermometers*—Thermometers having a range from −38 to +50°C and conforming to the requirements of Thermometer 5C as prescribed in Specification E 1, shall be used for insertion in the glass U-tubes and for measuring the temperatures of the baths.

X1.6.3 *Fluidity Temperature Test Bath*,[8] consists of a reservoir, a stirrer, and a motor and pump to circulate coolant through the coils of the tubing placed in the bottom of the test bath and passing through the cold bath. The flow of coolant through these coils can be controlled by a thermostat and a solenoid valve. It is possible that, where justified by the quantity of work, more than one such bath could be utilized to permit concurrent testing at more than one temperature (Fig. X1.2).

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[8] A kinematic viscosity bath is usually satisfactory.

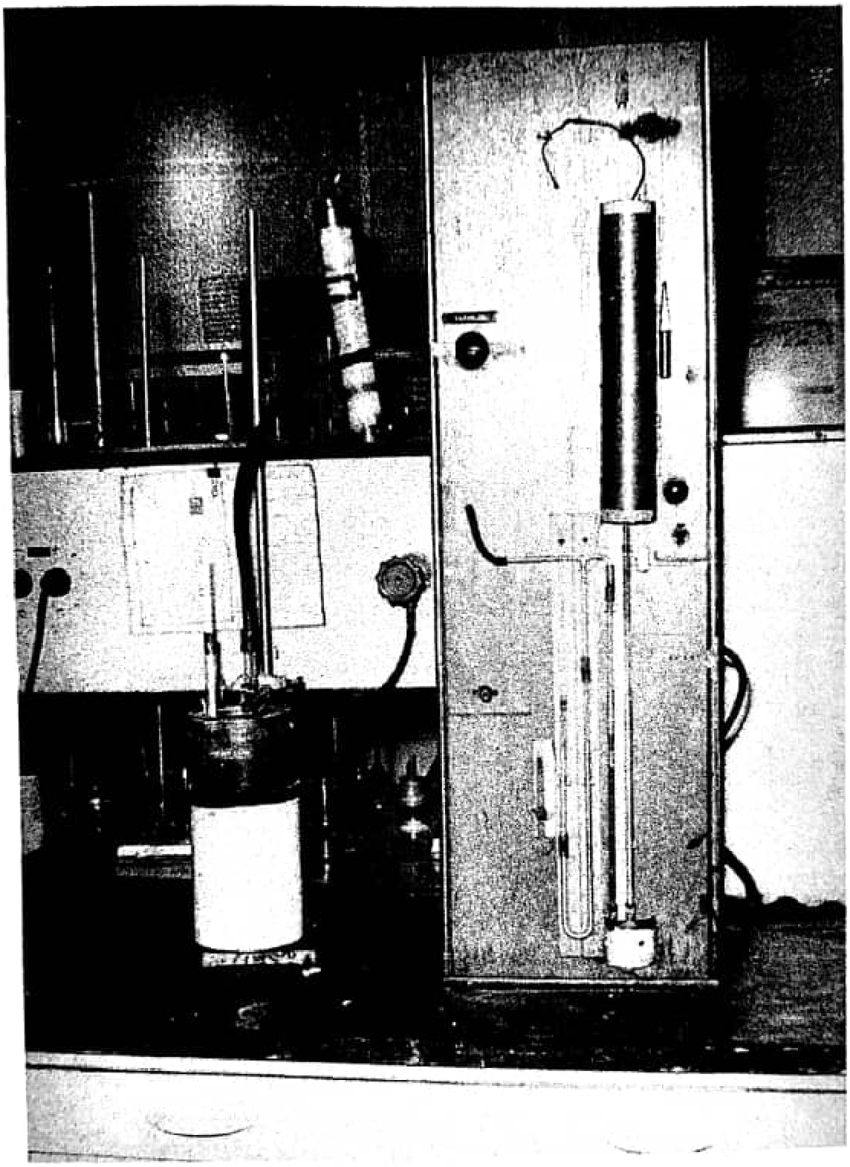

FIG. X1.2 Fluidity Temperature Apparatus

X1.6.4 *Mercury Manometer* calibrated in 10-mm divisions with a distinguishing mark at 152 mm (equivalent to 20.3 kPa).

X1.6.5 *Automatic Vacuum Controller*[9] *(as shown in Fig. X1.3 and Fig. X1.4)*—A device that gradually increased the vacuum applied to one end of the U-tube at the specified rate of 10 mm/4S.

X1.7 Preparation of Apparatus

X1.7.1 Adjust the automatic vacuum controller as follows: close the stopcock on the tube connecting the automatic vacuum controller to the fluidity tester. A pinchcock on the rubber tube will serve as well as a stopcock. Wind the thread attached to the steel rod around the pulley on the synchronous motor until the end of the rod is about 15 mm above the zero level of the mercury in the control manometer. Turn on the power switch. The thread will begin to unwind, lowering the steel rod. When the rod contacts the mercury, the relay will

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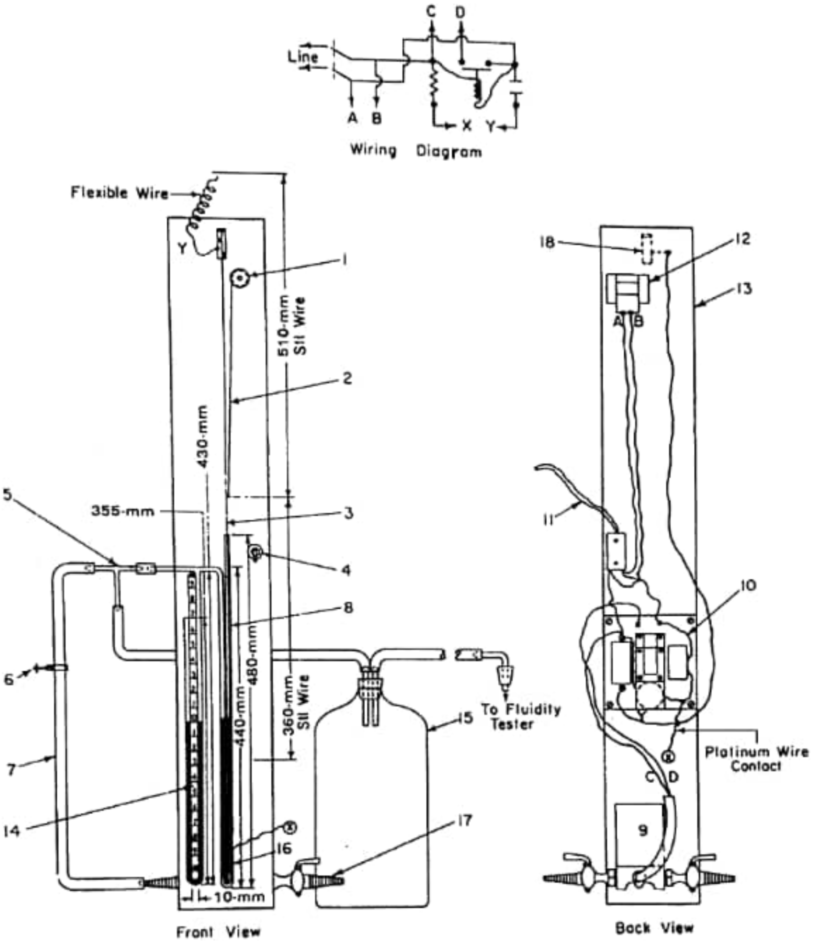
[9] This apparatus may be shop fabricated. Details of special parts are indicated in Figs. X1.3 and X1.4. Alternatively the apparatus can be purchased.



1—26-mm diameter face pulley
2—Thread
3—Steel rod
4—Switch-DPST
5—Tee, 90-mm long
6—Needle valve
7—Rubber or plastic tubing
8—6-mm heat-resistant glass tube
9—Solenoid valve
10—Electric relay
11—Electric cord to outlet
12—Synchronous motor
13—Plywood of approximately 10-mm thickness
14—Millimeter scale
15—4-L bottle
16—0.5-mm heat-resistant glass capillary
17—To vacuum line
18—Rod holder

FIG. X1.3 Assembly Automatic Vacuum Controller Apparatus

open the solenoid valve in the vacuum line and air will be pumped from the system at a rate limited by the needle valve. Adjust this needle valve until the descending mercury in the control manometer just leads the rod, reducing the relay operation to a minimum. When properly adjusted, the pulsations caused by the opening and closing of the solenoid valve should not exceed ±1 mm. In this manner the pressure in the system will be reduced gradually at a rate governed by the descent of the steel rod.

X1.8 Procedure

X1.8.1 Pour the sample as received into a thoroughly cleaned and dry standard fluidity U-tube, without contacting the upper walls of the tube, until the vertical height of the

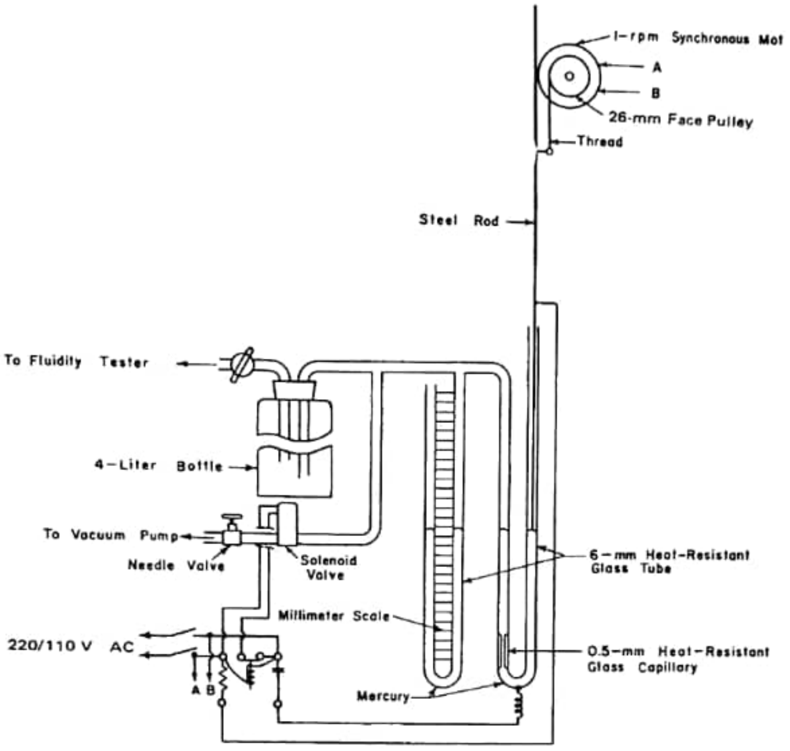


FIG. X1.4 Detail of Automatic Vacuum Controller

sample in the U-tube is 38 mm. Insert in one leg of each U-tube an ASTM Thermometer 5C in a cork that has been grooved to permit the passage of air. The thermometer must be placed in the center of the tube and its bulb immersed so that the beginning of the capillary is 3 mm below the surface of the specimen.

X1.8.2 Fix the tube in the bath set at the specific temperature, immersed to a depth of approximately 75 mm. Control the bath and sample temperatures within ±1°C and ±0.5°C, respectively, of the specified temperature of the test.

X1.8.3 Maintain the sample at the specified temperature for 30 min ± 30 s, with the U-tube connected to the automatic vacuum controller, and the stopcock or pinch-clamp open. Wind the thread on the pulley attached to the synchronous motor. Turn the power switch to the ON position. Apply suction automatically to the U-tube at the prescribed rate. Observe any movement of the specimen during a one-minute interval which is the time required to apply 152-mm Hg vacuum to the specimen in the U-tube. Immediately disconnect the U-tube from the automatic vacuum controller, turn off the power switch and rewind the thread. If the specimen has moved 2 mm or more during the time (1 min) the suction was applied, the specimen is considered fluid at the temperature of the test.

X1.9 Report

X1.9.1 Report the fluidity of the sample at a specified temperature as follows:

X1.9.1.1 If the sample fulfills the conditions of flow, as defined in X1.3.1, report fluidity: "Fluid at (temperature of test)" or fluidity at (temperature of test): "Pass."

X1.9.1.2 If the sample does not fulfill the conditions of flow, as defined in X1.3.1, report fluidity: "Not fluid at (temperature of test)" or fluidity at (temperature of test): "Fail."

X1.10 Precision and Bias

X1.10.1 As in the case of pass-fail data, no statement is made about either the precision or the bias of this method for measuring the fluidity of a residual fuel specimen since the result merely states whether there is conformance to the criteria for success specified in the procedure.

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D 97–04) that may impact the use of this standard.

(*1*) Added Test Method D 5853 to the Scope and Referenced Documents sections.

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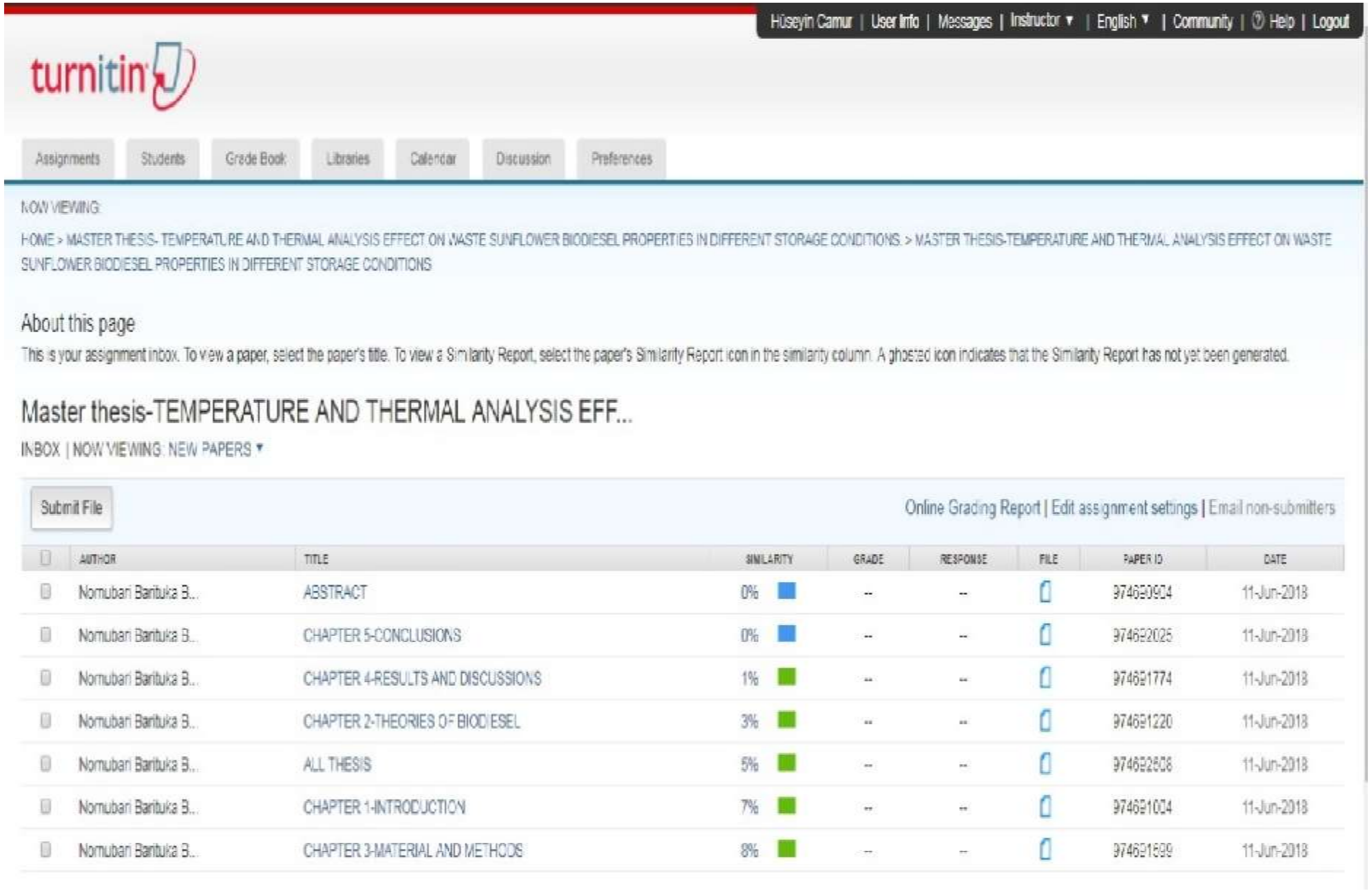

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