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TEMPERATURE AND THERMAL ANALYSIS EFFECT ON
WASTE SUNFLOWER BIODIESEL PROPERTIES
IN DIFFERENT STORAGE CONDITIONS

NEU
2018

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A THESIS SUBMITTED TO THE GRADUATE
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OF
NEAR EAST UNIVERSITY

By
NORNUBARI BARITUKA BORNU

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

NICOSIA, 201

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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.

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Date:

ACKNOWLEDGEMENTS

This thesis wouldn’t have been possible without the patience of my principal supervisor, Assist. Prof. Dr. Hüseyin ÇAMUR. I am very thankful and indebted to Dr. Youssef KASSEM and Dr. Ali ŞEFİK for their constant guidance and encouragement. To the crew of lecturers at the NEU Engineering department, I say great thanks. My gratitude to some of my course mates, who collaborated with me, especially during periods of group assignments and Examination. Their directives were never in any way minimal to my success at NEU.

My unlimited thanks and heartfelt love is dedicated to my parents Mr and Mrs Godswill Menedubabari Bornu, my brothers Barikuula Bornu, Kabolobari Bornu, my sister Joy Baridilo Bornu and my friends.

I also wish to thank my special friends, Mr. Tamusathi Nigel Babvu, Mr. Atim Gideon Atim, and Abdelrahman Alghazali for all the knowledge they taught me in Cyprus. Their ideas towards my success are unlimited.

To my parents and siblings…

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 kerosene which is an additive that reduces the viscosity and density of biodiesel, it was blended at different volume of B95K5, B90K10, B85K15 and B80K20. The effect of temperature and thermal analysis on the waste sunflower biodiesel properties in different storage conditions was experimentally determined. The kinematic viscosity was determined at 40°C and the density was measured at 15°C at ambient condition and at 40°C temperature controlled oven. The kinematic viscosity over the storage period increases by 6% for B90K10 stored at 40°C controlled oven. It was observed that kinematic viscosity and density increases over an increased storage period for the first 40-50 days. 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. The Newtonian thermal analysis was used to estimate the solid fractions in the solid-liquid mixture at CP, PP, and CFPP during solidification. The solid fractions were estimated as ~0.0048, 0.27→0.56 and ~0.63 respectively for CP, CFPP and PP for B80K20 ambient.

Keywords: Waste sunflower biodiesel; Cloud point; Pour point; Storage period; Kinematic viscosity

ÖZET

Biyodizel, atık ayçiçek yağı ve kullanılmış pişirme yağı gibi organik kalıntılar kullanılarak üretilen yenilenebilir bir yakıttır. Biyodizel, atık ayçiçek yağı yağ asidi metil esterlerinin transesterifikasyonuyla elde edilir. Bu çalışmada, atık ayçiçek yağı kullanılmış ve kullanılan ayçiçeği yağı B95K5, B90K10, B85K15 ve B80K20 gibi farklı miktarlarda kerosen ile karıştırılmıştır. Sıcaklık ve termal analizlerin, farklı depolama koşullarındaki atık ayçiçeği yağı biyodizel özellikleri üzerindeki etkisi deneysel olarak belirlenmiştir. Kinematik viskozite 40 ° C'de, yoğunluk ise 15 ° C'deki 40 ° C sıcaklık kontrollü fırında belirlendi. Depolama süresi boyunca kinematik viskozite, 6% B90K10 aralığında değişmiştir. Kinematik viskozite ve yoğunluğun depolama süresindeki artışla birlikte arttığı gözlenmiştir. Numunelerin davranışında Farklı depolama koşulları ve farklı karışım oranlarıyla birlikte numunelerin özelliklerinde çeşitlilik gözlendi. Bulutlanma noktası, akma noktası ve soğuk filtre tıkama noktası gibi soğuk akış özellikleri sırasıyla ASTM D2500, ASTM D6371-05 ve ASTM D97 standartları kullanılarak belirlenmiştir.

Anahtar kelimeler: Atık ayçiçeği biyodizel; Bulut noktası; Akma noktası; Saklama süresi; kinematik viskozite

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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
Z_N	Newtonian Zero

LIST OF SYMBOLS USED

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

Greek Symbols

μ	Dynamic viscosity	(N.s/m^2)
ν	Kinematic viscosity	(mm^2/s)
ρ	Density	(kg/m^3)
τ	Shear Stress	(N/m^2)

CHAPTER 1

INTRODUCTION

This chapter introduces us to biodiesel as a renewable form of energy, the importance of biodiesel and how biodiesel is obtained from different oil bearing sources. It also gives us the highlights of literatures that has been done on various types of biodiesel including the waste sunflower biodiesel. Furthermore, this chapter present to us the aim of this research and the outline of the research as stated in subsequent chapters.

1.1 Background

Renewable fuels from organic remains is being given more attention as alternative to reduce dependence on fossil fuel. Biodiesel acquired from sunflower oil, used cooking oils, vegetable oils etc; plays an important role among these fuels (Knothe et al., 2005).

Utilization of biodiesel in compression ignition engines is now broadly accepted as an alternative to fossil fuel diesel (Petrol diesel). There have been steady increase in the quantity of biodiesel produced around the EU countries, about 5.7 million and 4.9 million tonnes were produced in 2007 and 2006 respectively. In the world recently, EU contribute about 68% of the biodiesel produced.

However, in the EU biofuel production biodiesel forms about eighty percent of its production. In 2007 the United State recorded biodiesel production of 1.5million tonnes which made them the second largest producer of biodiesel (Mittelbach and Remschmidt, 2004).

Mechanical durability and economic advantage has made biodiesel engine widely used worldwide (Lee et al., 2004). According to Moron et al (2007) diesel engine have captivating attribute which makes sectors such as agriculture, road, construction, train transport, mining military etc to utilize it immeasurably, some of these attributes includes high torque, robustness and lower fuel consumption.

Presently, there is a higher request for energy (Mbarawa, 2010). This is as a result of diminishing fossil fuel reserves and instability in the price of fossil fuel. Unlike biofuel, the transesterification of sunflower bring about the production of biodiesel. Biodiesel can be produced from different type of feedstock, this trait makes biodiesel unique from other biofuel (Evangelos and Giakoumis, 2013). Some of the feedstock acquired from biodiesel includes cooking oil, sunflower oil animal fat, jatropha, soybean, rapeseed etc (Marzena and Piotr, 2008).

Biodiesel made from canola is prevalently used in Europe while soy biodiesel is commonly used in the United State. In order to obtain efficient performance of biodiesel equipment, petrol-diesel is blended with biodiesel in certain ratio this helps to reduce the price of fuel (Kinast et al., 2003). Complete removal of water, glycerin, alcohol, catalyst, free fatty acids, soaps is very necessary in the production of biodiesel; they contaminant the methyl ester product and reduce the standard of the biodiesel, this is done to ensure quality control measures in the production of biodiesel (Kinast et al., 2003). As compared to petro-diesel, the most significant benefit of biodiesel is that it is renewable.

The human environment is a key factor to the survival of mankind, as such environmental laws has been put in place to caution environmental emission from any product. This had made biodiesel highly sort after renewable fuel since it reduces environmental emission. Consequently, biodiesel when compared to diesel fuel has high pour point and high viscosity (Seung et al., 2008).

Biodiesel should be utilize in the period of six months from production date, this is because of the instability of biodiesel and also some of the activities such as exposure to light, heat and water can be instrumental in minimizing the quality of the biodiesel. However, the utilization of additives in its storage improves the shelf life (Ralph et al., 2009).

1.2 Literature Review

Severally approach have been used to forecast the kinematic viscosity, density and the cold flow properties of biodiesel. Some of the good qualities of biodiesel are its clean combustion behavior, renewability and biodegradability while some of the draw backs are its unfavorable cold flow properties (ie, high cloud point CP, pour point PP, and cold filter plugging point CFPP) (Hanna and Ma, 1999). These are the properties of biodiesel that suggest the rate at which the biodiesel begin to congeal when subjected to a weather of low temperature and it starts to clog the engine filter (Freire et al, 2012).

Among these disadvantages, the major problem associated with biodiesel are cold flow behavior and the instability of the biodiesel.

The calorific value of the biodiesel can be decreased by water and this facilitate the corrosion of biodiesel engine, also during long term storage of biodiesel microorganism are formed by the aid of water and give rise to the formation of oxidation product (Schlink, and Faas, 2009). Around the world today, there is a greater demand for compatibility of technical parameters as regards products and services. This is as a result of increase in globalization as such international standards are developed to harmonize the world standards (Moser et al., 2009). Standards are set by various committee as prescribed by law in different regions, according to European committee for standardization (CEN) specification for biofuel are accounted for in details by EN1421 whereas ASTM D6751is described in the United State by the American Society for Test and Materials.

Sometimes, the accuracy of the product and services differs for international standards and as such they are not certain. For the convergence of some rules various guidelines have to be studied extensively. For standards to be generally accepted, they have to be reviewed regularly so as to meet the market demand and methodologies as it is obtained at the moment. According to Alptekin et al (2009) biodiesel made from soybean and waste palm oil, was experimentally investigated to obtain a first degree empirical equation which relate density and biodiesel percentage blend as displayed in equation (1.1).

$$\rho = Ax + B \tag{1.1}$$

Where

The density (g/cm^3) is given by ρ

Considering various type of biodiesel A and B should be their constants,

The biodiesel fraction should be given by the value x

Bhale (2009) investigated that when biodiesel is blended with diesel and heated at a temperature below 25°C there is a tendency for an increment in the biodiesel viscosity,

With the proper knowledge of mixing law as Grune Nissan and Katti-Chaudhri laws, the viscosity of biodiesel can be obtained. The laws are expressed mathematically thus;

$$\ln(\eta_{max}) = m_1 \ln(\eta_1) + m_2 \ln(\eta_2) \tag{1.2}$$

Where:

Components 1 and 2 have η_1 and η_2 as their kinematic viscosities (mm^2/s) respectively

Components 2 and 1 have m_2 and $m_1$as their mass respectively

Considering the kinematic viscosity (mm^2/s) of the blends to be η_{max} is.

At 40°C the difference in viscosity as a function of distinctive percentage blends of various biodiesel as soybean oil, biodiesel cottonseed oil biodiesel, waste palm oil biodiesel, and sunflower oil biodiesel, was calculated using an empirical equation considering the fraction of biodiesel as the main parameter in the mixture. The investigation came out that the measured value was similar to the empirical value (Alpetekin and Canakci, 2009).

$$\eta_{blend} = Ax^2 + Bx + C \tag{1.3}$$

Where

Kinematic viscosity (mm^2/s) be taken to be η,

The fraction of biodiesel is taken as x, A, B, C are coefficients

According to Riazi and Al-Otaibi (2001), a condition for the evaluation of consistency of fluid hydrocarbons and petrol-diesel mixture at numerous temperature derived from index of refraction was obtained. Notwithstanding, in this study, the condition the evaluation of sub-atomic weight, particular gravity, index of refraction and elevated temperature of blends as input is needed.

$$\frac{1}{\mu} = \frac{B}{I} + A \tag{1.4}$$

According to the condition above, consider μ as dynamic viscosity

A and B are constants particular to every segment and I should be taken as index of refraction.

Examination of an adjusted condition to decide the viscosity at various temperature was demonstrated by Tat and Van Gerpen (1999) as follows

$$\ln(\eta) = A + \frac{B}{T} + \frac{C}{T^2} \tag{1.5}$$

Let:

The temperature in K be taken as T, consider the kinematic viscosity (mm^2/s) as η

A, B and C are constants.

The constants A, B and C which shift depending on the type biodiesel and biodiesel fraction has placed constrain on the utilization of the Tat and Van Gerpen's condition.

The measure of resistance of flow over time by a liquid is the kinematic viscosity of that fluid, thus, its discoveries gives great parameter for similarity with ASTM guidelines (Babagana et al., 2012).

The aftereffect of Moradi, et al.(2013) in any case, demonstrated that expanding the biodiesel volume fraction in biodiesel– diesel mixture builds the kinematic viscosity, subsequently volume of mix ought to be considered.

Okoro et al.(2011) said that mixing biodiesel with petro-diesel should be possible to amend viscosity values to support its use in motor engine.

All fuels have comparative conduct at temperature moving towards the development of crystals, there is an increment in the viscosity of biodiesel and the viscosity of the mixture changes among the biodiesel and petro-diesel depending on their mixing proportion (Tat and Gerpen, 1999).

1.3 Research Aim

The aim of this work is to determine experimentally the temperature and thermal analysis effect on waste sunflower biodiesel properties in different storage conditions. To investigate the kinematic viscosity, density and cold flow properties of waste sunflower biodiesel blended with kerosene at different percentages of B95K5, B90K10, B85K15 and B80K20 stored at ambient temperature and also at 40°C controlled oven, to determine the solid fraction of waste sunflower biodiesel using the computer aided cooling curve analysis (CA-CCA) by employing the Newtonian thermal analysis and also the effect of temperature, storage period and blend composition on the biodiesel properties.

1.4 Thesis outline

Chapter one provides general information of preceding work done by researchers on this topic, the reviews of those works and a concise introduction of biodiesel as a renewable energy source. Chapter two provide further details about biodiesel, the advantages and disadvantages of biodiesel, analyze critically the kinematic viscosity, density and the cold flow properties of biodiesel. It also describes the various theories used in obtaining the various parameters. Chapter three focuses on the materials and methods used in arriving at the various results obtained in this work, chapter four gives the results of the various analysis carried out in this work; chapter five focuses on the conclusions and recommendations for future work that may be carried out on this work also the references are given at the end of the work.

CHAPTER 2

THEORIES OF BIODIESEL

This chapter gives us the definition of biodiesel and the various theories used in the analysis these samples. It also states the advantages and disadvantages of biodiesel, the concept of viscosity where we have kinematic viscosity and dynamic viscosity. It further explains the cold flow properties of biodiesel, density, Newtonian thermal analysis and the factors affecting kinematic viscosity.

In this chapter oxidation stability and acid number is outlined in detail, the chapter went further to explain the types of fluid (Newtonian and Non-Newtonian fluid).

2.1 Definition

Biodiesel can be alluded to as a sustainable fuel got from an inexhaustible oil bearing sources, for example, vegetable oils sunflower oil, used cooking oil, animal fats and used frying oils through the process of transesterification. The transesterification is accomplished with monohydric alcohols like methanol and ethanol within the sight of an alkali catalyst (Josh and pegg., 2007). Methanol is one of the most utilized alcohol, which makes a blend of unsaturated fat methyl esters (FAME). It is picking up consideration as an alternative fuel.

Estimating the dependence of physicochemical properties of biodiesel with the temperature is very pertinent, this is because of higher request in production and utilization of biofuel (Blangino et al., 2008).

Biodiesel can be mixed in any extent or used alone in a diesel engine, a framework known as the "B" factor is used in various part of the world today to express the concentration in percentage of biodiesel in any fuel mix. Biodiesel when blended with petro-diesel in different volume fraction can be expressed as, "BXX" with "XX" representing the content of biodiesel contained in the mixture.

- 100% biodiesel is alluded to as B100
- 90% biodiesel, 10% petro-diesel is marked B90
- 95% biodiesel, 5% petro-diesel is marked B95

- 80% biodiesel, 20% petro-diesel is marked B80

Biodiesel can be mixed with petro-diesel at any proportion or ratio to make biodiesel blends.

2.2 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.
- 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.
- 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.

2.3 Disadvantages 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.
- 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.

2.4 The Concept of Viscosity

When dealing with liquid transported through pipeline viscosity (μ) has been utilized broadly. The measure of resistance of flow over time by a liquid is the kinematic viscosity of that fluid. It is a fundamental characteristic property of all fluids which defines the integral of the interaction forces of the molecules. Viscosity can be considered as the thickness of a fluid, 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.

Viscosity is the internal resistance in intermolecular movement in fluid, the molecules of the fluid are held together by cohesive forces and as such they are tightly strong. However, as heat is been applied to the fluid the molecules gain energy and they begin to slide over each other, this heat breaks the bond between the molecules and we consider fluid to have dissolved. However, when the molecules are slide they starts gradually followed by a rapid movement as a result of increase in temperature, this makes the fluid viscous.
Some parameters that depends on viscosity for its determination are:

- Reynolds number
- Prandlt number

2.5 Types of Viscosity

Dynamic viscosity and kinematic viscosity are the two types of viscosity majorly discussed in fluid dynamics. However, they can be used exchangeably if the density of the fluid is known.

2.5.1 Dynamic Viscosity

The dynamic viscosity or shear viscosity of a fluid shows the fluid resistance to flow in such fluid the adjacent layer move parallel to each other at a non-identical speed. Consider a fluid flow in a layer confined between two horizontal plates, fixed at one end and horizontally moving at a regular speed as shown in the figure below

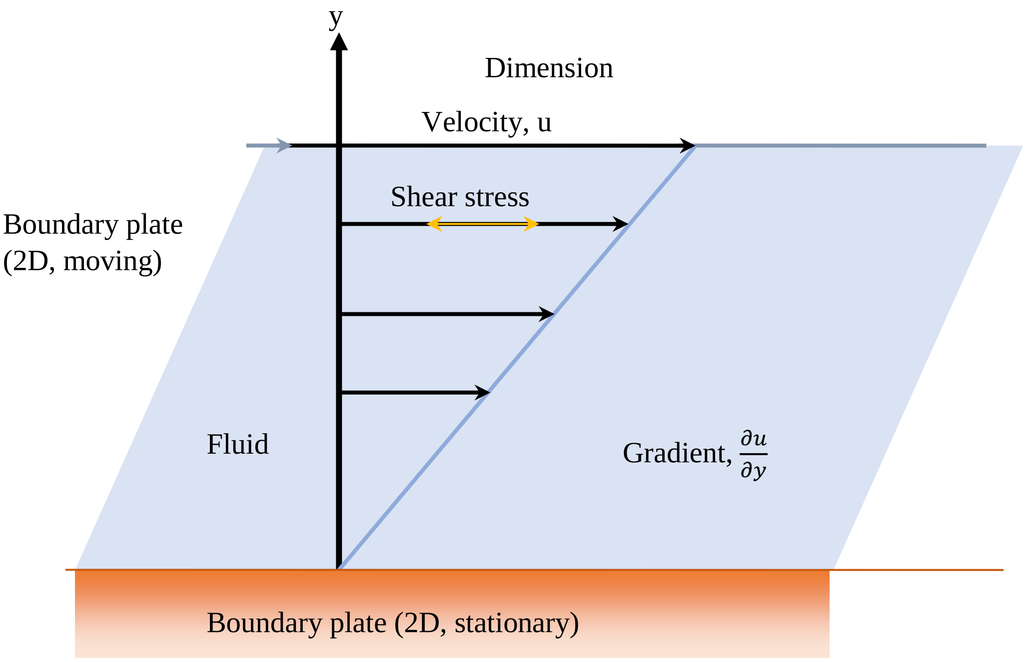


Figure 2.1: The flow of liquid between two plates in parallel (Wang, 1991)

Given two plates, let the movement of the top plate be too little that particles of the fluid moves parallel thereto, the movement of the fluid particle will shift directly at the base to u

at the top. At each layer the fluid particle will move faster than the one underneath, frictional force will exist between them which will in turn generate a force resisting their relative motion. A force is applied by the fluid in a reversed direction on the top plate which creates a force equivalent to that which occurs at the bottom but in reverse direction, for the top plate to be in constant motion and external force (F) is needed whose speed u will be directly proportional to the area (A). However, the force F and the separation (y) will be inversely proportional

$$F = \mu A \frac{\partial \mu}{\partial y} \tag{2.1}$$

Let:

The dynamic viscosity be µ.

$\frac{\partial u}{\partial y}$ be the shear rate of deformation

Sir Isaac Newton suggested that the mathematical differential expression as seen below can be used to express the viscous force.

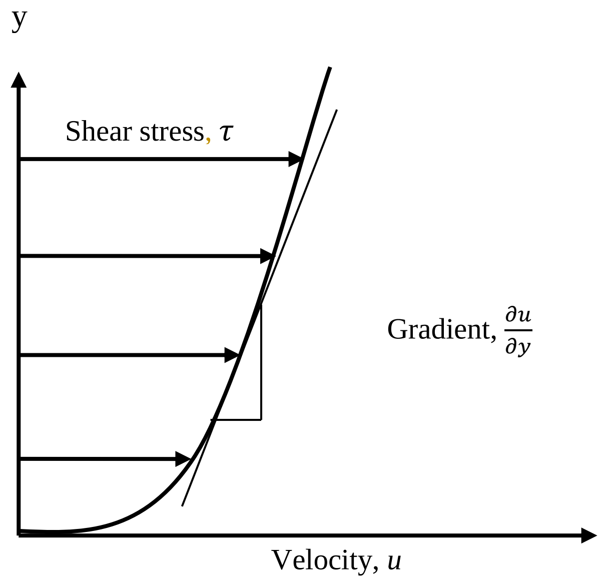


Figure 2.2: Expression of the differential viscous force (Munson et al, 1998)

$$\tau = \frac{\partial u}{\partial y} \, . \mu \qquad 2.2$$

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

Consider:

$\tau = \frac{F}{A} \, . \, \frac{\partial U}{\partial y}$ as the velocity of shear

The formular can be obtained considering the perpendicularity of the y-axis to the fluid and based on concept of fluid along parallel line.

2.5.2 Kinematic Viscosity

The kinematic viscosity (also called "momentum diffusivity") can be stated as the proportion of the absolute viscosity to the density of a substance at a similar temperature.

It is thus denoted by the letter v and is measured in $\frac{mm^2}{s}$ expressed mathematically thus,

$$v = \frac{\mu}{\rho} \qquad (2.3)$$

Let:
v be the kinematic viscosity,
ρ is the fluid density
μ dynamic viscosity.

2.5.3 Viscosity and Affecting Factors

Temperature: the temperature of a fluid is proportional to the viscosity. Accompanied by its shear rate the most dominating influence is the temperature. An increase in temperature, lowers the viscosity of the substance. Hence, reduction in temperature gives rise to an increment in viscosity. The temperature of any substance is related inversely proportional to its viscosity. Depending on the substance how much it is influenced by temperature as little as 1°C decrease in temperature causes 10% increase in viscosity. By increasing the temperature the viscosity of the fluid increases in molecular motion. (Moradi, et al, 2013)

Pressure: Ordinary, the viscosity of a fluid increases with increasing pressure, similar to the influence of temperature on viscosity, liquids are less influenced by the application of pressure. This is because liquids (other than gases) are almost non-compressible at low or medium temperature

When pressure increases, fluid viscosity also increases. However viscosity increases for a pressure change from 0.1 to 30 mPa will produce the same viscosity change as 1K (1^0C.) For an enormous pressure difference of 0.1 to 200 mPa, a viscosity change of a factor of 3 to 7 occurs. This is experienced for low molecular liquids. For higher viscosity changes, the factor can rise to 2000. Since pressure is inversely proportional to volume

As the pressure increases, the volume pressure in the material structure decreases due to compression. The molecules in the substance come closer and move less freely. The internal frictional force increases, resistance increases and consequently viscosity increases. (Thomas, 2011).

Conditions at Room Temperature: it is noted that the conditions exude when liquids are subjected to an external force. This results to both temperature and pressure changes. Temperature and pressure changes can cause a fluid to develop different type of flows and consequently viscosity changes. The flow conditions might be laminar or turbulent. Laminar flow is the only flow that can be used to test a fluid's viscosity. In a lamina fluid flow, the fluid moves in very tinny layers, this causes the molecules to be fixed in the layers. Such a fluid-flow presents an orderly structure. (Marzena and Piotr, 2008).

Table 2.1: Diesel Fuel Kinematic Viscosity Standard (Knothe & Steidley, 2005)

Standard	Location	Fuel	Method	Kinematic viscosity (mm^2/s)
ASTM D975	United States	Petro-diesel	ASTM D445	1.9-4.1
ASTM D6751	United States	Biodiesel	ASTM D445	1.9-6.0
EN590	Europe	Petro-diesel	ISO 3104	2.0-4.5
EN 14214	Europe	Biodiesel	ISO 3104	3.5-5.0

ASTM= America Society for Testing and Materials
ISO= International Standards Organization

2.6 Viscosity Measurement

There are different type of viscometer used in the measurement of kinematic viscosity. The viscosity of some fluid cannot be ascertain by single value hence they demand more parameters before it can be measured accurately; for such a fluid a rheometer is used to measure the kinematic viscosity (Sahin & Sumnu, 2006).

The kinematic viscosity of a fluid is acquired by estimating the time taken for a fluid flowing under gravity to pass through a calibrated capillary viscometer tube. They two main types of viscometers includes:

- Rotational viscometer
- Capillary viscometer

2.7 Theory of Capillary Viscometer

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

For the accuracy of Hagen-Poiseuille equation certain assumption where made which includes:

- The capillary pipe most be straight capillary pipe with regular cross section which will allow laminar free flow of fluid and the cross section longer than the diameter of the pipe
- The fluid should a Newtonian fluid and also incompressible

Consider a completely created laminar fluid flow along a straight vertical pipe of round cross section as appeared 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 et al., 2007).

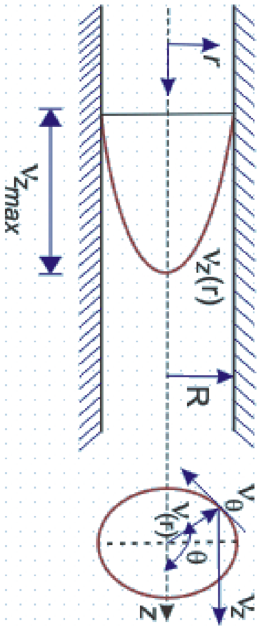


Figure 2.3: Diagrammatic illustration of Hagen -poiseuille fluid flow (Viswanath et al., 2007).

Let

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

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

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

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 volume of liquid

t be the time taken

$$\mathrm{V} = \frac{\mu}{\rho} \tag{2.14}$$

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

Δpgh

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,

$$v = k.t \tag{2.18}$$

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. The moment the K-value is obtained the fluids viscosity can be acquired by measuring the elapsed time for a known volume of the fluid to flow between two graduation mark (Viswanath et al., 2007).

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

2.8 Kinetic Energy Correction (HC)

The experiment is likely to be affected by certain factors when permit errors as a result of measuring the viscosity of the fluid. To caution this effect, some measures are put in place to balance the accuracy of the result.

This requires that the kinetic energy correction (HC)be subtracted from the elapsed time measured and the equation becomes

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

Where: K is the viscometer capillary number constant

y is the kinetic energy correction

2.9 Density

One of the necessary parameter in the analysis of the biodiesel is its density (ρ), it is the mass per unit volume of the biodiesel. The parameter affect greatly the performance of biodiesel such as:

- Cetane number
- Heating value
- Viscosity

Which are all linked to the mass density; the density of a biodiesel is expressed mathematically thus (Ramírez-Verduzco et al., 2011).

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

Where: V is the biodiesel volume

m is the biodiesel mass

An increase in temperature causes a corresponding decrease in density hence density is temperature dependent, this is expressed mathematically thus

$$\rho = a + bT \tag{2.22}$$

Where: ρ is the density

a and b are correlation parameters,

T is the temperature in °C (Rodenbush et al., 1999).

2.10 Cold Flow Properties of Biodiesel

The properties which provide the details about the fluid behavior of biodiesel at low temperature is known as the cold flow properties of biodiesel. This includes

- Cloud point
- Pour point
- Cold filter plugging point

The minimum temperature at which biodiesel can be used increases as a result of higher level of saturation from vegetable to animal source (Kinast, 2003). When the biodiesel is subject to a low temperature, the constituent of the biodiesel nucleate to form crystal wax. This causes the biodiesel to develop startup problem for engines during the low temperature weather (Perez, 2010).

2.10.1 Cloud Point

In the study of biodiesel, the cloud point is the temperature at which the sample first indicates a crystal like formation in it when subjected to a low temperature (Ramadhas, 2011).

The susceptibility of biodiesel to plug filter or small orifice in cold weather operations is stipulated by the cloud point of the biodiesel sample. When compared to petro-diesel the cloud point of biodiesel is low as such it makes it difficult to operate a biodiesel engine in cold weather (Ramadhas, 2011). The cloud point measurement was done as per (ASTM D2500) standards.

2.10.2 Pour Point

When a biodiesel sample is subjected to cold weather condition for quite some time, it gets to a point where the biodiesel can no longer flow as a result of gel formation, at this point the biodiesel loses it flow ability, the temperature at which this happens is called the pour point of the biodiesel. It is lower than the cloud point (Duffield, 1998). The pour point measurement was done as per (ASTM D97-2005)

2.10.3 Cold Filter Plugging Point

This is defined as the minimum temperature at which a biodiesel sample with a volume still goes through a standard filter in a specific time 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)

2.11 Newtonian Thermal Analysis

The heat produced when freezing the biodiesel sample can be demonstrated by the equation of heat balance (Kierkus and Sokolowski, 1999).

$$\frac{dQ}{dt} - MC_p\frac{dT}{dt} = UA(T - T_0) \tag{2.23}$$

Let;

The mass of the sample is given by M,

The specific heat of the sample is given by C_p

T is the sample temperature,

The time is given by *t*,

The overall heat transfer coefficient is given by U,

The surface area of sample is given as A

The cooling bath temperature is given as T_0

The heat produced during freezing is given by Q

Assuming in course of cooling, the transformation phase did not occur i.e. $\frac{dQ}{dt} = 0$, the rate of cooling of the biodiesel sample can be written thus,

$$\frac{dT}{dt} = -\frac{UA(T - T_0)}{MC_p} = Z_N \tag{2.24}$$

The baseline or the Newtonian zero curve is known as Z_N

Hence, *L* which is the total latent heat can be determined

$$L = \frac{Q}{M} = C_P \int_{t_s}^{t_e} \left[\left(\frac{dT}{dt} \right)_{cc} - Z_N \right] dt \tag{2.25}$$

Where:

t_e and t_s are the end of freezing and start time and the cooling curve first derivative can be taken to be *cc*

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

$$L = C_p x \text{ (Area between derived cooling curve and zero curve)} \tag{2.26}$$

Considering specific heat specific heat (C_p) to be given, Equation 2.26 can be used to determine the latent heat, also given the Zero curve (Z_N) and the area between the first derivative (CC), the solid fraction at time *t* of solidification can be obtained (Fras et al., 1993).

2.12 Stability of Biodiesel

When biodiesel is stored, it has the tendency to interact with its environment which yield unfavorable results, the ability of the biodiesel to withstand the effect of degradation process caused by this action is known as biodiesel stability (Westbrook, 2003).

2.13 Oxidation Stability

One of the properties that possess a major drawback on the biodiesel stability stored over a period of time is oxidation stability. Oxidation stability is not the same as storage stability, in that oxidation occurs during production, storage period and in course of using the biodiesel (Knothe and Dunn, 2003).

When biodiesel degrades, it generates oxidation product that causes draw backs on the fuel properties and as such reduces the engine performance and fuel quality (Bouaid and Martinez, 2009).

2.14 Acid Number

Acid number is a measure of the quantity of fatty acids and mineral acid present in a biodiesel sample, its the amount of base expressed in milligrams of potassium hydroxide (KOH) needed to neutralize one gram of biodiesel sample. Acid number is a useful tool in examining the degradation of biodiesel in course of storage. Acid number increases with degradation (Knothe, 2006)

2.15 Types of Fluid

There are two major types of fluids, these are

- Non-Newtonian
- Newtonian fluids

Non-Newtonian fluid: A Non-Newtonian fluid is a fluid in which the shear stress is not directly proportional to deformation rate. Non-Newtonian fluids do not follow Newton`s law of viscosity. The viscosity of Non-Newtonian fluid is dependent on shear rate and shear stress history.

However, Non-Newtonian fluids are commonly classified as having time-independent or time-dependent behavior. Two familiar examples of are toothpaste and Lucite paint. Lucite paint is very thick when in the can, but becomes thin when sheared by brushing. Toothpaste behaves as a fluid when squeezed from the tube. However, it does not run out by itself when the cap is removed, solutions containing long chain polymers as well as slurries and suspensions are usually Non-Newtonian (Philip et al, 1992), some examples are:

- Bingham plastic
- Pseudo plastic
- Dilatat
- Bingham pseudo plastic

Newtonian Fluid: In a Newtonian fluid, the relation between the shear stress and shear rate is linear passing through the origin, In Newtonian fluid the shear stress is directly proportional to rate of strain or fluid with constant viscosity at affixed temperature and pressure.

A Newtonian fluid`s viscosity remains constant no matter the amount of stress applied for a constant temperature, they obeys newton`s law of viscosity which is given be the expression

$$\tau = \mu \frac{du}{dy} \qquad (2.27)$$

Where,

τ is the shear stress exerted by the fluid

μ is the constant of proportionality (fluid viscosity)

$\frac{du}{dy}$ is the velocity gradient perpendicular to the direction of shear

Common example of Newtonian fluid are air, water, gases, petroleum products, mineral oils etc (Robert et al, 2012). For this thesis it is noted that biodiesel falls in the category of Newtonian fluid because when a shear stress is applied on the fluid its fluidity or strain is proportional. As such we limit our study and analysis to Newtonian fluids.

2.16 SOLID FRACTION

The solid fraction is the amount of particulate matter in the fuel during cooling, for the solid fraction the ratio of the incremental cumulative area, A_n to the total area A_{Total} between the Newtonian curve and the cooling rate gives the solid fraction (f_s) during solidification.

$$f_s = \frac{A_n}{A_{Total}} \tag{2.28}$$

$$A_n = \sum_{i=1}^{n} \left[\left(\frac{1}{2} (T'_{i+1} + T'_i) - \frac{1}{2} (Z_{N_{i+1}} + Z_{N_i}) \right) \times (t_{i+1} - t_i) \right] \tag{2.29}$$

Where:

f_s is the Solid fraction

A_n is the incremental cumulative area

A_{Total} is the total area between the Newtonian curve and the cooling rate

T'_i is the rate

Z_{N_i} is the Newtonian zero curve

t_i is the instantaneous time.

CHAPTER 3

MATERIALS AND METHOD

This chapter highlights the various materials used in arriving at our experimental results, these materials includes: laboratory oven, viscometer holder, capillary viscometer, beaker, vacuumed syringe, pycnometer, stop watch etc. Furthermore, it explains the methods used in measuring the kinematic viscosity, the density and the cold flow properties.

3.1 Materials

For the study, locally available waste sunflower oil (WSFO) biodiesel obtained from the cafeteria in Near East University Cyprus have been used for the analysis. The waste sunflower biodiesel fuel was processed by transesterification process from methanol and waste sunflower oil (WSFO). The waste sunflower biodiesel fuel was mixed with kerosene at 5%, 10%, 15% and 20% respective proportion. They were mixed thoroughly into a homogenous solution and kept in a 750mL closed glass bottles.

Table 3.1: Waste sunflower biodiesel blends at various percentage

Materials	**B95K5**	**B90K10**	**B85K15**	**B80K20**
WSFO Biodiesel (ml)	2850	2700	2550	2400
Kerosene (ml)	150	300	450	600

For each biodiesel blend, four of the 750mL closed glass bottles were prepared. Two bottles of each sample was stored at ambient (room) temperature and the other two were stored in a temperature controlled oven at 40°C.

These samples were stored at their respective locations for ten days before the measurement of the density and kinematic viscosity. The samples were taken out periodically after every 10 days to study the storage condition effect, density and the kinematic viscosity.

Figure 3.1: Biodiesel sample

Figure 3.2: Biodiesel sample stored in an oven

3.2 Laboratory Oven

The laboratory oven is made up of thermostat and is thermally insulated, it provides uniform temperatures throughout its interior, the heat is generated by a double lamp source of 75W controlled by a thermostat. The lamps are covered with Aluminium foil sheet to prevent the lamps from having direct contact with itself and also the biodiesel sample stored in it.

Figure 3.3: 40°C Temperature Controlled Oven

3.3 Temperature Measurement

In obtaining the density and kinematic viscosity of a bodies temperature plays a crucial role as both parameters are temperature sensitive. A 2°C change in temperature causes a significant increase kinematic viscosity of the biodiesel.

To achieve a constant and accurate temperature, a thermostat and a mercury thermometer was used in the cooling bath and the beaker respectively.

The thermostat which is connected to compressor automatically turns on and off the compressor while maintaining the temperature of the cooling bath by ±1°C.

Figure 3.4: Thermostat reading

3.4 Accessories

In achieving an accurate kinematic viscosity and density some accessories listed below were used.

Glass Pipette: This is used to convey the measured volume of biodiesel sample into the viscometer

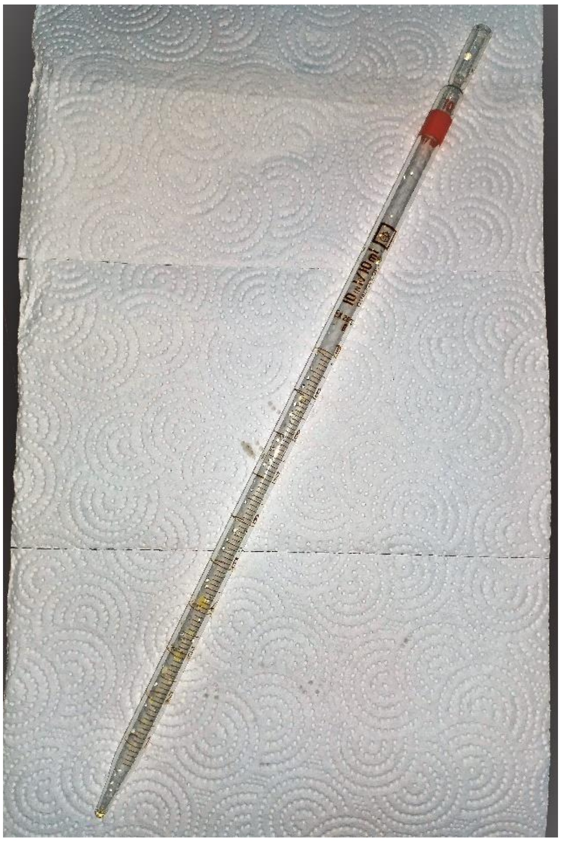


Figure 3.5: Pipette

Viscometer holder: This is used in holding the ubbelohde capillary vertically upright in the cooling bath and beaker.

Figure 3.6: Viscometer holder

Beakers: these are glass bowls used in measuring the volume of biodiesel sample and also for storing water that is to be heated or cooled, it also serves as a heating bath for ubbelohde capillary viscometer.

Figure 3.7: 2000ml Beaker

Stop Watch: This device was used in measuring the time with 0.01seconds least count, it is utilize in measuring the accurate time the start and finish of the standard procedures.

Vacuumed Syringe: This is used for supping (suction) the biodiesel sample during measurement.

Figure 3.8: Vacuum Syringe

3.5 METHODS

3.5.1 Experimental Setup for Viscosity and Density Measurement at 40°C and 15°C Respectively and the Cold Flow Properties of Biodiesel

3.5.2 Kinematic Viscosity measurement

For this experiment the measurement of the kinematic viscosity (v) were made utilizing the ubbelohde capillary viscometer. In determining the kinematic viscosity of the sample, The experiment was carried out according to ASTM D455 standard.

It was measured at the temperature of 40°C with the accuracy of ±0.1%. To obtain an accurate result the heating bath was heated to a temperature of 40°C±0.1°C, while 20mL of the biodiesel sample was measured with a pipette and poured into the ubbelohde capillary viscometer.

The ubbelohde capillary viscometer was placed in the viscometer holder and kept in the heating bath, the biodiesel sample was allowed in the heating bath for 20minutes at a temperature of 40°C for the sample to acquire homogenous temperature of 40°C with that of the heating bath.

The suction syringe was used to sup the biodiesel sample into the calibrated glass tube of the viscometer.

The flow rate for the calibrated glass capillary viscometer tube was recorded, the stop watch was used to record the time in this test. This procedure was repeated three consecutive times while taking the average time.

It is requires that the kinetic energy correction (HC) be subtracted from the elapsed time measured and multiplied by the viscosity constant K to acquire the kinematic viscosity given in Equation 3.1

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

Let: 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

The instrument (capillary viscometer) constant K was specified by the manufacturer and chosen according to the manufacturers' catalogue on table 3.1.

For this study, the ubbelohde capillary viscometer used was type (I) for the kinematic viscosity measuring range of $2-10\left({mm^2}/{s}\right)$ hence, K=0.009820

The diagram below displays the setup for measurement of the biodiesel kinematic viscosity

Figure 3.9: Experimental setup for kinematic viscosity measurement

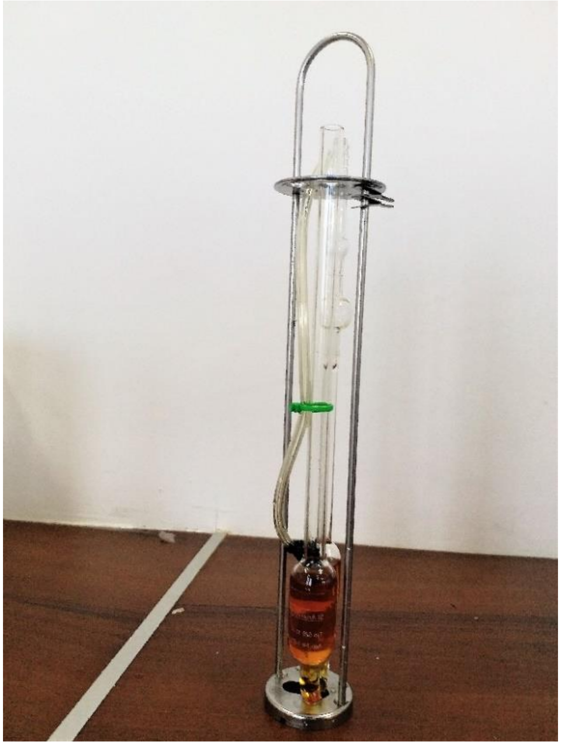


Figure 3.10: Ubbelohde Viscometer setup

Table 3.2: Ubbelohde Viscometer Technical Specification

Type No.	**Capillary No**	**Capillary Dia. 1± 0.01[mm]**	**Constant K**	**Measuring range $[\mathbf{mm^2/s}]$**
525 03	0c	0.36	0.002856	0.6 … 3
525 10	I	0.58	0.009820	2 … 10 ~
525 13	Ic	0.78	0.02944	6 … 30
525 20	II	1.03	0.08947	20 … 100
525 23	IIc	1.36	0.2812	60 … 300

Table 3.3: Kinetic energy correction factor table 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 3.3: Continue

Flow time	**Capillary No**						
	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

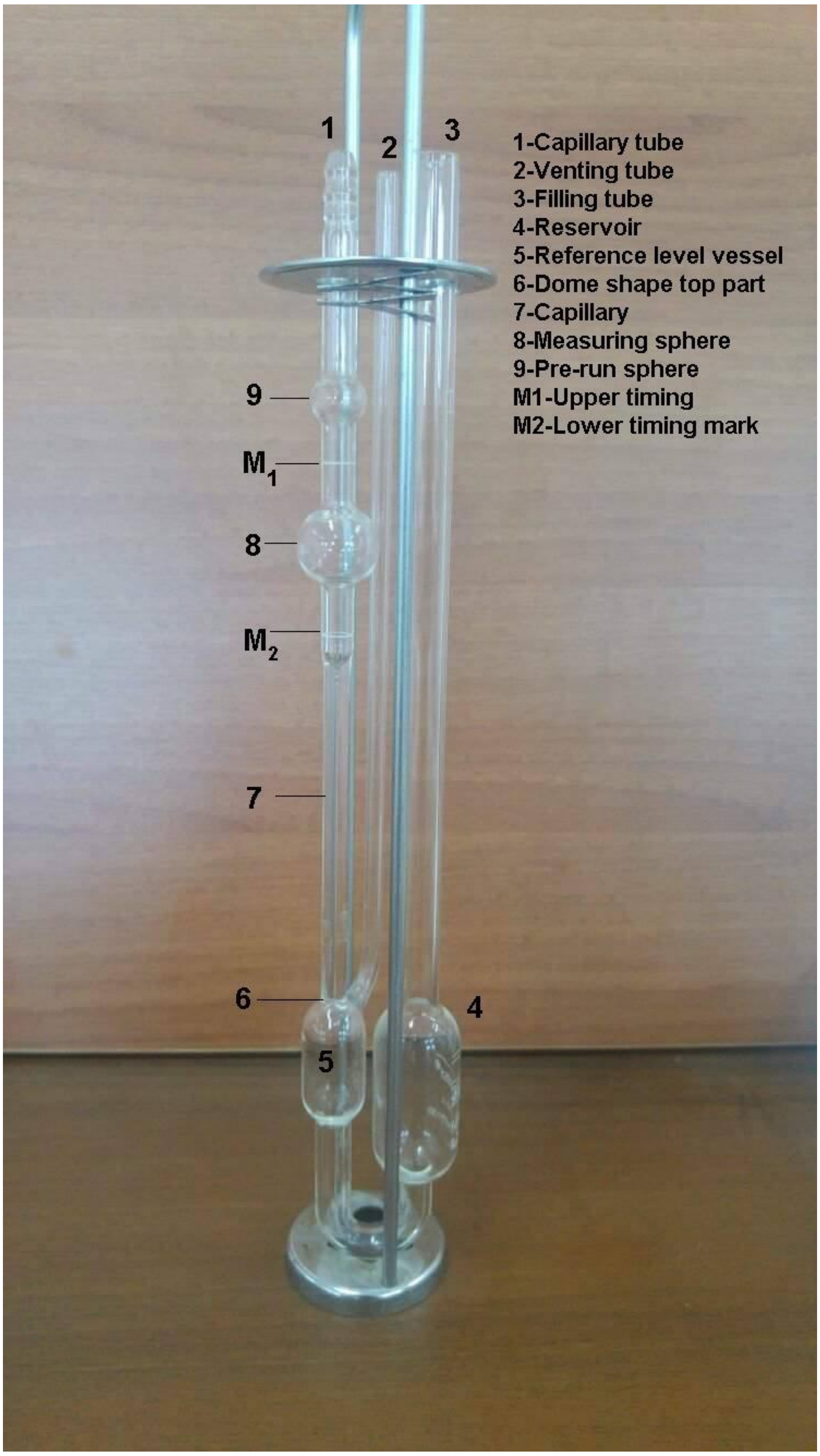
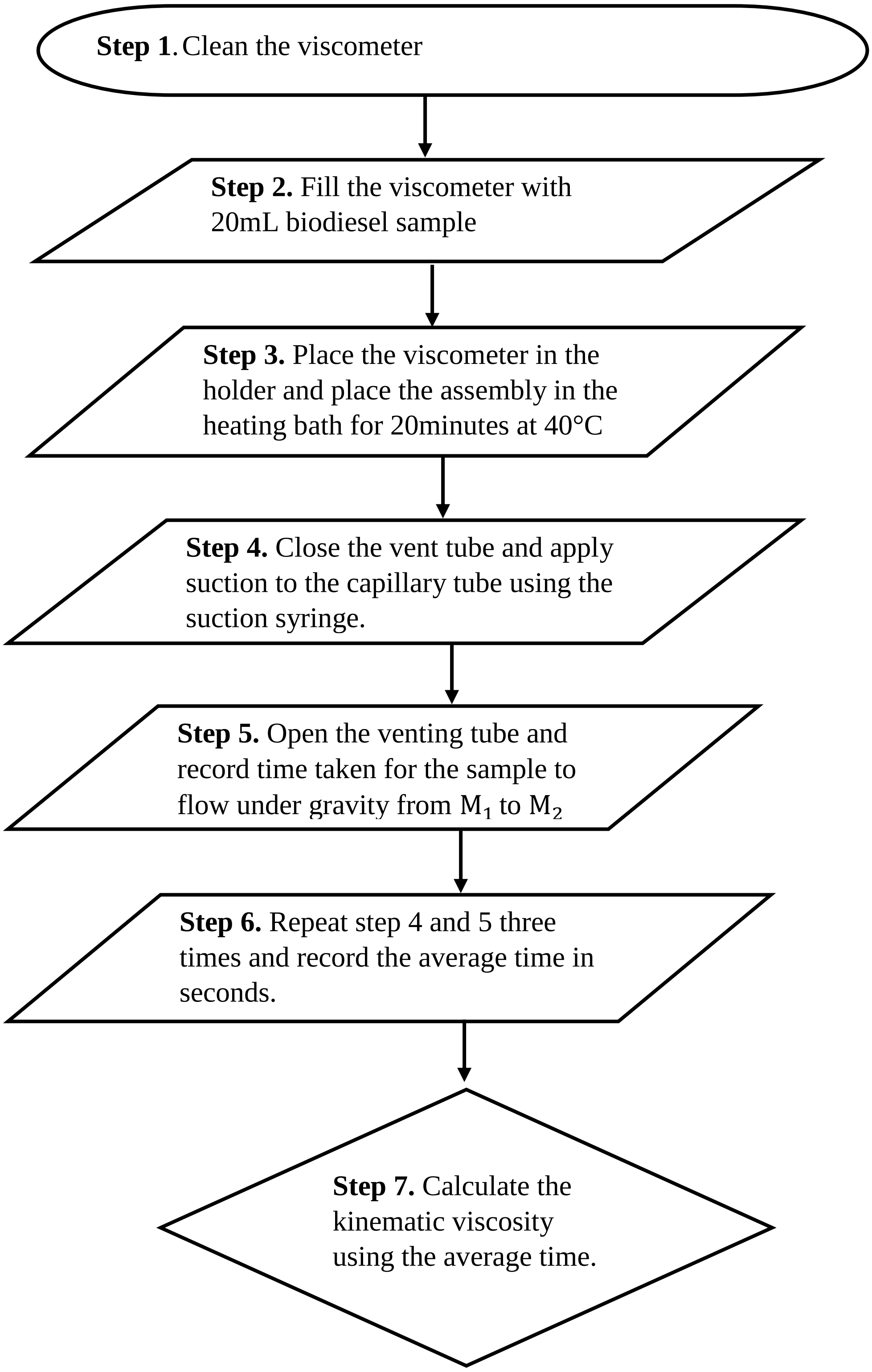


Figure 3.11: Calibration of Ubbelohde Viscometer



Step 1. Clean the viscometer

Step 2. Fill the viscometer with 20mL biodiesel sample

Step 3. Place the viscometer in the holder and place the assembly in the heating bath for 20minutes at 40°C

Step 4. Close the vent tube and apply suction to the capillary tube using the suction syringe.

Step 5. Open the venting tube and record time taken for the sample to flow under gravity from M_1 to M_2

Step 6. Repeat step 4 and 5 three times and record the average time in seconds.

Step 7. Calculate the kinematic viscosity using the average time.

Figure 3.12: Flow Chart procedure for Kinematic Viscosity Measurement

Table 3.4: kinematic viscosity calculation of WSFO biodiesel at 40°C (B95 K5)

Experimental runs	**Time (secs)**	**Constant K for type I viscometer**	**Kinetic energy correction factor (y)**	**Kinematic viscosity (v)** $(\boldsymbol{mm^2/s})$
1	375.56	0.009820	0.07	$v = K(t - y)$
2	375.19			
3	375.17			
	$t_{avg} = 375.30$			3.6848

3.5.3 Density Measurement

For the measurement of density, standards procedures was followed. The biodiesel density was measured with a device called pycnometer with a bulb of 99.693mL, the device is used in the determination of the fluid density. The excess fluid and air bubbles escape through the glass stopper.

The density of the biodiesel was measured using an electronic weighing balance, the density of the sample was measured at a stipulated temperature of 15°C the setup for the density measurement is shown in Figure 3.14.

To obtain the density of the samples, the pycnometer was cleaned properly with cleaning solution, rinsed with acetone and allowed to dry, the mass of the empty pycnometer was measured with an electronic weighing balance and recorded. The pycnometer was then filled completely with biodiesel sample and placed in a temperature controlled cooling bath at 15°C for 20minutes after which the mass was measured again with the electronic weighing balance.

For the measurement of density, the pycnometer empty mass was subtracted from the mass measured when filled with biodiesel and then divided by the volume of the pycnometer. Hence, Equation 3.2.

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

Where:

The density be given by ρ (kg/m^3)

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

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

V be taken as volume of the pycnometer

Figure 3.13: Setup of pycnometer in the cooling bath

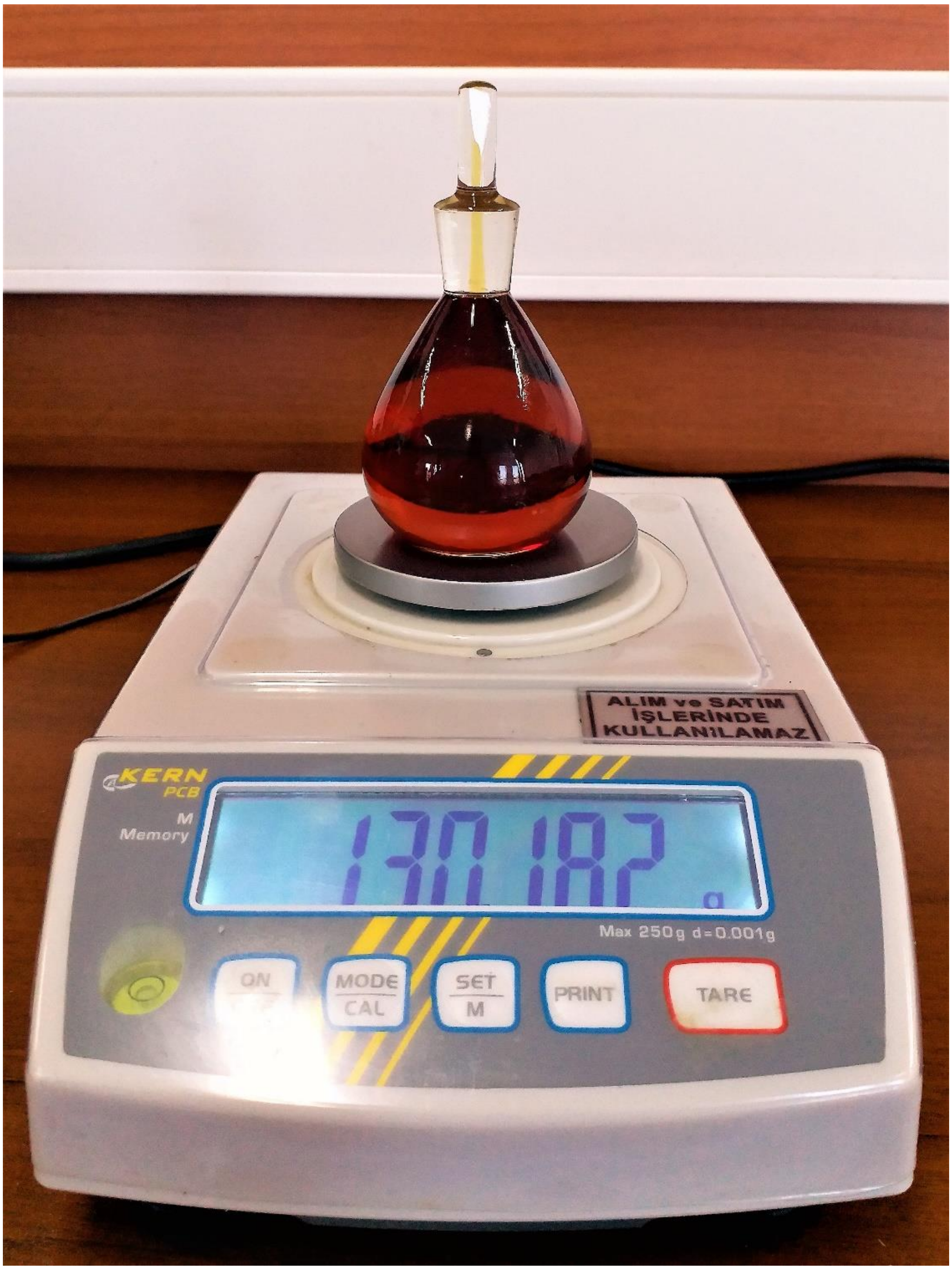
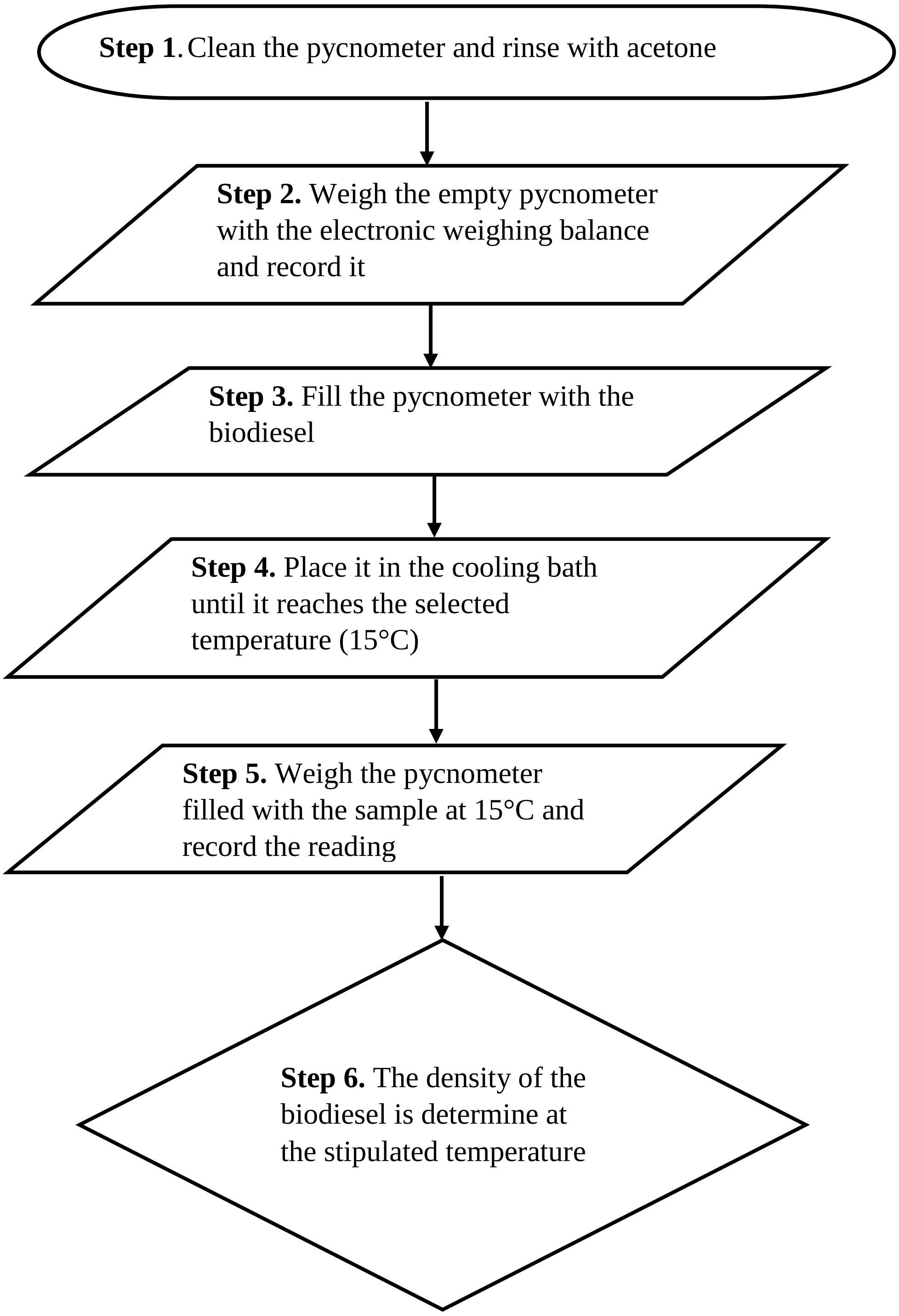


Figure 3.14: Experimental setup for density measurement



Step 1. Clean the pycnometer and rinse with acetone

Step 2. Weigh the empty pycnometer with the electronic weighing balance and record it

Step 3. Fill the pycnometer with the biodiesel

Step 4. Place it in the cooling bath until it reaches the selected temperature (15°C)

Step 5. Weigh the pycnometer filled with the sample at 15°C and record the reading

Step 6. The density of the biodiesel is determine at the stipulated temperature

Figure3.15: Flow Chart procedure for Density Measurement

3.5.4 Measurement of Cold Flow Properties

To obtain the cloud point (CP), pour point (PP) and cold filter plugging point (CFPP) for this study, the properties were examined for waste sunflower biodiesel blended with kerosene in different proportions of B90 K10 and B80 K20.

The samples were stored in 40°C temperature controlled oven and also and also room temperature for over 50days; the test was carried out as per American Standard Test Method, ASTM D2500 for cloud point, ASTM D6371-05 for cold filter plugging point and ASTM D97-2005 for pour point.

For accurate measurement of the cold flow properties of the sample, the experimental setup was built according to specifications given in the various ASTM standards above, the main component of the setup includes

- Data logger
- Cooling bath
- Refrigeration unit
- Glass of test jar
- Insulator (Styrofoam)
- Coil of compressor system
- Thermocouples
- Thermostat of the compressor system
- Coolant (Alcohol)

In measuring the cold flow properties of the samples, ethanol (Alcohol) was used to fill the bath as the coolant and was insulated properly to maintain the internal temperature of the bath and also to prevent heat transfer.

The temperature of the coolant was cooled down by the coil connected to the compressor that that was controlled using a thermostat which switches on and off in order to stabilize the temperature at the required temperature. For proper homogeneity of the ethanol (coolant) a stirrer was used to stir the cooling bath ethanol.

A cylinder jacket which is used in clamping the test jar was placed in the middle of the cooling bath. One T-type thermocouple was connected to the data logger and placed at the middle of the cooling bath to keep record of the temperature (T_o) of the coolant.

The test jaw was filled to the volume of 45mL and fitted into the cylinder jacket. Two T-type thermocouples for temperature readings were used to measure the temperature in the test jar as shown in Figure 3.15 below. Thermocouple T_1 was placed 3.5mm above the bottom of the test jar and thermocouple T_2 was placed 3mm below the top sample mark of the test jar.

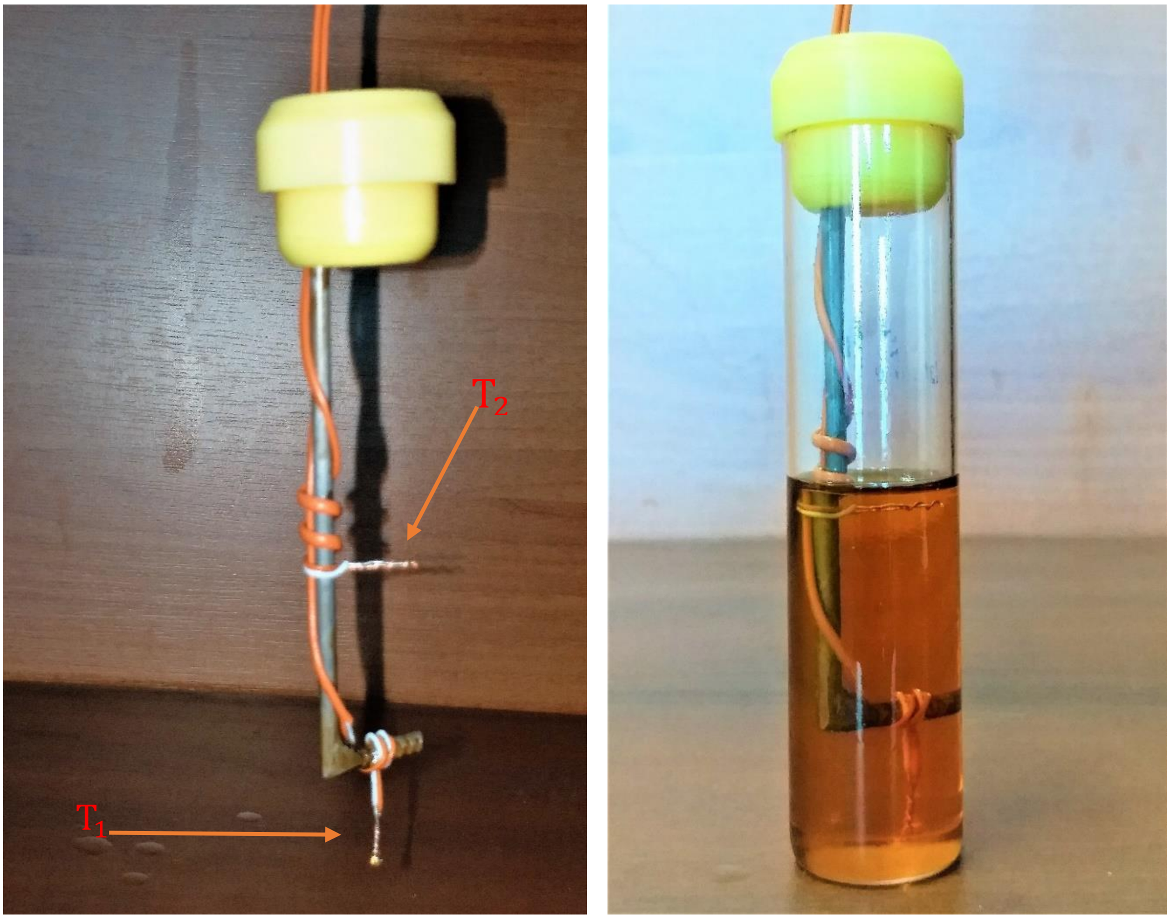


Figure 3.16: Glass of test jar and thermocouple labelling for cold flow properties

The test jar was isolated thermally from the jacket by cork support and stopper assembly. The thermocouple T_1 was used to measure the temperature at cloud point and T_2 was used to measure the temperature at pour point. Prior to the insertion of the test jar into the cylinder jacket of the cooling bath, the bath was cooled to -17°C, the test jar assembly containing the biodiesel sample was then immersed into the cylinder jacket in the cooling bath containing alcohol of -17°C.

To obtain the cloud point (CP), the sample in the test jar was inspected periodically at an interval of 1°C until a cloud of wax crystal first appeared in the sample under controlled temperature. The temperature reading of the thermocouple T_1 was recorded and the sample was allowed in the cooling bath to continue cooling until the biodiesel sample was no longer able to be poured as a result of gel formation, at this point the temperature of the thermocouple T_2 was recorded to be the pour point of the biodiesel.

3.5.5 Cooling Curve

For the cooling curve analysis, three T-type thermocouples for temperature reading were used to measure the temperature of the biodiesel sample in the test jar as shown in Figure 3.16. The three thermocouples were placed at the midpoint of the sample in the test jar. The thermocouplesT_1, T_2 and T_3 were placed at an equal distance from each other horizontally.

Before the data was collected the temperature of the cooling was brought down to -17°C and that of the biodiesel sample was heated to 67°C; the test jar assembly containing the biodiesel sample of 67°C was placed in the cylinder jacket in the cooling bath of -17°C. The temperature readings were recorded from the thermocouples using data logger; the readings were recorded and stored in the computer at an interval of one second using the data logger application as shown in the Figure 3.17, the sample was allowed in the cooling bath at -17°C for four hours without interruption. The data obtained from the software was taken for analysis using the Newtonian thermal analysis theorem.

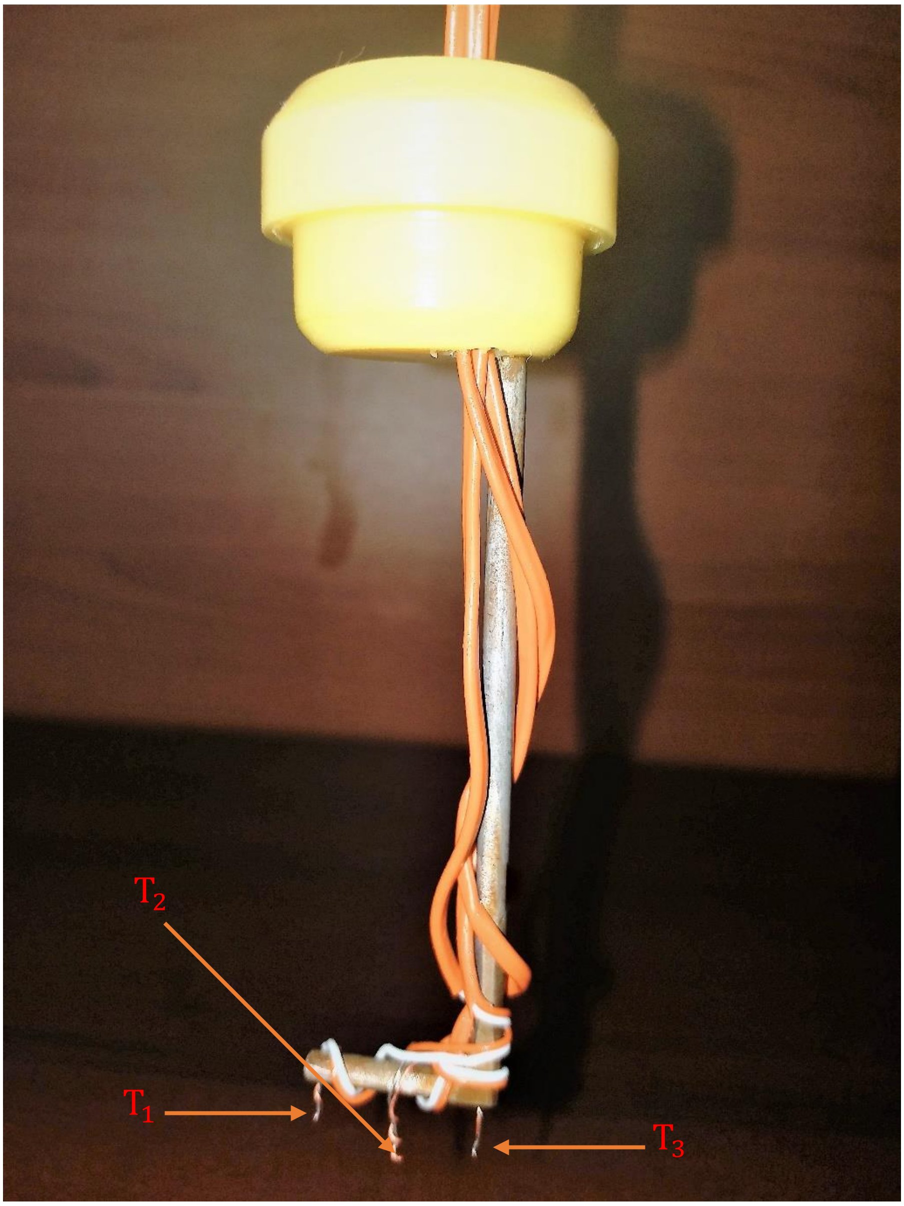
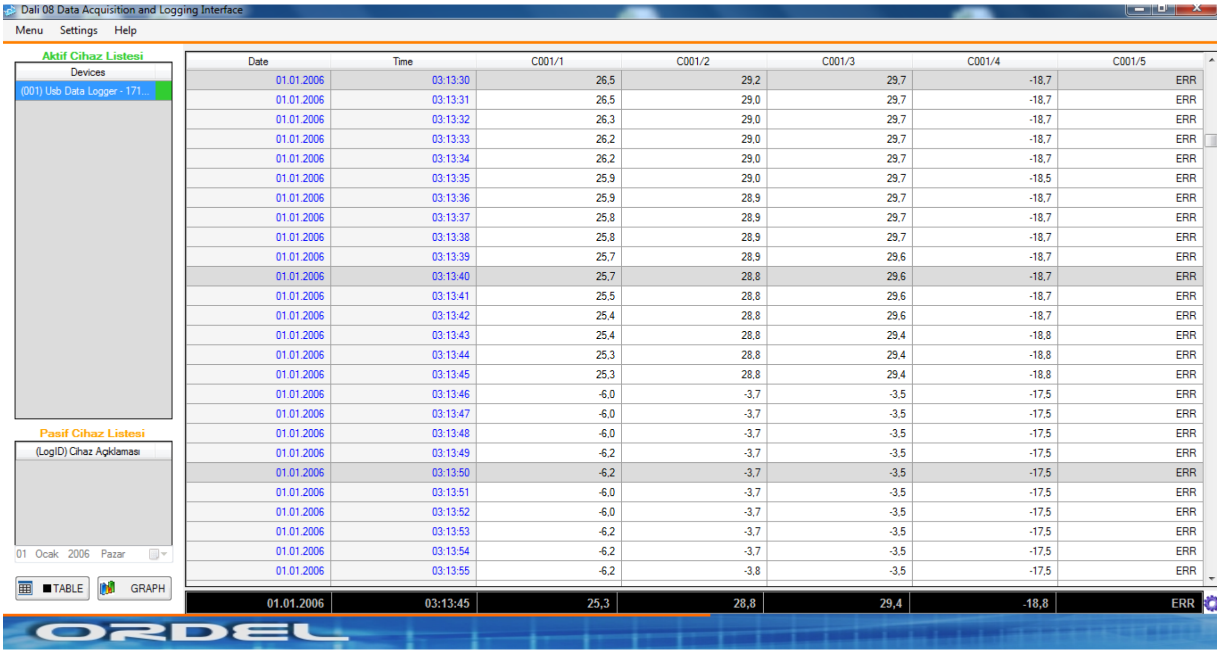


Figure 3.17: Thermocouple labelling for cooling curve



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	ERR
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	ERR
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	ERR
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 3.18: Data logger reading

3.6 Determination of Oxidation Stability and Acid Value

The oxidation stability and acid value of waste sunflower biodiesel blends B90 K10 and B80 K20 were estimated according to test method EN14112 and EN14104 respectively.

These tests were carried out on these sample after they were blended and stored for over a period of 40days at room temperature.

CHAPTER 4

RESULTS AND DISCUSSION

This chapter outline all the results obtained in this thesis and also gives detail analysis and explanation of the results obtained. The effects of the storage conditions were clearly shown on the graphs, it also display how the effects of the volume of kerosene was manifested on the biodiesel samples. The chapter further highlights the results of the cold flow properties (Cloud point, Pour point and Cold filter plugging point), it also compare the results of the cold flow properties obtained from the cooling curve using the Newtonian thermal analysis, the experimental results and the results obtained from the solid fraction. It also state the result of the acid number and the oxidation stability measured after the first 30days and subsequent 60days of the experiment.

4.1 RELIABILITY OF THE EXPERIMENTAL RESULT

To ensure the validity of the kinematic viscosity results, a repeatability test was carried out for each biodiesel sample; the measurement of the flow time was carried out repeatedly at a constant temperature of 40°C three times. The average flow time was recorded for the calculation of the kinematic viscosity, the essence of this is to reduce errors resulting from the time and temperature differences.

Relative density which is also another factor used in determining the efficiency of biodiesel blends, the measurement of the mass of the biodiesel sample was carried out twice at a constant temperature of 15°C and the average mass was taken to calculate the density of the biodiesel sample.

When concluded, we should be able to show that the experimental values obtained correlates with standards test methods which will establish that the experimental data falls within the range of the specified standards.

4.2 Effects of Storage Period on Kinematic Viscosity of Biodiesel Blends stored at ambient temperature

Table 4:1 shows the result of experimental data collected over a storage period of 90 days for waste sunflower biodiesel blended with kerosene at various proportion, from the plot in Figure 4.1, it is observed that the kinematic viscosity of the various blends increases with increase in storage period. However, there are fluctuations in the increment of these results; this is as a result of inconsistency in the storage temperature. The storage condition vary with the atmospheric (room) temperature for the biodiesel samples stored at ambient condition, this is due to instability in the weather condition which affects the ambient temperature.

Table 4.1: Kinematic viscosity (mm^2/s) of WSFO biodiesel over a period of 90 days at ambient temperature for various blends

DAYS	B95 K5	B90 K10	B85 K15	B80 K20
10	3.6848	3.4431	3.2682	3.0754
20	3.7151	3.4926	3.2817	3.112
30	3.7052	3.4928	3.2754	3.0755
40	3.7478	3.5374	3.3355	3.1527
50	3.7520	3.5485	3.3391	3.1865
60	3.7738	3.5525	3.3586	3.2245
70	3.8126	3.5772	3.3521	3.2122
80	3.8016	3.5976	3.3614	3.2314
90	3.8221	3.5934	3.3682	3.2413
SD	**0.049I96**	**0.051942**	**0.040155**	**0.066503**

The standard deviation measures how concentrated the data points are around the mean; the more concentrated the data point the smaller the standard deviation. Considering the various samples used for this plot, the standard deviation is 0.0491, 0.0519, 0.0401 and 0.0665 for B95K5, B90K10, B85K15 and B80K20 respectively and the mean is 3.7572, 3.5372, 3.3266 and 3.1679 respectively for the kinematic viscosity of the samples in ambient condition, this reflect that the data points are very much concentrated around the mean.

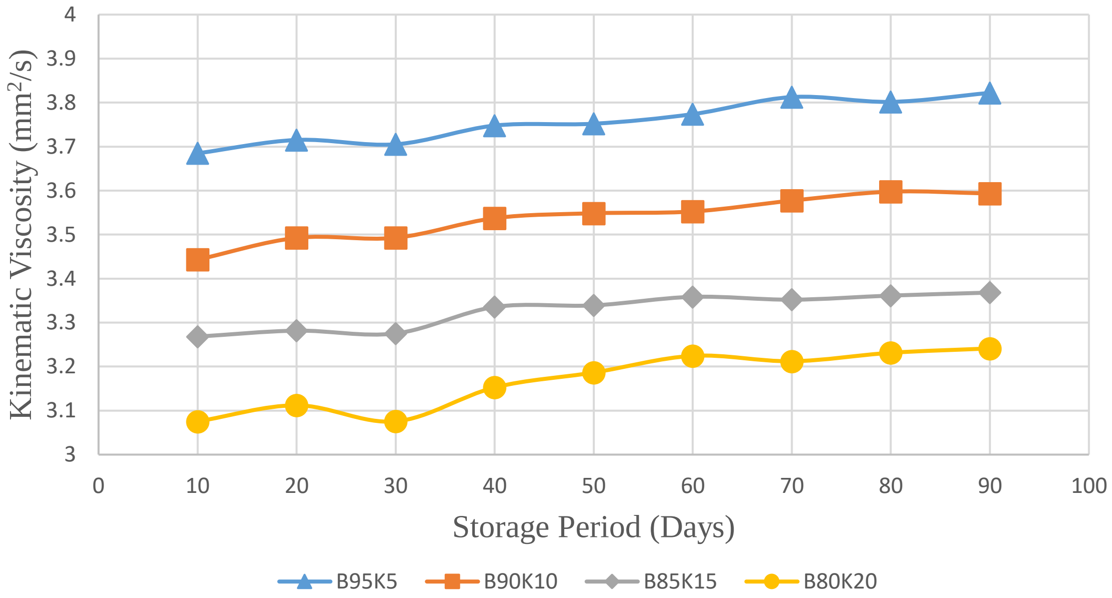


Figure 4.1: Kinematic Viscosity vs Storage Period at Ambient Temperature

4.3 Effects of Storage Period on Kinematic Viscosity of Biodiesel Blends stored at 40°C Oven

Table 4.2 display the results of the experimental data collected over a period of 90 days for waste sunflower biodiesel blended with kerosene and its kinematic viscosity measured at an interval of 10 days.

The sample was stored at 40°C temperature controlled oven. From Table 4.2 and Figure 4.2 it is observed that the kinematic viscosity increases with an increment in the storage period and also decreases with increase in the volume of kerosene. It is noticed as compared to the sample stored at ambient temperature that those stored at 40°C oven possess noticeably gradual increment in its kinematic viscosity. However, in Figure 4.2 it is observed that sample B90K10 and B80K20 behaves similarly in a different manner, in that its increment is not steady and it does not maintain a plateau, it shows a sharp fluctuations which could be interpreted as indicating that the attractive force between the biodiesel molecule became weaker which lead to decrease in the kinematic viscosity of the sample. The kinematic viscosity over the storage period increases by 6% for B90K10.

Table 4.2: Kinematic viscosity (mm^2/s) of WSFO biodiesel over a period of 90 days at temperature controlled oven of 40°C for various blends

DAYS	**B95K5**	**B90K10**	**B85K15**	**B80K20**
10	3.6481	3.3855	3.2143	3.0050
20	3.6889	3.5225	3.2741	3.1734
30	3.7112	3.4883	3.2916	3.2277
40	3.7864	3.5117	3.3114	3.2580
50	3.7498	3.5629	3.3477	3.2626
60	3.7500	3.6098	3.3507	3.2805
70	3.7580	3.5382	3.3349	3.2210
80	3.7965	3.5581	3.3893	3.2463
90	3.8113	3.5911	3.3971	3.2615
SD	**0.050539**	**0.066191**	**0.057805**	**0.084845**

The standard deviation measures how concentrated the data points are around the mean; the more concentrated the data point the smaller the standard deviation. Considering the various samples used for this plot, the standard deviation is 0.0505, 0.0661, 0.0578 and 0.0848 for B95K5, B90K10, B85K15 and B80K20 respectively and the mean is 3.7379, 3.5297, 3.3234 and 3.2152 respectively for the kinematic viscosity of the samples in oven condition, this reflect that the data points are very much concentrated around the mean.

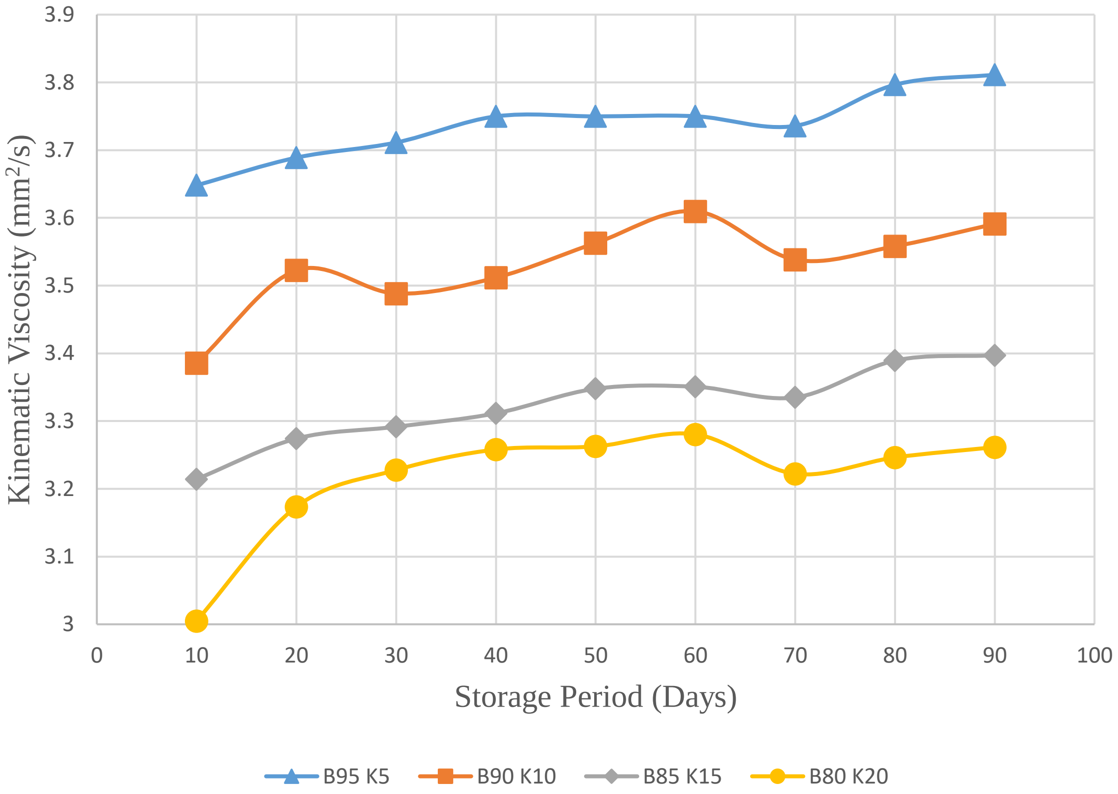


Figure 4.2: Kinematic Viscosity vs Storage Period at 40°C Oven

4.4 Graphical Comparism for kinematic viscosity (mm^2/s) of sample blends based on storage conditions (40°C controlled Oven and Ambient Temperature)

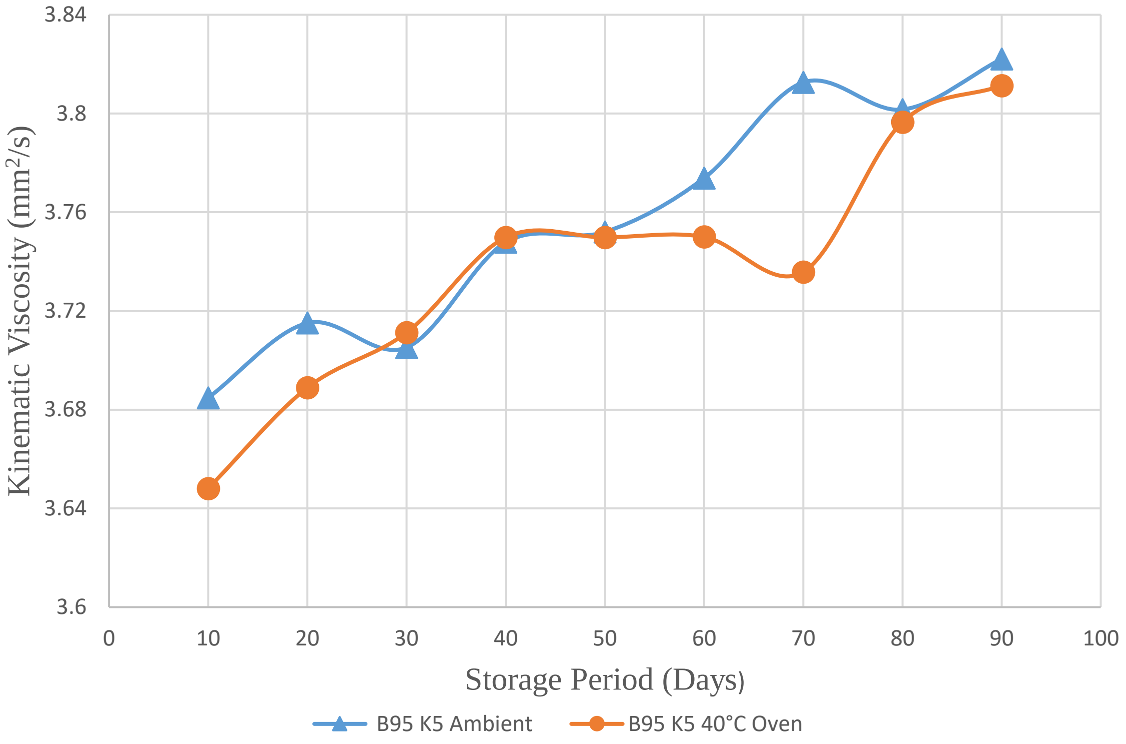


Figure 4.3: kinematic Viscosity vs Storage Period B95k5 (40°C Oven and Ambient Temperature)

From Figure 4.3, it is observed that there is no much difference in the kinematic viscosity between the B95K5 ambient and B95K5 oven sample in that both samples follows the same trend, this reflect that the storage condition does not necessarily affect the kinematic viscosity for B95K5 sample at 40°C oven and ambient. However, it has been noticed that kinematic viscosity of both sample at 40˚C temperature falls within the 1.9-6.0 mm^2/s range according to ASTM D445 standard.

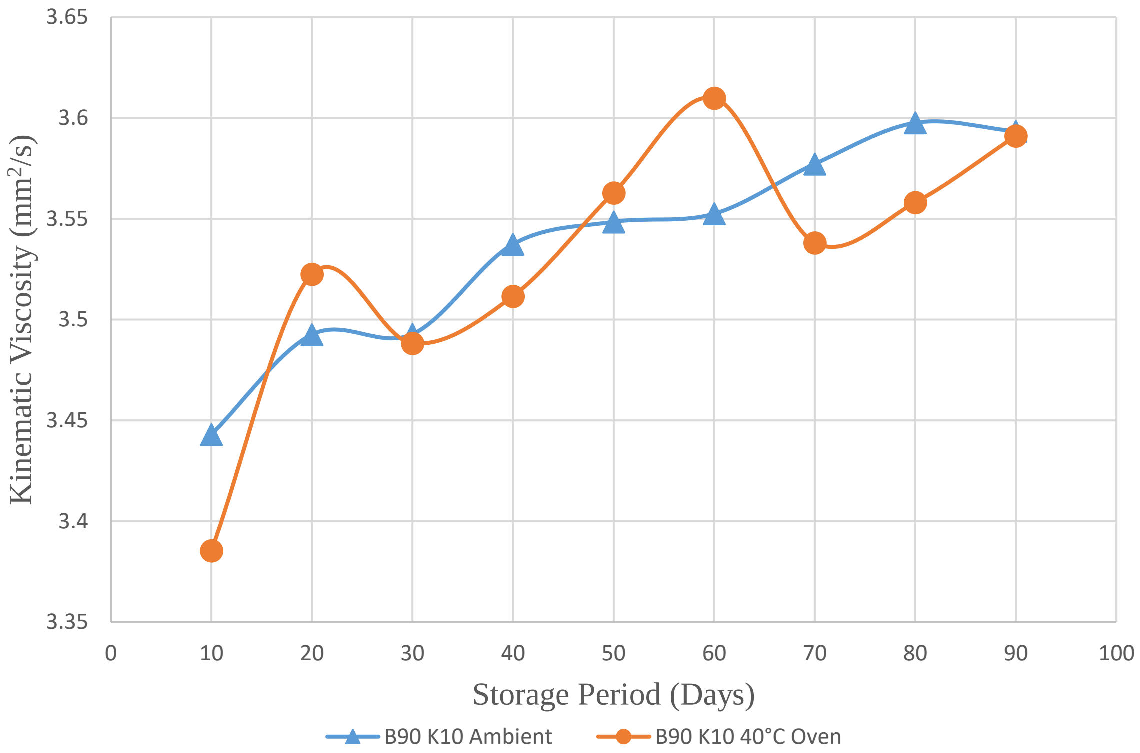


Figure 4.4: Kinematic Viscosity vs Storage Period B90K10 (40°C Oven and Ambient Temperature)

Similar to Figure 4.3, Figure 4.4 does not show a significant difference in B90K10 at ambient and oven sample, only that its kinematic viscosity is low when compared to B95K5 ambient and oven. This suggest that the storage condition does not necessarily affect the kinematic viscosity for B90K10 waste sunflower biodiesel within the 90 days. However, it has been noticed that kinematic viscosity of both sample at 40˚C temperature falls within the 1.9-6.0 mm^2/s range according to ASTM D445 standard.

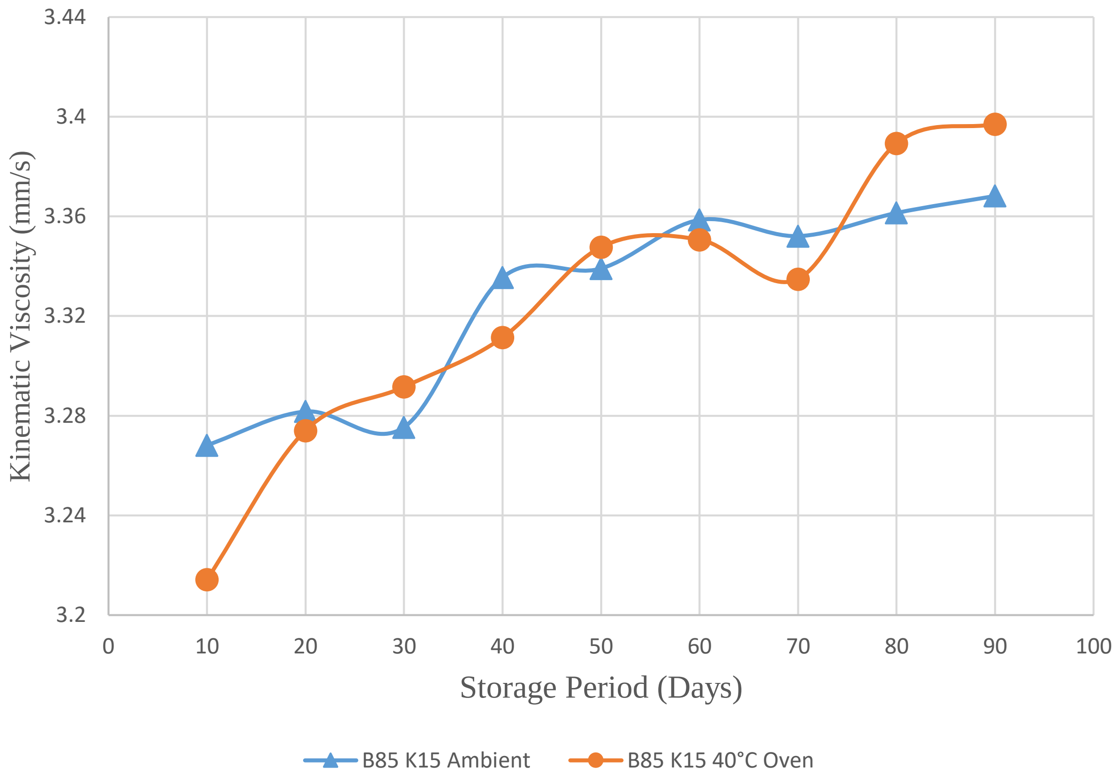


Figure 4.5: kinematic Viscosity vs Storage Period B85K15 (40°C Oven and Ambient Temperature)

Figure 4.5 follows the same trend with the Figure 4.3 and Figure 4.4, it does not reflect much difference in the storage conditions of the sample. This suggest that the storage condition does not necessarily affect the kinematic viscosity for B85K15 waste sunflower biodiesel within the 90 days. However, it has been noticed that kinematic viscosity of both sample at 40˚C temperature falls within the 1.9-6.0 mm^2/s range according to ASTM D445 standard.

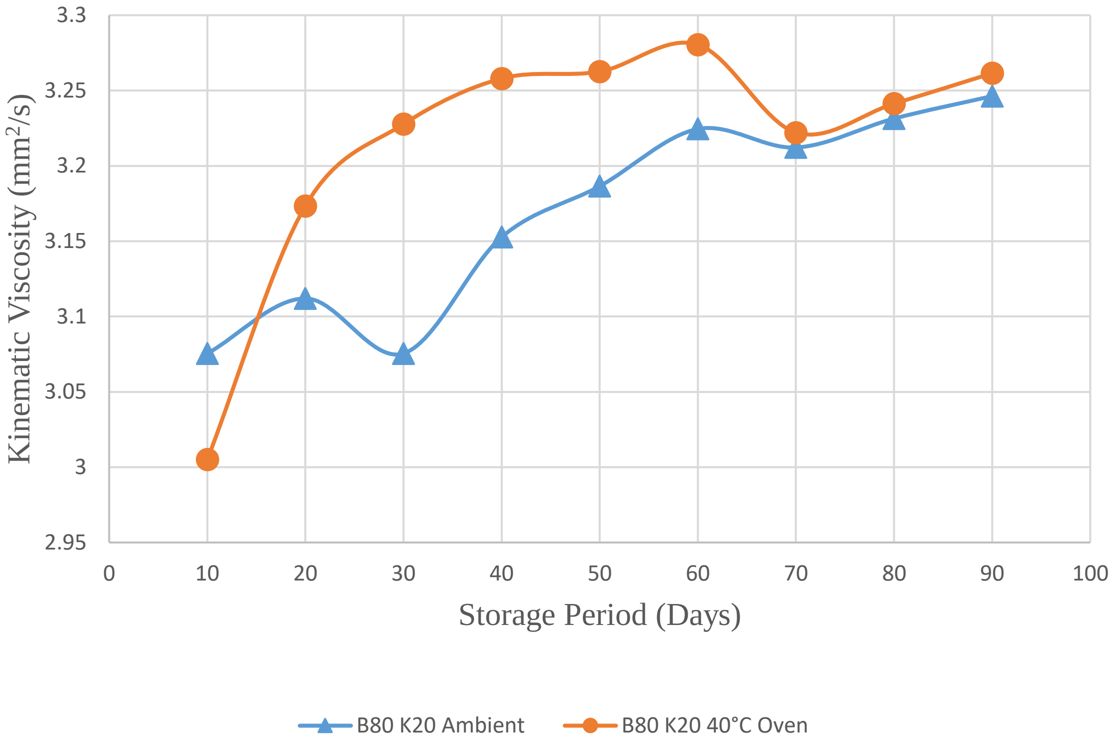


Figure 4.6: kinematic Viscosity vs Storage Period B80K20 (40°C Oven and Ambient Temperature)

From figure 4.6, it is observed that a huge difference is reflected on the samples in ambient and oven condition for B80K20, this is as result of increase in the volume of kerosene which reduces the kinematic viscosity and making the temperature effect of the sample in the oven to be felt. The increase in kerosene volume causes the intermolecular forces in the sample to be weaker thereby reducing the kinematic viscosity and causing any temperature different to be significant. However, it has been noticed that kinematic viscosity of both sample at 40˚C temperature falls within the 1.9-6.0 mm^2/s range according to ASTM D445 standard.

4.5 Effects of Storage Period on Density of Biodiesel Blends stored at 40°C Controlled Oven

Table 4.3 shows a variation in density of waste sunflower biodiesel blends stored over a period of 90 days and measured at 15°C after every 10 days in a temperature controlled oven of 40°C.

It is observed that the density increases with an increment in storage period for the first 50 to 60 days and then tends to stay constant as a result of oxidation process causing increase in polymer and fatty acid chains. It is noticed as compared to the kinematic viscosity that the density of the sample decreases with an increase in the proportion of the blend as depicted in Table 4.3 and Figure 4.7 shown in sample B95K10 and B80K20.

Storing the sample at 40°C causes also a fluctuating increment in the biodiesel sample as displayed by the sinusoidal behavior of the plot in Figure 4.7.

Table 4.3: Density (kg/m^3) of WSFO biodiesel over a period of 90 days at temperature Controlled oven of 40°C for various blends

DAYS	B95K5	B90K10	B85K15	B80K20
10	871.345	868.255	865.456	864.092
20	872.318	867.874	864.835	862.648
30	873.571	870.492	865.196	864.493
40	874.023	872.923	867.764	867.714
50	875.588	873.751	867.801	867.714
60	875.828	872.428	868.205	868.136
70	876.014	873.415	868.315	868.158
80	876.132	873.584	868.923	868.211
90	876.193	873.963	868.974	868.438
SD	**1.8215**	**2.3873**	**1.6450**	**2.2039**

The standard deviation measures how concentrated the data points are around the mean; the more concentrated the data point the smaller the standard deviation. Considering the various samples used for this plot, the standard deviation is 1.8215, 2.3873, 1.6450 and 2.2039 for B95K5, B90K10, B85K15 and B80K20 respectively and the mean is 874.55, 871.85, 867.27 and 866.57 respectively for the density of the samples in oven condition, this reflect that the data points are very much concentrated around the mean.

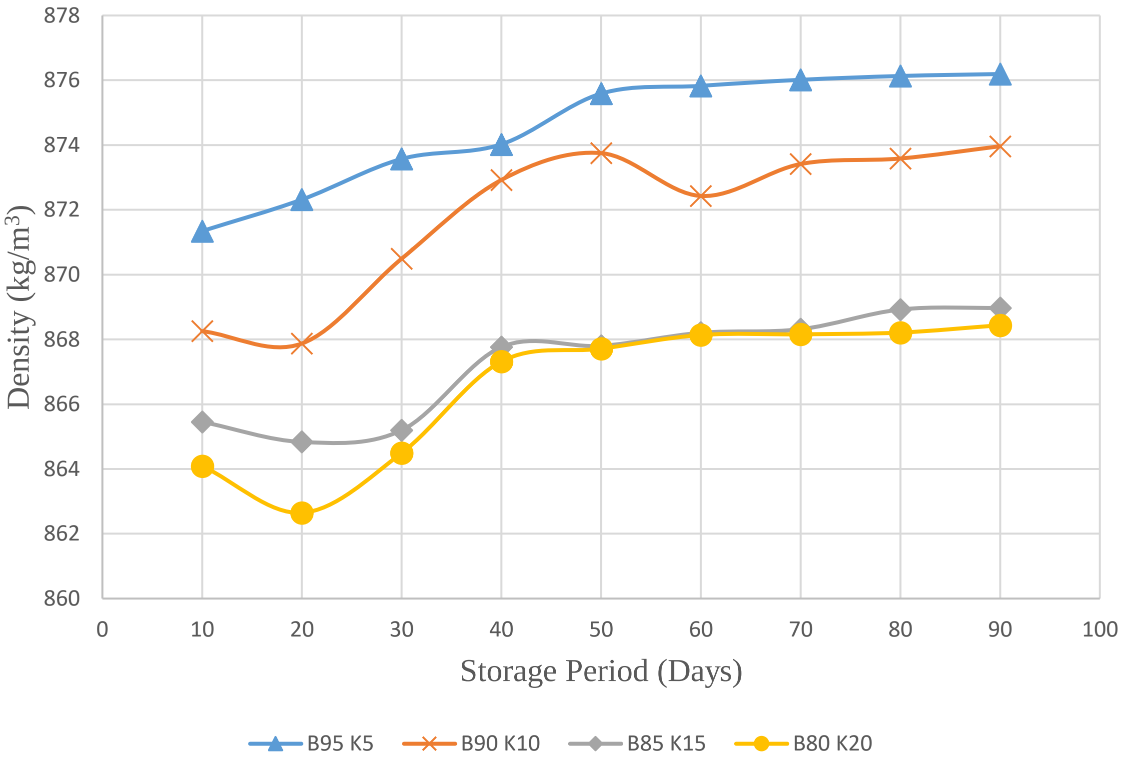


Figure 4.7: Density vs Storage Period at 40°C Oven

4.6 Effects of Storage Period on Density of Biodiesel Blends stored at ambient temperature

The density of the waste sunflower is affected by the condition in which it is stored, this is shown the plot in Figure 4.8 and Table 4.4. The density was measured at 15°C; it is observed that the density increases as the storage period increases for the first 40 and 50 days and tend to stay constant for the remaining period as in the case of the oven sample, moreover, there are some irregular changes in the behavior of the sample and this is caused by the variation in the atmospheric temperature. The room (ambient) condition vary from day to day depending on the weather condition of that period, this causes the density not to behave in a regular pattern,

However, the increase in the proportion of the kerosene in the biodiesel blends sample also decreases the density of the waste sunflower biodiesel. This is observed in Table 4.4 as shown below.

Table 4.4: Density (kg/m^3) of WSFO biodiesel over a period of 90 days at ambient temperature for various blends

DAYS	B95 K5	B90 K10	B85 K15	B80 K20
10	871.144	867.553	863.517	858.004
20	872.959	869.589	864.554	859.899
30	872.859	869.088	865.276	861.244
40	873.431	870.843	867.964	863.751
50	874.625	872.644	869.544	863.521
60	875.974	873.682	869.619	864.253
70	875.314	873.721	869.783	864.621
80	875.528	873.533	870.317	864.552
90	875.300	873.948	870.113	864.235
SD	**1.6117**	**2.41042**	**2.6744**	**2.3895**

The standard deviation measures how concentrated the data points are around the mean; the more concentrated the data point the smaller the standard deviation. Considering the various samples used for this plot, the standard deviation is 1.6116, 2.4102, 2.6744 and 2.3895 for B95K5, B90K10, B85K15 and B80K20 respectively and the mean is 874.12, 871.62, 867.85 and 862.67 respectively for the density of the samples in ambient condition, this reflect that the data points are very much concentrated around the mean.

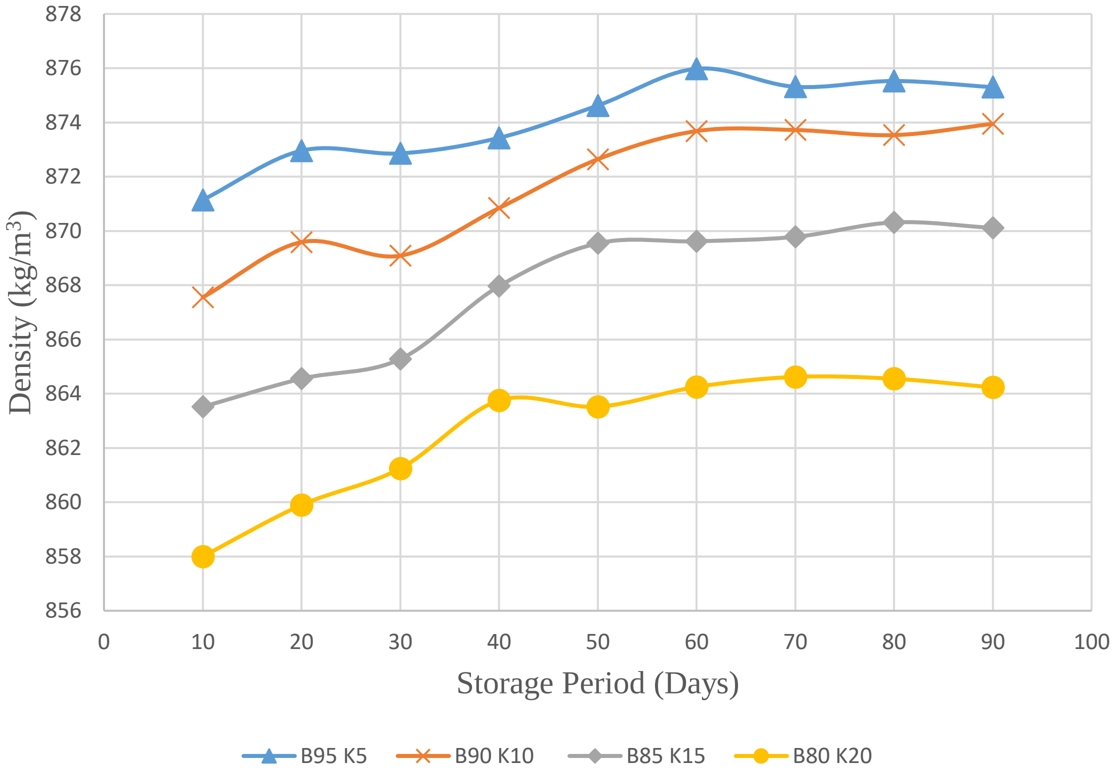


Figure 4.8: Density vs Storage Period at Ambient Condition

4.7 Graphical Comparism for the density (kg/m^3) of sample blends based on storage conditions (40°C Controlled Oven and Ambient Temperature)

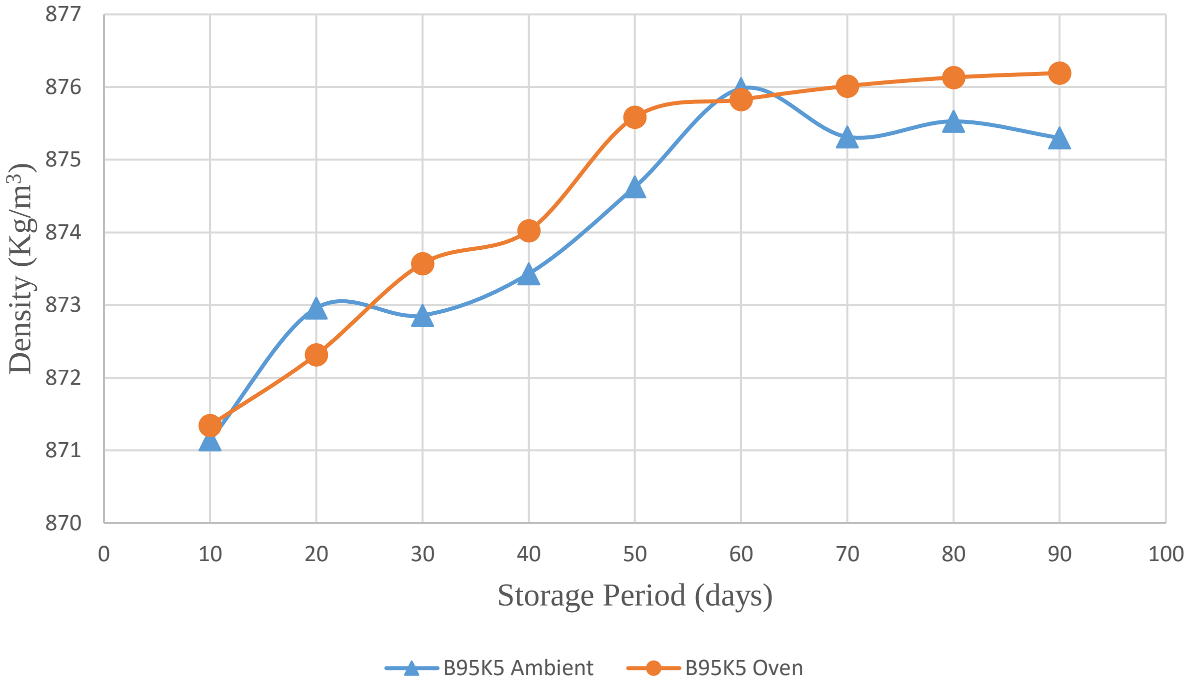


Figure 4.9: Density vs Storage Period B95K5 (40°C Oven and Ambient Temperature)

From Figure 4.9, it is observed that there is no much difference in the density between the B95K5 ambient and B95K5 oven sample in that both samples follows the same trend, this reflect that the storage condition does not necessary affect the density for B95K5 sample at 40°C oven and ambient within the 90 days.

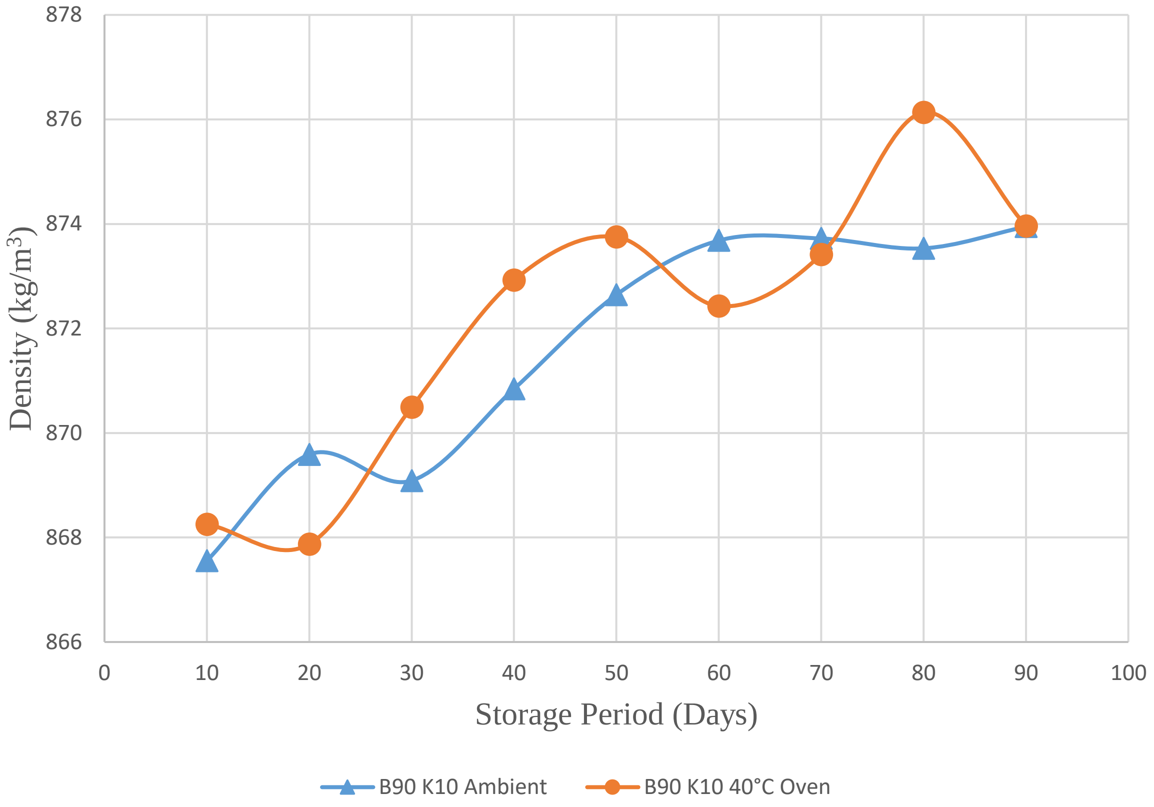


Figure 4.10: Density vs Storage Period B90K10 (40°C Oven and Ambient Temperature)

Similar to Figure 4.9, Figure 4.10 does not show a significant difference in B90K10 at ambient and oven sample, only that its kinematic viscosity is low when compared to B95K5 ambient and oven. This suggest that the storage condition does not necessarily affect the density for B90K10 waste sunflower biodiesel within the 90 days.

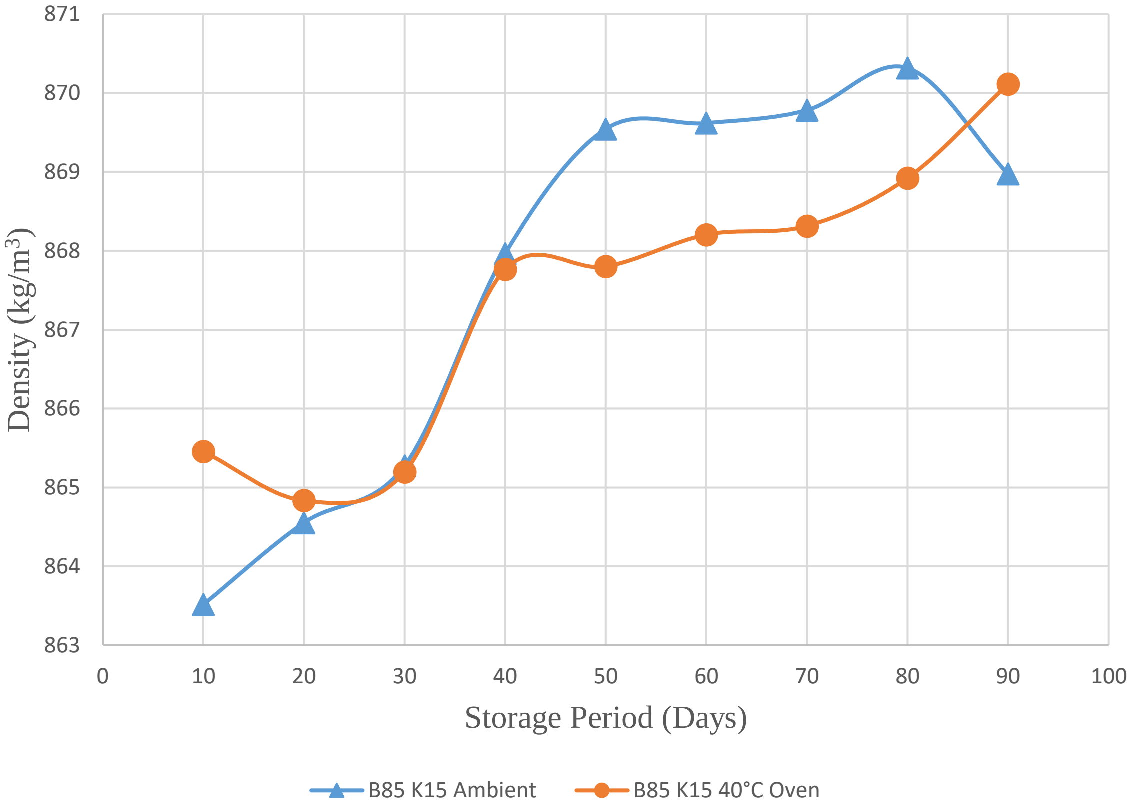


Figure 4.11: Density vs Storage Period B85K15 (40°C Oven and Ambient Temperature)

Figure 4.11 follows the same trend with the Figure 4.10 and Figure 4.9, it does not reflect much difference in the storage conditions of the sample. This suggest that the storage condition does not necessarily affect the density for B85K15 waste sunflower biodiesel within the 90 days.

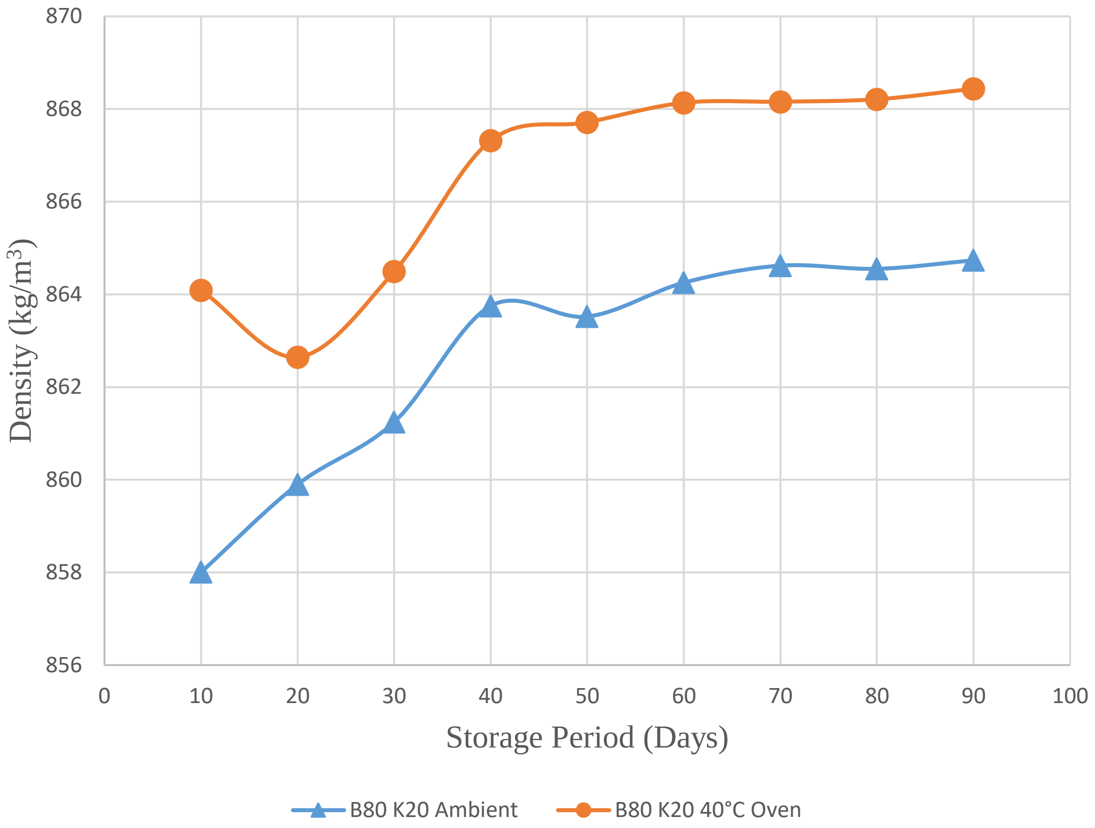


Figure 4.12: Density vs Storage Period B80K20 (40°C Oven and Ambient Temperature)

From Figure 4.12, it is observed that the density of the sample in the 40°C oven tends to be higher than that stored at ambient for B80K20, this is because of thermal instability of the oven sample brought about by relative increase in the volume of kerosene which causes the rate of oxidation at higher temperature to increase which in turns increases the weight of the biodiesel in 40°C oven due to formation of insoluble, this causes the sample stored in the oven to exhibit higher density as compared to that stored at ambient temperature.

4.8 Cold flow properties of waste sunflower biodiesel blends

The cold flow properties of the waste sunflower biodiesel was determine according to ASTM Standards, for the result below the cold flow properties was done according to ASTM D97-2005, ASTM D2500 and ASTM D6371-05. Table4.5 and Table 4.6 shows the results for various samples of the waste sunflower biodiesel blends at different temperature conditions.

It is observed that there was a significant different in the kinematic viscosity before freezing and after freezing of the sample. The kinematic viscosity for B90K10 increased by 11% after freezing and the density increased by 0.07%; for B80 K20 the kinematic viscosity increased by 1.75% whereas the density increased by 0.22% for all the samples stored at ambient conditions.

For samples stored at 40°C controlled oven, it is observed that for B90K10 the kinematic viscosity increased by 2.2% after freezing and the density increased by 0.25% also for B80K20 the kinematic viscosity increased by 1.28%; and the density increased by 0.08%.

From the results it’s shown that the storage condition actually affect the biodiesel blends, it’s also observed that the cloud point (CP) and pour point (PP) are affected by the blend proportion. Increase in blend proportion increases the cold flow properties.

Heating the sample to 67°C before freezing the biodiesel promotes polymerization and produces higher molar mass compounds which causes an increase in the oxidation process of the biodiesel sample, this increases the weight of the biodiesel due to rapid formation of insoluble crystals. As such increases the kinematic viscosity and density of the sample after freezing. This is demonstrated in Table 4.5 and Table 4.6.

Table 4.5: Cold Flow properties Data for Waste Sunflower Biodiesel Stored at Ambient Temperature

	Cloud point °C	Pour point °C	Kinematic Viscosity (Before Cooling) mm^2/s	Kinematic Viscosity (After Cooling) mm^2/s	Density (Before Cooling) kg/m^3	Density (After Cooling) kg/m^3
B90 K10	-5.0	-7.4	3.4857	3.8721	868.471	869.147
B80 K20	-6.7	-8.9	3.1639	3.2193	859.289	861.236

Table 4.6: Cold Flow Properties Data for Waste Sunflower Biodiesel Stored at 40°C Controlled Oven Temperature

	Cloud point °C	Pour point °C	Kinematic Viscosity (Before Cooling) mm^2/s	Kinematic Viscosity (After Cooling) mm^2/s	Density (Before Cooling) kg/m^3	Density (After Cooling) kg/m^3
B90 K10	-5.5	-8.1	3.5629	3.6406	872.923	875.188
B80 K20	-6.3	-9.8	3.2626	3.3046	867.318	867.994

4.9 Oxidation Stability and Acid Value

The oxidation stability and acid value was carried out in Tubitak Marmara Research Centre Kocaeli Province Turkey. The test was carried out on B80K20 and B90 K10, the test was carried out according to standards EN1404 and EN14112 as shown on Table 4.6 and Table 4.7 below. It is observed that the oxidation stability increased with storage period.

Table 4.7: Oxidation Stability and Acid Value for B80 K20

TEST DESCRIPTION UNITS	TEST METHOD	TEST RESULT (MARCH)	TEST RESULT (MAY)
Acid Number	EN14104	0.77	
(mgKOH/g)	ASTM D664		0.51
Oxidation Stability			
(hours)	EN14112	2.26	2.45

Table 4.8: Oxidation Stability and Acid Value for B90 K10

TEST DESCRIPTION UNITS	TEST METHOD	TEST RESULT (MARCH)	TEST RESULT (MAY)
Acid Number	EN14104	0.85	
(mgKOH/g)	ASTM D664		0.51
Oxidation Stability			
(Hours)	EN14112	1.90	2.50

4.10 Cooling Curve Analysis

The cold flow properties of the biodiesel sample was conducted according to standard. The plot in Figure 4.14. shows the cooling curve, the cooling rate, cooling bath temperature and the Newtonian (Zn) for biodiesel sample B80K20 ambient, in determining the cold flow properties (CP and PP) a plateau is observed on the cooling curve (T vs t) the CP (-5.9) is located just before the start of the plateau which correspond to the shape change in the slope of the graph and is related with the nucleation of the solid crystals in the freezing biodiesel sample. The PP (-9.4) is taking into account as the end of the plateau region after which there is a continuous decline in temperature, this is shown on the curve in Figure 4:14.

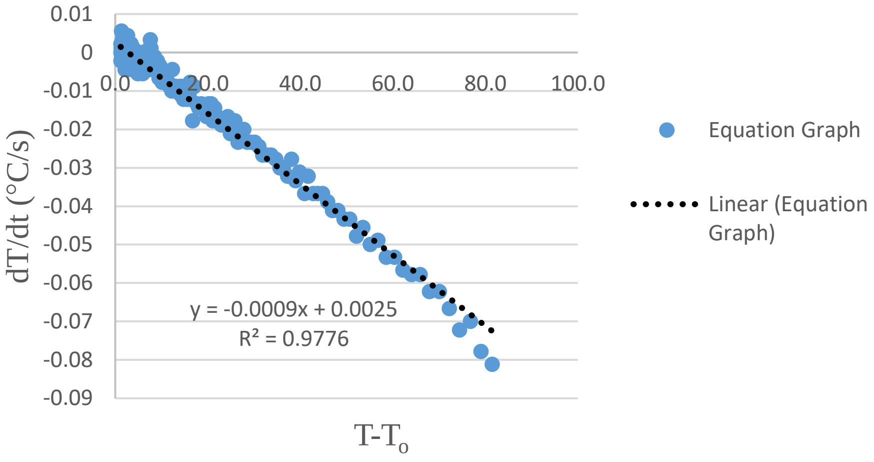


Figure 4.13: dT/dt vs T-To B80K20 Ambient Curve

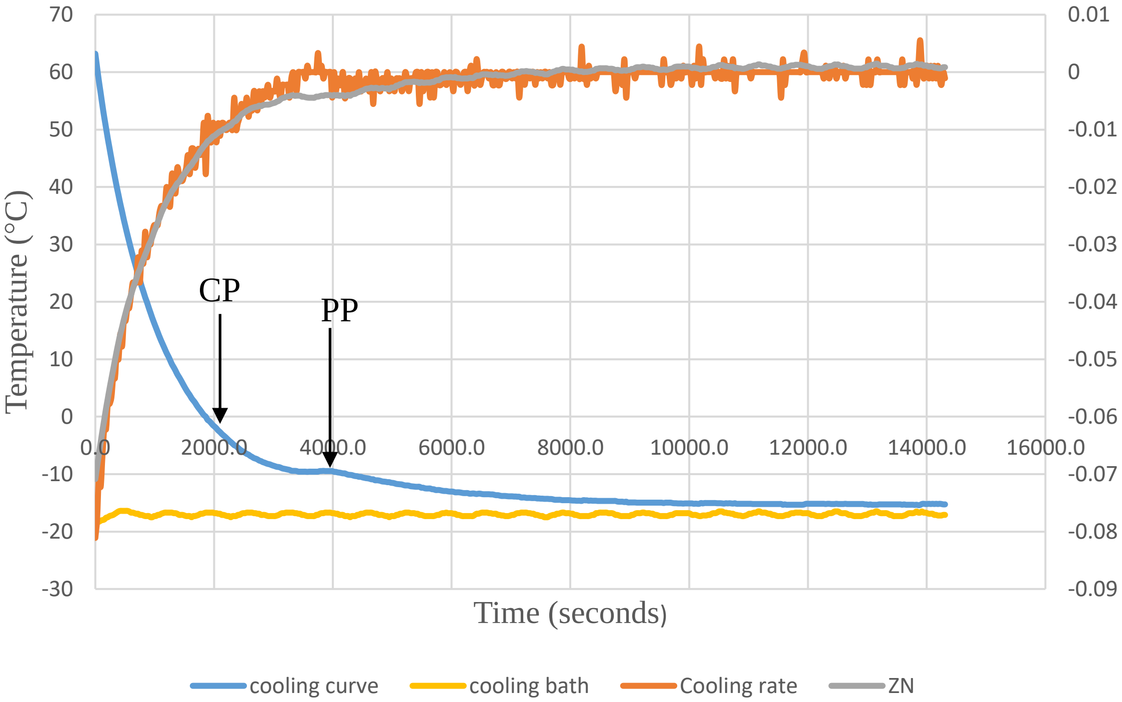


Figure 4.14: Cooling curve and Newtonian Zero Curve for B80K20 Ambient Sample

4.11 Cooling curve for B90K10 Ambient

The plot in Figure 4.16. shows the cooling curve, the cooling rate, cooling bath temperature and the Newtonian (Zn) for biodiesel sample B80K20 ambient, in determining the cold flow properties (CP and PP) a plateau is observed on the cooling curve (T vs t) the CP (-5.8) is located just before the start of the plateau which correspond to the shape change in the slope of the graph and is related with the nucleation of the solid crystals in the freezing biodiesel sample. The PP (-8.1) is considered as the end of the plateau region after which there is a continuous decline in temperature, this is shown on the curve in Figure 4:16.

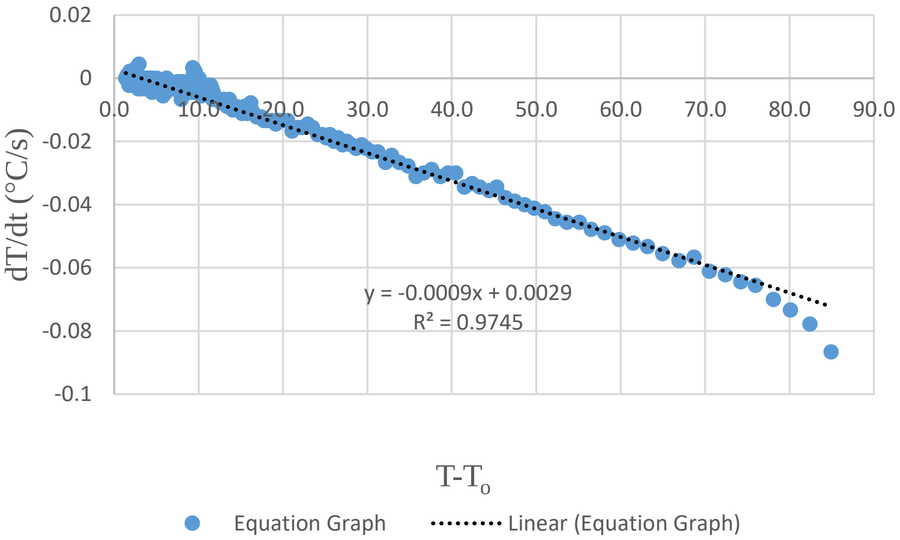


Figure 4.15: dT/dt vs T-T_o B90K10 Ambient Curve

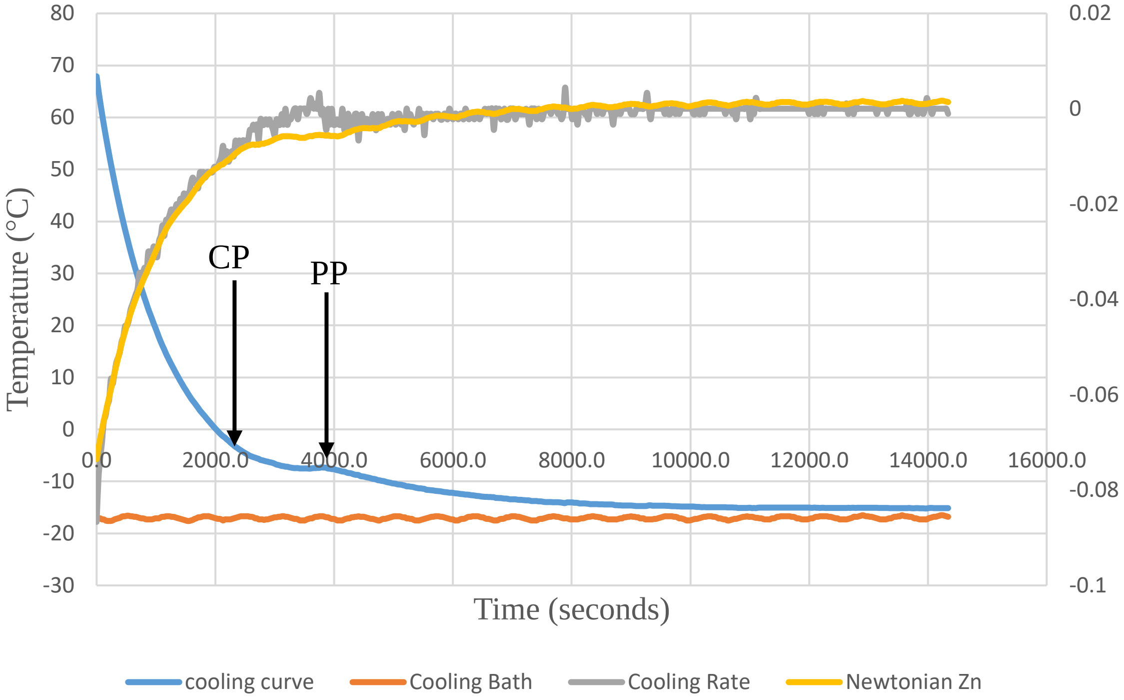


Figure 4.16: Cooling curve and Newtonian Zero Curve for B90K10 Ambient Sample

4.12 Cooling curve for B80K20 Oven

The plot in Figure 4.17. shows the cooling curve, the cooling rate, cooling bath temperature and the Newtonian (Zn) for biodiesel sample B80K20 ambient, in determining the cold flow properties (CP and PP) a plateau is observed on the cooling curve (T vs t) the CP (-5.5) is located just before the start of the plateau which correspond to the shape change in the slope of the graph and is related with the nucleation of the solid crystals in the freezing biodiesel sample. The PP (-9.2) is considered as the end of the plateau region after which there is a continuous decline in temperature, this is shown on the curve in Figure 4:17.

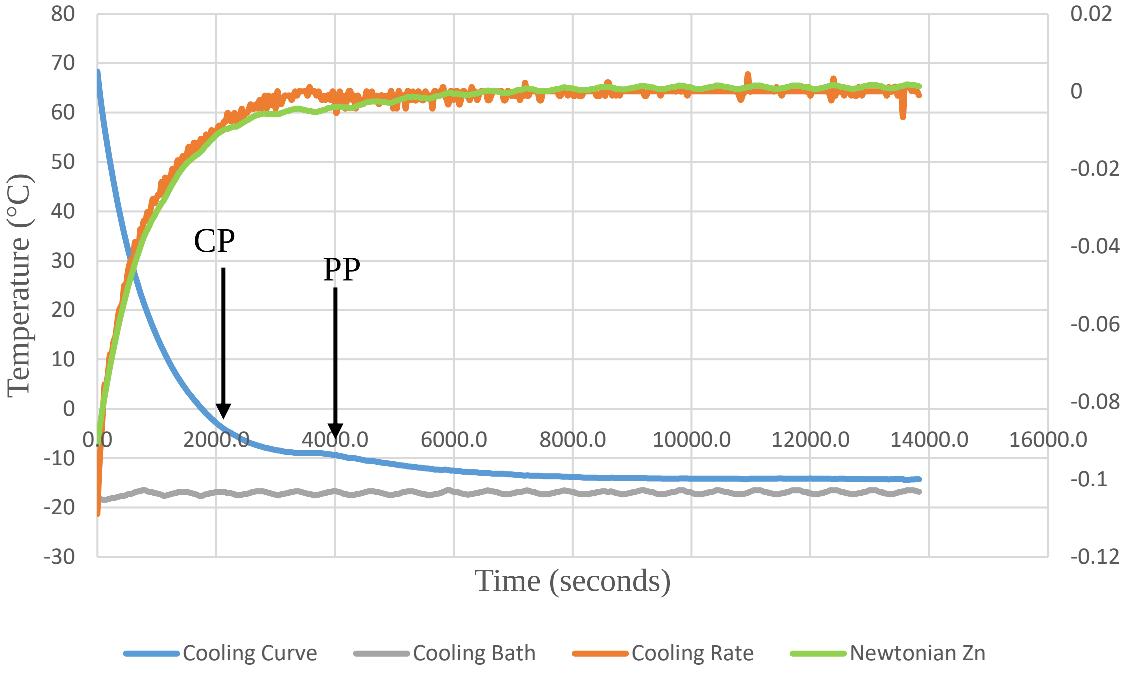


Figure 4.17: Cooling curve and Newtonian Zero Curve for B80K20 Oven sample

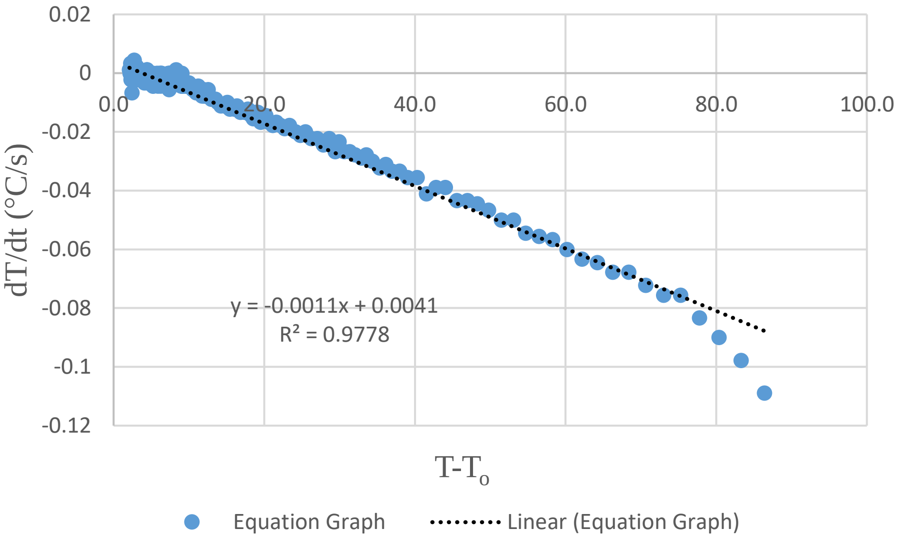


Figure 4.18: dT/dt vs T-T_o B80K20 Oven Curve

4.13 Cooling curve for B90K10 Oven

The plot in Figure 4.19. shows the cooling curve, the cooling rate, cooling bath temperature and the Newtonian (Zn) for biodiesel sample B80K20 ambient, in determining the cold flow properties (CP and PP) a plateau is observed on the cooling curve (T vs t) the CP (-5.9) is located just before the start of the plateau which correspond to the shape change in the slope of the graph and is related with the nucleation of the solid crystals in the freezing biodiesel sample. The PP (-9.4) is considered as the end of the plateau region after which there is a continuous decline in temperature, this is shown on the curve in Figure 4:19.

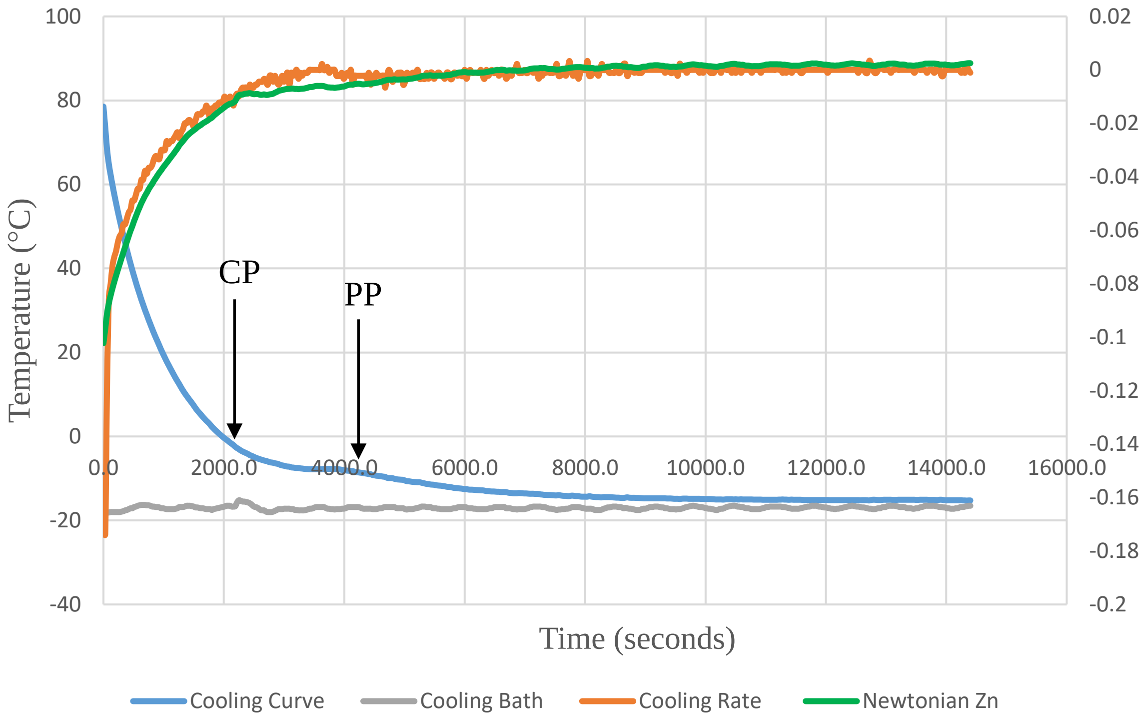


Figure 4.19: Cooling curve and Newtonian Zero Curve for B90K10 Oven sample

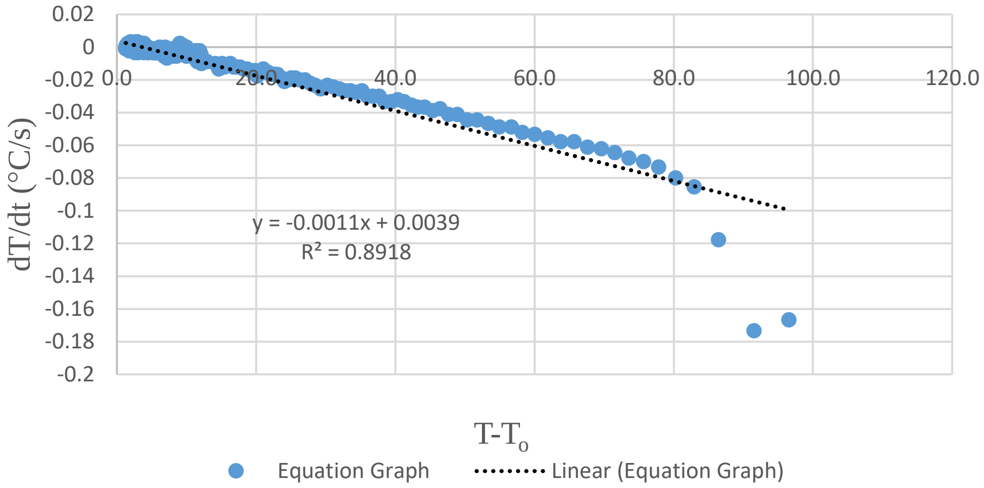


Figure 4.20: dT/dt vs T-T_o B90K10 Oven Curve

4:14 Solid Fraction

The solid fraction which is the amount of particulate matter in the fuel during cooling is got from the plot showing the variation of the solid fraction during freezing, the points on the vertical axis of the graph that corresponds to the respective cold flow properties (CP, PP and CFPP) gives the solid fraction of the sample.

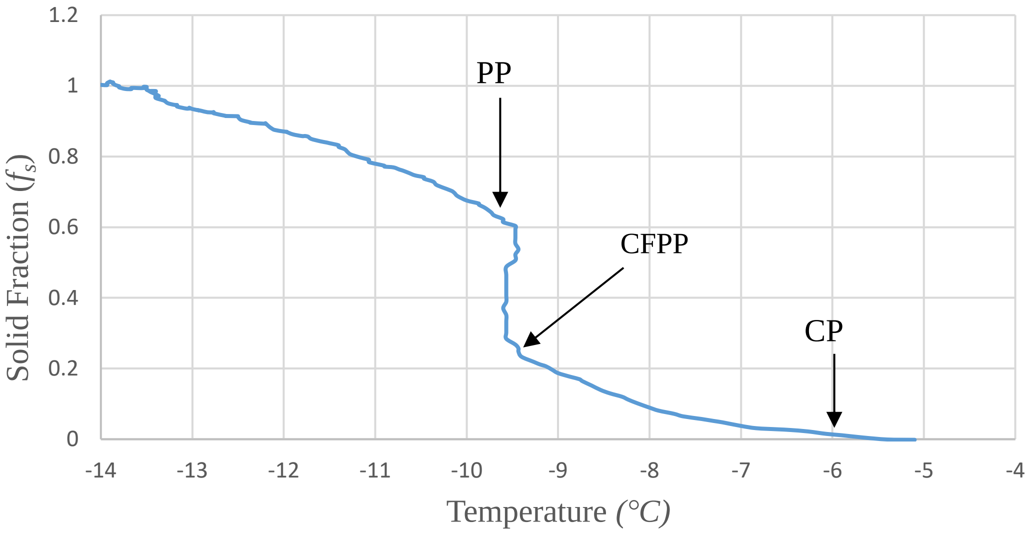


Figure 4.21: Variation of solid fraction during freezing of B80K20 Ambient

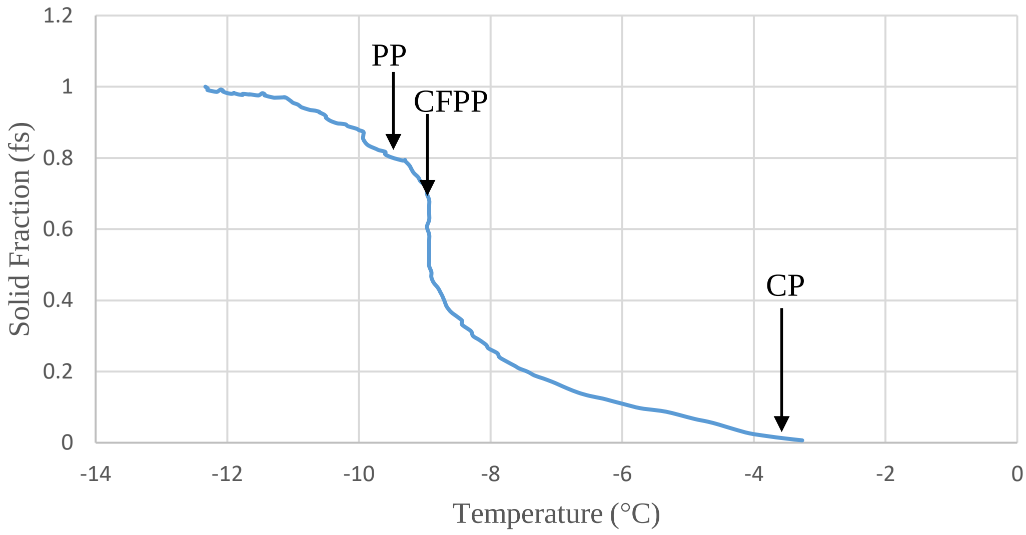


Figure 4:22: Variation of solid fraction during freezing of B80K20 Oven

Table 4.9: Cold Flow Properties for experimental value, cooling curve Analysis values and Solid fraction Values

SAMPLE	EXP- CP (°C) ASTM D2500	EXP-PP (°C) ASTM D97-05	CCA-CP (°C)	CCA-PP (°C)	Solid Fraction (f_s)		
					CP	PP	CFPP
B90K10 Ambient	-5.0	-7.4	-5.8	-8.1	-3.3	-7.5	-6.5
B90K10 Oven	-5.5	-8.1	-5.1	-8.6	-3.5	-7.7	-7.4
B80K20 Ambient	-6.7	-8.9	-5.9	-9.4	-5.7	-9.8	-9.3
B80K20 Oven	-6.3	-9.8	-5.5	-9.2	-3.8	-9.5	-8.8

From table 4.8, it is observed that the results for the cold flow properties using the three methods falls approximately within the same range, this suggest that the results are within the range as specified by ASTM standard.

Table 4.10: Solid fraction at CP, CFPP and PP while biodiesel is freezing for B80K20 Oven

Cold Flow Properties	**Temperature (°*C*)**	**Solid Fraction (*fs*)**
CP	-3.8	~0.002
CFPP	-8.8	0.43-0.68
PP	-9.2	~0.76

Table 4.11: Solid fraction at CP, CFPP and PP while biodiesel is freezing for B80K20 Ambient

Cold Flow Properties	**Temperature (°*C*)**	**Solid Fraction (*fs*)**
CP	-5.7	~0.0048
CFPP	-9.3	0.27-0.56
PP	-9.8	~0.63

From Table 4.9 and 4.10 and also from Figure 4.21 and 4.22, it is observed that the cold flow properties values varies, for B80K20, the solid fraction for the sample stored in a temperature controlled oven increases as compared to the sample stored at ambient. However, the pour point (PP) for B80K20 stored at temperature controlled oven is lower than the sample stored at ambient condition.

CHAPTER 5

CONCLUSIONS

This chapter brings to an end of this report, it state the various conclusions of the report and the various recommendations which will help in further improving any subsequent analysis that will be done based on this report.

5.1 Conclusions

All analysis and data stated in this report was conducted and data collected according to their various stipulated standards in the Mechanical Engineering laboratory.

The cold flow properties of the waste sunflower biodiesel was measured according to ASTM D97, ASTM D2500 and ASTM D6371-05 for cold filter plugging point (CFPP), cloud point and pour point respectively. The cold flow properties of the waste sunflower biodiesel sample were measured for different storage conditions. Hence, the results in Table 4.5 and Table 4.6 were obtained for different blend proportions.

The kinematic viscosity of the waste sunflower biodiesel blend sample was also measured according to standards, the different blend proportions were measured at 40°C also at different storage conditions. The samples were measured at 40°C controlled oven and ambient temperature with the ratio of B95K5, B90K10, B85K15 and B80K20 stored aver a period of time. It was observed that:

- The kinematic viscosity increases with an increase in storage period for the first 40 to 50 days of storage
- The kinematic viscosity decreases with increase in the volume of kerosene
- It was noticed that there were no significant change in the kinematic viscosity for both samples stored at ambient and the sample stored in the 40°C controlled oven for B95K5, B90K10 and B85K15.
- The increase in kerosene volume causes the intermolecular forces in the sample to be weaker thereby reducing the kinematic viscosity and causing any temperature

different to be significant, this was the case for B80K20 as it displays a different trend from others.

Furthermore, the density was measured according to standards at 15°C, it was measured at different storage conditions of 40°C controlled oven and ambient condition at an interval of 10days, the sample was blended with kerosene at different volume and measured accordingly, hence it was observed that:

- The density of the waste sunflower biodiesel increases with increase in storage period
- Increase in the volume of kerosene reduces the density
- The density of the sample stored at 40°Ccontrolled conditions increases steadily with storage period, while the samples stored in the ambient condition exhibited some level of fluctuations.
- It was noticed that there were no significant change in the density for both samples stored at ambient and the sample stored in the 40°C controlled oven for B95K5, B90K10 and B85K15.
- It was observed that the density of the sample in the 40°C oven tends to be higher than that stored at ambient for B80K20, this is because of thermal instability of the oven sample brought about by relative increase in the volume of kerosene which causes the rate of oxidation at higher temperature to increase which in turns increases the weight of the biodiesel in 40°C oven due to formation of insoluble, this causes the sample stored in the oven to exhibit higher density as compared to that stored at ambient temperature.

The thermal analysis of the sample were carried out using the Newtonian thermal analysis for B90K10 and B80K20, in the Newtonian thermal analysis the cooling curve, the cooling rate, the cooling bath temperature and the Newtonian (Zn) was determine for the samples at various storage conditions. The Newtonian thermal analysis was carried out to determine the variation of the solid fraction of the sample while it was freezing. The solid fractions were estimated as ~0.0048, 0.27⟶0.56 and ~0.63 respectively for CP, CFPP and PP for B80K20 ambient and ~0.002, 0.43⟶0.68 and ~0.76 respectively for CP, CFPP and PP for B80K20 Oven.

5.2 Recommendations

- It is therefore recommended that further study be done on the sample by increasing the temperature of the biodiesel blends to 60°C to observe the behavior.
- Also it is recommended that the density of the blends be reduced to 0°C and the behavior be investigated to observe its properties
- It is also recommended that further analysis should also be done using Fourier Thermal analysis and the results compared
- It is not clear that density and kinematic viscosity keeps increasing with time. Hence, I recommend that storage period should be further increased for the experiment to clarify that.

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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

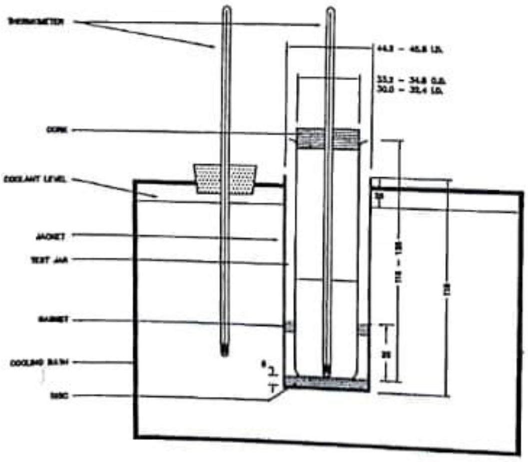
4.1 The specimen is cooled at a specified rate and examined periodically. The temperature at which a cloud is first observed at the bottom of the test jar is recorded as the cloud point.

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.

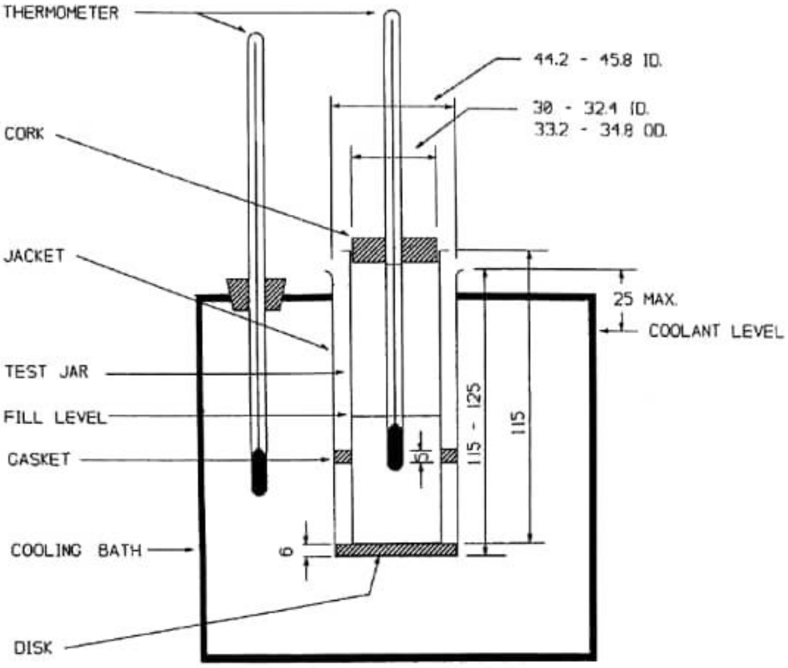
[2] Statements defining this test and its significance when applied to electrical insulating oils of mineral origin will be found in Guide D 117.

[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:

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
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

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

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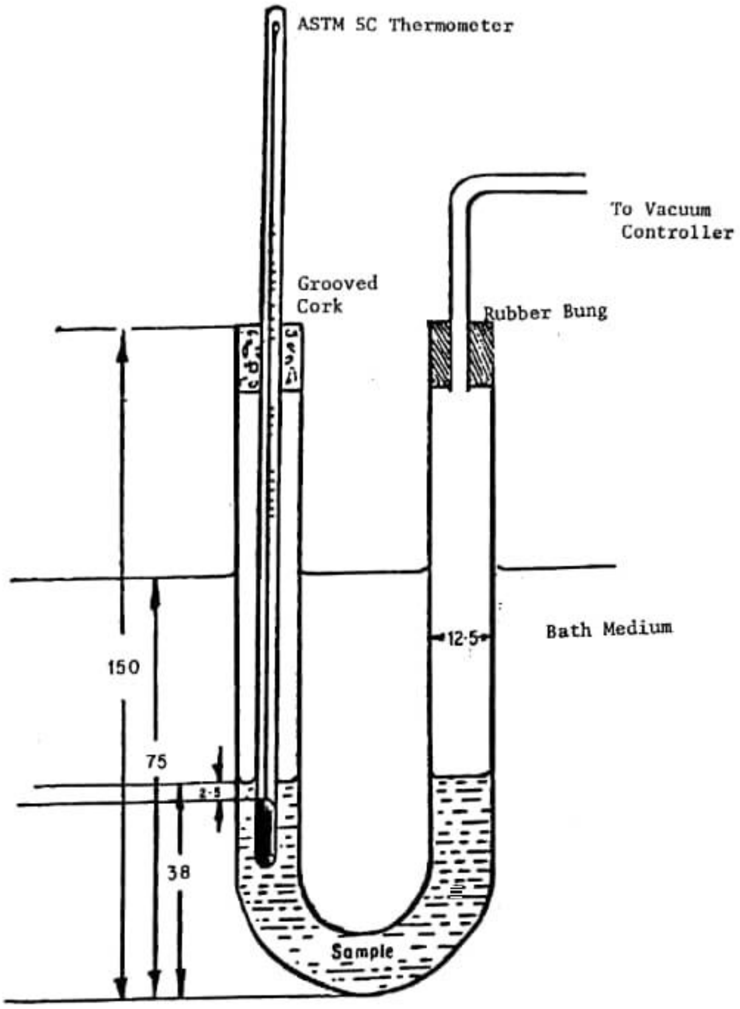
[7] The cloud point procedure formerly part of this test method now appears as Test Method D 2500.



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.

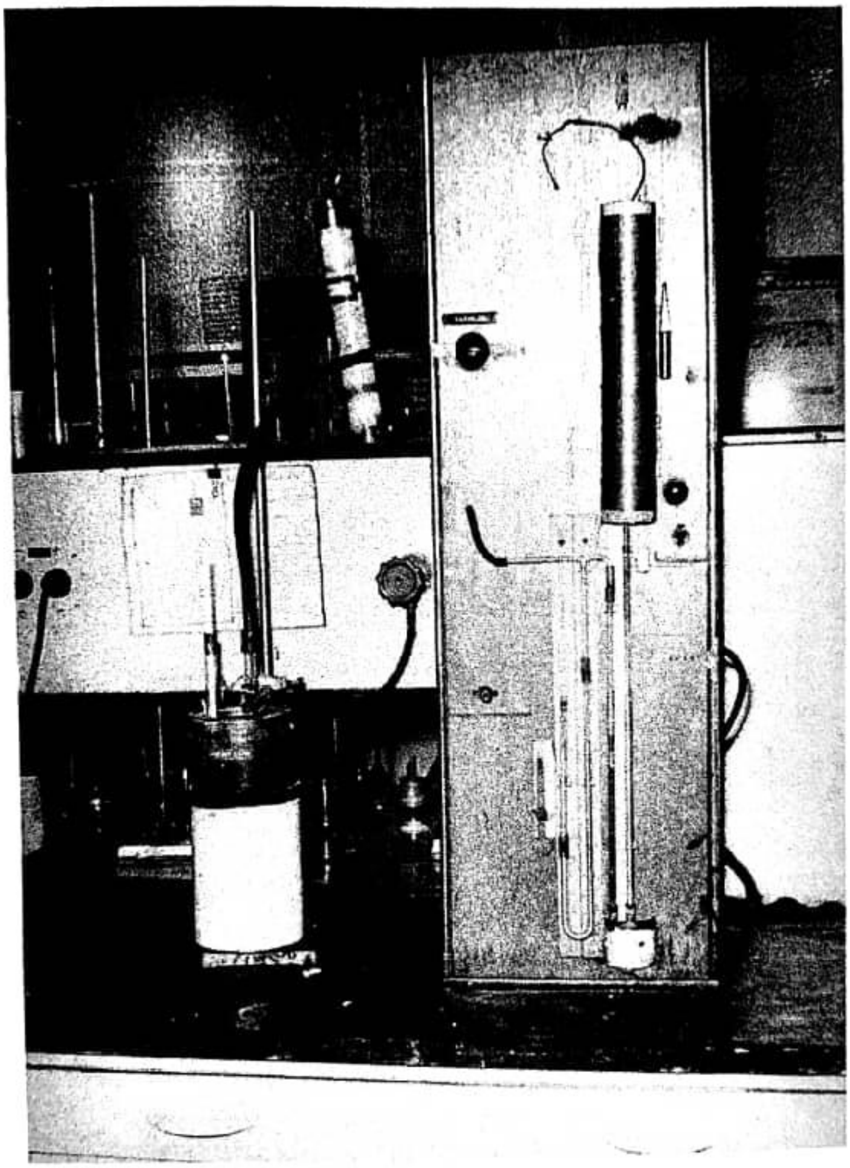

FIG. X1.2 Fluidity Temperature Apparatus

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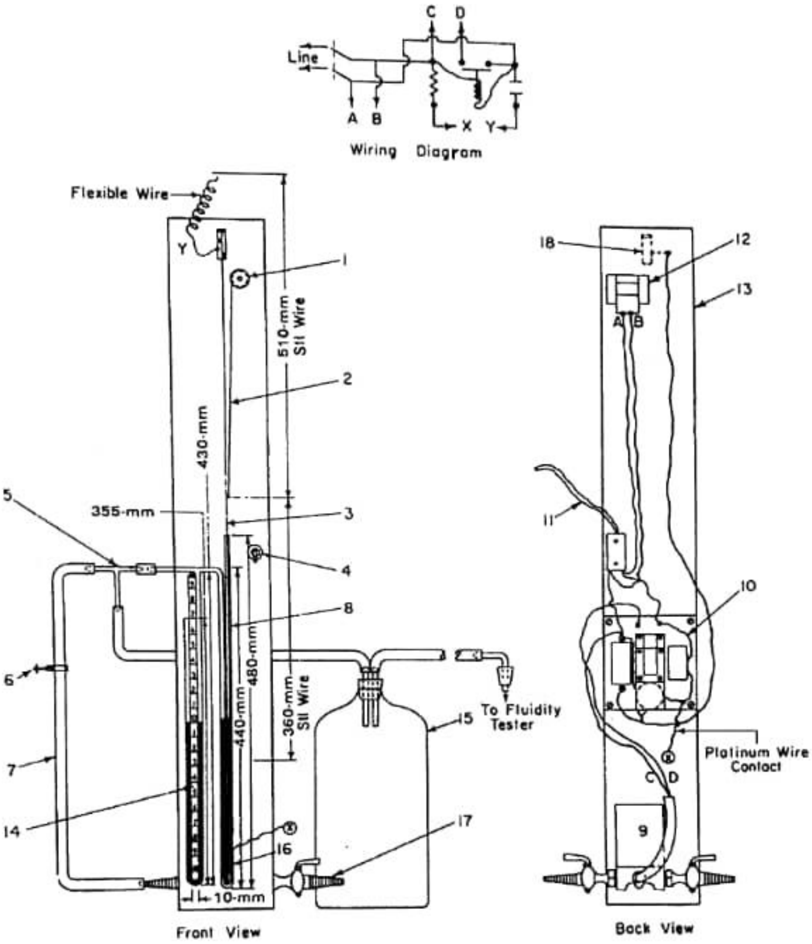
[9] This apparatus may be shop fabricated. Details of special parts are indicated in Figs. X1.3 and X1.4. Alternatively the apparatus can be purchased.



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

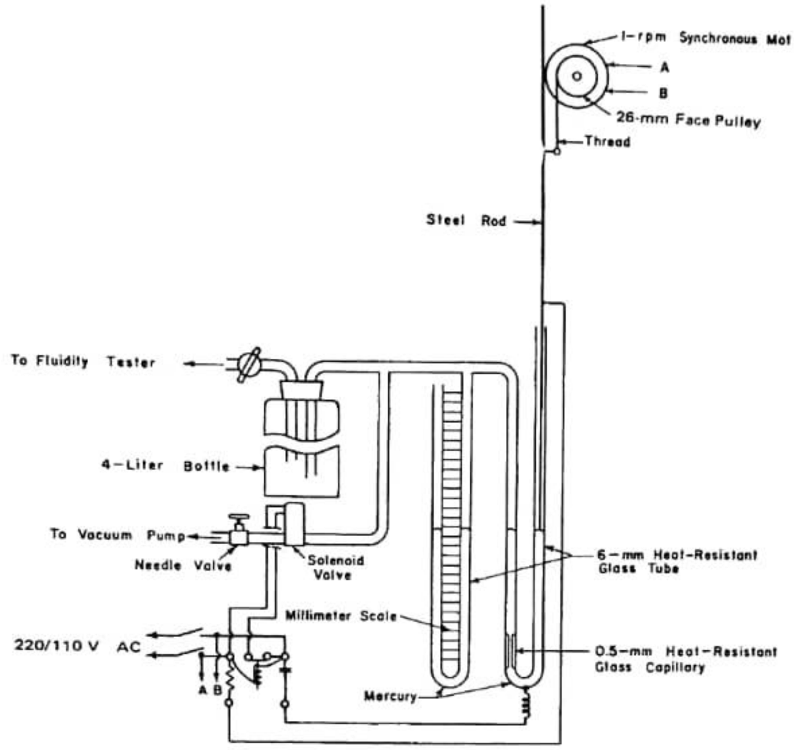


FIG. X1.4 Detail of Automatic Vacuum Controller

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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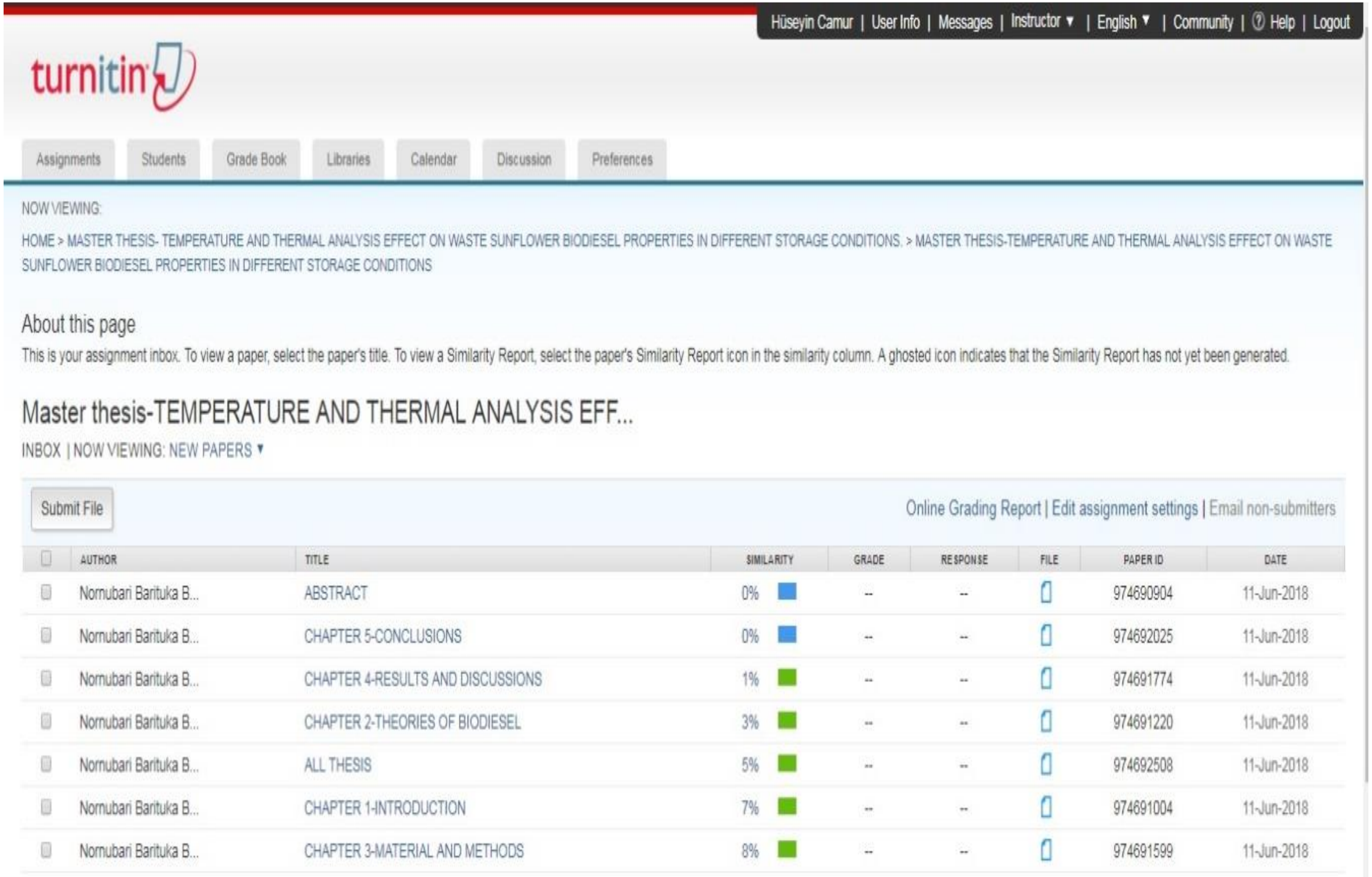

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