

**FFA DETERMINING SENSOR MODULES FOR OIL QUALITY
ASSESSMENT FOR PREDICTING HIGH PERFORMANCE LOW
EMISSION BIODIESEL**

Submitted in partial fulfilment of the requirements for the award of

Bachelor of Engineering degree in
Mechanical Engineering

By

J. KISHOR KUMAR (39150068)

G. SUSHIL (39150126)



**DEPARTMENT OF MECHANICAL ENGINEERING SCHOOL OF
MECHANICAL ENGINEERING**

SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY

(DEEMED TO BE UNIVERSITY)

**Accredited with Grade "A" by NAAC | 12B Status by UGC | Approved by
AICTE JEPPIAAR NAGAR, RAJIV GANDHI SALAI, CHENNAI - 600 119**

APRIL - 2023



SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)

Accredited with grade “A” by NAAC | 12B Status by UGC | Approved by AICTE
JEPPIAAR NAGAR, RAJIV GANDHI SALAI, CHENNAI – 600119

www.sathyabama.ac.in

DEPARTMENT OF MECHANICAL ENGINEERING 2022

BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **J KISHORKUMAR (39150068)** and **G SUSHIL (39150126)** who carried out the project entitled “**FFA DETERMINING SENSOR MODULES FOR OIL QUALITY ASSESSMENT FOR PREDICTING HIGH PERFORMANCE LOW EMISSION BIODIESEL**” under our supervision from November 2022 to April 2023

Internal Guide

Dr. J. JAYAPRABAKAR, M.E., Ph.D.,

Head of the Department

Dr. G. ARUNKUMAR, M.E., Ph.D.,

Submitted for Viva voce Examination Held on 26.04.2023

Internal Examiner

Dr. J. Hemanandh, M.E., Ph.D.,

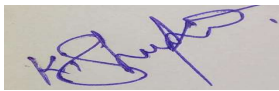
External Examiner

Dr. J. Jayaprabakar, M.E., Ph.D.,

DECLARATION

I J. Kishor Kumar (39150068) and G. Sushil (39150126) hereby declare that the Project Report entitled “**FFA determining sensor modules for oil quality assessment for predicting high performance and low emission biodiesel**” done by me under the guidance of **Dr J. Jayaprabakar M.E., Ph.D** is submitted in partial fulfilment of the requirements for the award of Bachelor of Engineering degree in Mechanical Engineering.

1. 

2. 

DATE: 26.04.2023

PLACE: CHENNAI

SIGNATURES OF THE CANDIDATES

ACKNOWLEDGEMENT

I am pleased to acknowledge my sincere thanks to Board of Management of **SATHYABAMA** for their kind encouragement in doing this project and for completing it successfully. I am grateful to them.

I convey my thanks to **Dr. S. Prakash, M.E., Ph.D., Dean, School of Mechanical Engineering** and **Dr. G. Arunkumar, M.E., Ph.D., Head of the Department, Dept. of Mechanical Engineering** for providing us necessary support and details at the right time during the progressive reviews.

I would like to express our sincere and deep sense of gratitude to my Project Guide **Dr. J. Jayaprakash, M.E., Ph.D.**, for his valuable guidance, suggestions and constant encouragement paved way for the successful completion of my project work.

I wish to express my thanks to all Teaching and Non-teaching staff members of the Department of Mechanical Engineering who were helpful in many ways for the successful completion of the project.

ABSTRACT:

In the current period as there has been a depletion for the traditional conventional fuel sources that has been used by the internal combustion engines. To meet the requirements the alternate fuel energies have been used by us over these years. In these alternate sources used biodiesel has played an important role. Biodiesel has been used as a potential contender for the conventional diesel. These, biodiesel can be obtained from different feedstocks. The pure biodiesel can be used in the diesel engines but it has its own setbacks such as the higher densities, lower cetane number and the calorific value is also lesser, these affects the performance, emission, combustion of the engine.

These drawbacks with the biodiesel withheld it from using it as a substitute for the conventional diesel. These issues can be reduced down to an extent by the usage of blends with biodiesel. The usage of the nanoparticles as blend in the biodiesel the has a comparatively positive effects in the performance, combustion and emission characteristics of the diesel engine. Different types of nano catalyst blends were used and their engine characteristics were analyzed.

The effect of the brake thermal efficiency (BTE) and the brake specific fuel consumption (BSFE) are also analyzed and accordingly the nano catalyst blends are used with the biodiesel. Also, the emission gases such as the CO₂, HCO, CO, PM has also subsequently decreased on the usage of the nano catalyst blends. Hence, in the studies and the experiments conducted by the researchers it is seen that the usage of the biodiesel blends with nano catalysts, can be used in the internal combustion engines which in turn can also subsequently increase the engine efficiency, performance and also, it's emissions can be reduced to a considerable extent.

TABLE OF CONTENT

CHAPTER NO	TITLE	PAGE NO
1	INTRODUCTION	12
	1.1 Bio Diesel introduction	12
	1.2 Feedstock sources for biodiesel	12
	1.3 Generations of Bio Diesel	14
	1.3.1 The First Generation of Biofuels	15
	1.3.2 The Second Generation of Biofuels	15
	1.3.3 The Third Generation of Biofuels	16
2	LITERATURE SURVEY	17
	2.1 Introduction	17
	2.2 Process to asses oil quality	17
	2.3 FFA analysis for oil	18
	2.4 Gas chromatography for oil sample	18
3	AIM AND SCOPE OF WORK	19
	3.1 Aim	19

3.2 Scope of The Work	19
-----------------------	----

CHAPTER NO	TITLE	PAGE NO
4	METHODOLOGY, EXPERIMENTAL SETUP AND EQUIPMENT'S	20
	4.1 Materials	20
	4.2 Biodiesel production from waste cooking oil	20
	4.2.1 FFA	21
	4.2.2 Density	22
	4.2.3 Viscosity	23
	4.3 Gas Chromatography	25
	4.4 Moisture content measurement	30
	4.5 Flash point & fire point measurement	30
	4.6 Copper strip corrosion test	31
	4.7 Cloud point and pour point measurement	32
	4.8 Calorific value measurement	33

CHAPTER NO	TITLE	PAGE NO
5	RESULTS AND DISCUSSIONS	34

CHAPTER NO	TITLE	PAGE NO
6	CONCLUSION AND SUMMARY	36
7	REFERENCES	38

LIST OF FIGURES

FIGURE NO	TITLE	PAGE NO
4.1	Oil heating for FFA determination	21
4.2	FFA determining process	22
4.3	Change of colour after adding indicator	22
4.4	Density calculation process	23
4.5	Kinematic viscometer	24
4.6	Kinematic viscosity calculation process	24
4.7	Gas chromatography flow diagram	25
4.8	Gases used in GC	27
4.9	GC machine	27
4.10	GC calculation	28
4.11	GC graph for waste cooking oil	28
4.12	Moisture content	30
4.13	Flash and fire point values measurement	31
4.14	Copper strip corrosion test	32
4.15	Cloud point and pour point measurement	33
4.16	Calorific value measurement	33

LIST OF TABLES

TABLE NO	TITLE	PAGE NO
1.1	Country and its potential feedstocks	13
1.2	Generations of biofuel	14
4.1	GC waste cooking oil table	29

ABBREVIATIONS

GC - Gas Chromatography

FFA - Free Fatty Acids

ML- Machine Learning

AV - Acid Value

MS- Mass Spectrometry

FID - Flame Ionisation Detector

HPLC - High Performance Liquid Chromatography

CE - Capillary Electrophoresis

LLE - Liquid Liquid Extraction

TLC - Thin Layer Chromatography

CHAPTER-1

INTRODUCTION

1.1 Biodiesel introduction:

Biodiesel is known as methyl ester which is oxygenated fuels consisting of long chain fatty acids derived from vegetable oils either edible or non-edible, animal fats and waste restaurant greases. Biodiesel is non-explosive, biodegradable, non-flammable, renewable as well as non-toxic. It is used in internal combustion engines (diesel engine) as alternative of diesel fuel with no modification or small modification with same or better performance compared to ordinary diesel fuel. Biodiesel is being used in developing and developed countries like Europe and USA to reduce the environmental pollution and dependency on fossil diesel fuel. Biodiesel contains no petroleum products and it is compatible with petro-diesel fuel. Biodiesel can be blended at any proportion with diesel fuel and it makes stable blend when blended together to be used in a diesel engine.

Biodiesel is made from sources such as plants, agricultural waste agricultural crops, vegetable oils. In the last few decades biodiesel play vital role as a substitute fuel in the work of research due to its feasible and ecologically sound properties. It has properties same as conventional fossil fuels such as petrol and diesel certain properties are better than petrol and diesel properties like as high cetane number, high flashpoint, high combustion efficiency, lower CO₂, NO_x emission, better lubrication, lower sulphur content etc. Petro diesel has a flashpoint off 337 K whereas biodiesel has the flashpoint of 423K that provides it to be non-explosive and non-flammable that makes transportation handling storage easy and safe. Also, this could be used in engines as such directly without any alterations to be made that adds up as a complementary property. Also, it was estimated that by the year of 2020 the requirement of biodiesel will rise twice or thrice

1.2 Feedstock sources for biodiesel:

The widespread variation of choosing feedstocks for methyl ester production is a great advantage. According to the literature, only feedstock accession considers two-third of total biodiesel production costs. More than 350

oil-bearing crops are recognized universally as a biodiesel sources which can be distributed into four major categories such as (a) Edible vegetable oil: peanut oil, soybean oil, sunflower oil, Safflower, corn oil, Rice bran oil, palm oil, coconut oil, used fried oil, olive oil, rapeseed oil, mustard oil, milkweed seed oil and linseed oil, wheat, (b) Non-edible vegetable oil: *Jatropha curcas*, *Pongamia glabra* (karanja), *Madhuca indica* (Mahua), *Salvadora oleoides* (Pilu), cotton seed oil, Tobacco, *Calophyllum inophyllum*, *Eruca Sativa* Gars, terebinth, rubber seed oil, desert date, fish oil, Jojoba, Neem oil, leather pre-fleshings, apricot seed, *Pistacia Chinensis* Bunge Seed, Sal oil, *Moringa Oleifera* and *Croton megalocarpus*, (c) Waste or recycled oil and (d) Animal fats: chicken fat, pork lard, beef tallow and poultry fats. Besides some other feedstock are: Algae, Bacteria, Micro-algae, Terpenes, Latexes, Fungi etc.,

Table 1.1 Country and its potential feedstocks

Country	Potential feedstock
Argentina	Soybeans
Australia	Beauty leaf, <i>Jatropha curcas</i> , <i>Pongamia</i>
Bangladesh	Rubber seed, <i>Pongamia Pinnata</i>
Brazil	Soybeans/Palm oil/Castor/Cotton oil
Canada	Rapeseed/Animal fat/Soybeans/Yellow grease and Tallow
China	<i>Jatropha</i> /Waste cooking oil/Rapeseed
Cuba	<i>Jatropha curcas</i> , Moringa, Neem
France	Rapeseed/Sunflower
Germany	Rapeseed
Ghana	Palm
Greece	Cottonseed
India	<i>Jatropha</i> / <i>Pongamia</i> (karanja)/Soybean/Sunflower/Rapeseed
Indonesia	Palm oil/ <i>Jatropha</i> /Coconut
Iran	Palm, <i>Jatropha</i> , castor, Algae
Ireland	frying oil/animal fats

Italy Rapeseed/Sunflower
 Japan Waste cooking oil
 Kenya Castor Malaysia Palm oil
 Mexico Animal fat/ Waste oil
 New Zealand Waste cooking oil/Tallow
 Norway Animal fats
 Pakistan Jatropha curcas
 Peru Palm, Jatropha
 Philippines Coconut/Jatropha
 Singapore Palm oil
 Spain Linseed oil/Sunflower
 Sweden Rapeseed
 Thailand Palm/Jatropha/Coconut
 Turkey Sunflower, Rapeseed
 UK Rapeseed/Waste cooking oil
 USA Soybeans/waste oil/Peanut
 Zimbabwe Jatropha

1.3 Generations of biodiesel:

The biodiesel is classified into three generations such as 1st generation, 2nd generation and 3rd generation biofuels. The examples of it and its properties are shown in the below given table.

Table-1.2 Generations of biofuel

Biofuel	Feedstock source	Advantages	disadvantages
<u>1st generation</u> * Wheat * Barley * Sugarcane * sugar beet * plant oil	Edible oil	simple conversation process	• Relative low oil yield • Biofuel debate • Causes deforestation and destroying ecosystem.
<u>2nd generation</u> *	Non-edible oil	• Abundance availability number of non-edible	• Intractable structure of the

Lignocellulosic biomass * Agricultural waste * Forest waste * Municipal solid waste		crops worldwide. • No debate between food vs fuel economy.	feedstock
<u>3rd generation</u> * Microalgae * Macroalgae * Microbes	Algal biomass	• High growth rate • Its cultivation reduces global warming.	• It requires advance technology for biodiesel conversion

1.3.1 First generation biofuel

These first generation of biofuels are produced from agricultural crops, which are a vital food source for humans. In this generation the biodiesel is made from corn starch, sugar from potatoes and vegetable oil obtained from the crops which humans consume on a daily basis. Scientist feared that it may cause an instability in the environment because of the growing demand for the oils. This would also create competition within the farmers. Thus, leading to usage of excess fertilizers and pesticides to increase the crop yield. This would change the chemical properties of the biodiesel to a certain extent. So, this type of production of biodiesel was not entertained.

1.3.2 Second Generation Biofuel

The fear of the scientists that the first generation may cause imbalance and shortage of food supply. This led to change of feed stock to non-edible sources such as harvest wastes, municipal solid waste, foods that already fulfilled their purpose (Used cooking oil). These second-generation biofuels are made from cellulosic biomass or waste plant materials. This helps to reduce the burden on the edible oil for preparation of biodiesel. So here no arable land is used solely for production of biodiesel, Biodiesel production from waste animal fat and improvement of its characteristics by synthesized nickel and magnesium additive. Its waste product is used for production of biodiesel. Non-edible food second generation feedstock include used cooking oil, jatropha oil, grasses and

other seed oils. To convert cellulose biomass to biodiesel a series of pre-treatment is required

1.3.3. Third generation biofuel

Scientists found that algae is rich in oil content and it is unwanted plant. It grows in wastewater and absorbs oxygen from the water bodies. It also does not require any arable land for growing. So this feature has made algae very efficient to grow. These algae can be grown in pond and wastewater treatment plants.

It is not used commercially because its extraction is costly and pretreatment is required. Eventually after extraction is made easier it will be commercially used on a large scale.

CHAPTER 2

LITERATURE SURVEY

2.1 Introduction:

The global production of vegetable oil was estimated to hit 187 million metric tons for 2016/17. The quality of edible oil is of primary importance to both manufacturers and consumers of deep-fried food products. Although the roles of saturated fats in a healthy diet are the subject of renewed debate (Parodi [2016](#)), there are still concerns linked to the level of saturated fatty acids in food (Dawczynski et al. [2015](#)). Edible oil is subject to significant chemical changes in use and storage (Choe and Min [2007](#)), while numerous studies have demonstrated that some degradation products (Mozaffarian et al. [2006](#); Oomen et al. [2001](#)) are hazardous to human health. Therefore, monitoring oil degradation has been an important topic.

2.2 Process to assess oil quality

The main processes that reduce the quality of oil during frying can be broadly categorised into hydrolysis, oxidation and polymerization (Bordin et al. [2013](#)). These result in the generation of FFAs, aldehydes, ketones, acids, and many other products (Fritsch [1981](#)). Many quality control methods have been developed, each responding to a different subset of the complex array of oil degradation products. These include FFA value (FFA), total polar material (TPM), iodine value (IV), peroxide value (PV), p-anisidine value (p-AnV) and thiobarbituric acid reactive substances (TBARS) (Shahidi and Zhong [2005](#)).

This work focuses on the analysis of FFAs. Hydrolysis of the ester linkage of triacylglycerols (TAG) to produce FFAs, diglycerides (DAG), monoglycerides (MAG), and glycerol (Choe and Min [2007](#)) leads to various undesirable changes, including production of off-flavours, decrease of smoke point and acceleration of further hydrolysis (Choe and Min [2007](#), Frega et al. [1999](#)). Hydrolysis also leads to a drop of surface tension of the oil thereby, increasing oxygen accessibility during frying and thus promoting oxidative degradation of oil (Choe and Min [2007](#)).

2.3 FFA analysis for oil

The acid–base titration of FFA, using phenolphthalein for endpoint determination has been the most commonly used in routine assessment of frying oil quality. Although the method was significantly improved following the introduction of technologies such as potentiometric endpoint determination, it still suffers from several drawbacks, including requirements for large amounts of organic solvents and a large sample size. The classic FFAs test requires 20 g of oil and 150 ml of solvent (IUPAC [1979](#)). Another limitation of the FFA test is that it measures an overall level of titratable acids and does not identify the profile of FFAs. Such additional information would help develop further understanding of the kinetics of the reaction and could lead to significant improvements to quality control in fried food production. GC of fatty acid methyl esters (FAMES) produced using a catalyst such as BF_3 is a classic method for analysis of fatty acid profiles in edible oils (Wirasmita et al. [2013](#)). However, this also cleaves ester bonds if TAG, DAG or MAG are present (O'Keefe and Pike [2010](#)) which produces difficulties if the focus of analysis was on FFA.

2.4 Gas Chromatography for oil sample

Therefore, a suitable FFA extraction process is needed. Researchers have used ultrafiltration (Keurentjes et al. [1992](#)), solid phase extraction (SPE) (Paik et al. [2009](#)), thin layer chromatography (TLC) (Sampels and Pickova [2011](#)), supercritical fluid extraction (SFE) (Cao and Ito [2003](#)), and selective esterification (Kail et al. [2012](#)). These studies had different degrees of success, but all were relatively complicated and expensive to operate. Therefore, simpler liquid–liquid extraction (LLE) may be an attractive option for many. For example, Shah and Venkatesan ([1989](#)) used aqueous isopropyl alcohol as a solvent, while Rodrigues and Meirelles ([2008](#)) used ethanol and water. This study further investigated the feasibility of extracting FFA using a simple LLE system supported by GC analysis of extracted and unextracted fractions.

CHAPTER 3

AIM AND SCOPE

3.1 Aim:

There are many researches who have carried out various experiments and research works to determine the level of FFA. However, they are using different type of cooking oil. A few researches are also reported regarding the palm cooking oil but they are using different method such as gas chromatography of detectors. All methods to determine the amount of free fatty acids (FFA) in waste cooking oil are reviewed in this paper. And further as FFA is one of the main factors to determine the biodiesel purity and ease of production as only based on the FFA content the steps required to carry out the biodiesel production is determined

3.2 Scope:

Biodiesel plays a vital role as an alternate for the conventional fuel sources. It also plays an important role in the reduction of the pollutant particles suspended into the air and the atmosphere. In this work the FFA are collectively collected for biodiesel is analysed using software and sensors which will be extensively done in this work and using those interpreted data, it is used to predict the high performance and the low emission of the biodiesel.

CHAPTER 4

MATERIALS AND METHODS

The basic properties of the raw feedstock for the production of the biodiesel is found such as the density, viscosity, FFA, Gas chromatography (GC), cloud point, pour point, fire point, flash point, calorific value etc., are found and analysed for both the raw feedstock before producing the biodiesel and also after the pure biodiesel production. The produced biodiesel is the added with the nanoparticles that are nano catalyst. As these blends have adverse effects on the performance and emission characteristics of the produced biofuel when blended with it. Several nano catalysts are studied to be blended with the biodiesel based upon its resulting characteristics and suitable nano catalysts are blended with the produced biodiesel to get positive results. Then for the produced pure biodiesel blend the engine testing is done and its characteristics are analysed.

4.1 Materials

- Waste cooking oil
- GC calculator
- Various nano catalysts
- Sensor development for analysis
- Engine for analysis

4.2 Biodiesel production from waste cooking oil

First for the production of biodiesel raw materials such as waste cooking oil for the biodiesel production is acquired from various sources. Then the work for the biodiesel production is started. That includes analysing the density, viscosity, FFA, cloud point, pour point, fire point, flash point, calorific value, GC analysis for the acquired raw feedstock waste cooking oil. There characteristics are found for both the raw waste cooking oil acquired before biodiesel production and also after the production of biodiesel.

4.2.1 Free Fatty Acid (FFA):

Titration of FFA is done over the following steps

- Take 5 gm of oil (waste cooking oil sample)
- Add 25 ml isopropyl alcohol and drops 0.1 normality NaOH.
- After that heat the sample at 60 degrees Celsius.
- After that let it cooled down to room temperature.
- After that add 2 drops of phenolphthalein indicator, titrate against 0.1 normality NaOH.
- At the end pink colour will be appearing.

$$\text{FFA} = \text{Normality} \times \text{burette reading} \times 28.2 / \text{sample of weight}$$

Above mentioned is the formula for FFA

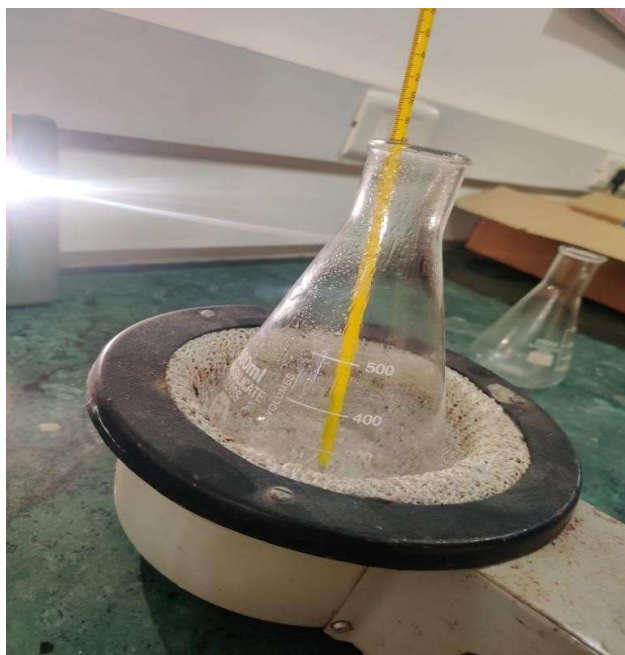


Fig 4.1 oil heating for FFA determination

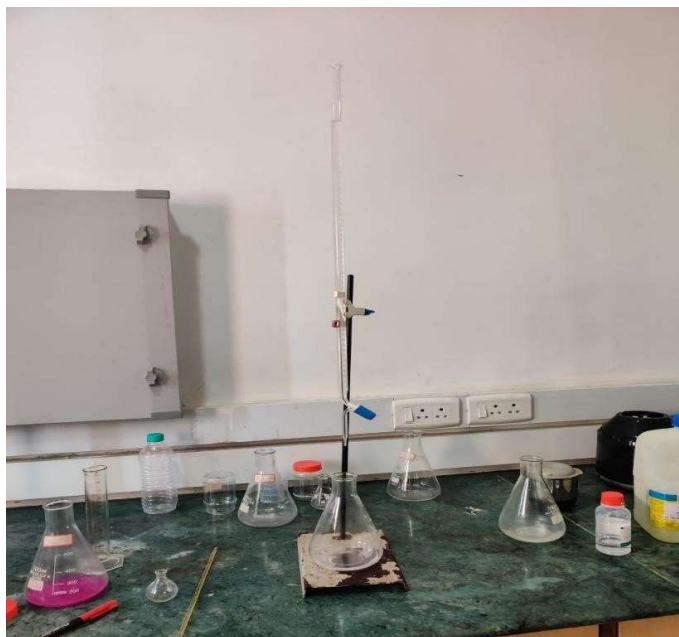


Fig 4.2 FFA determining process

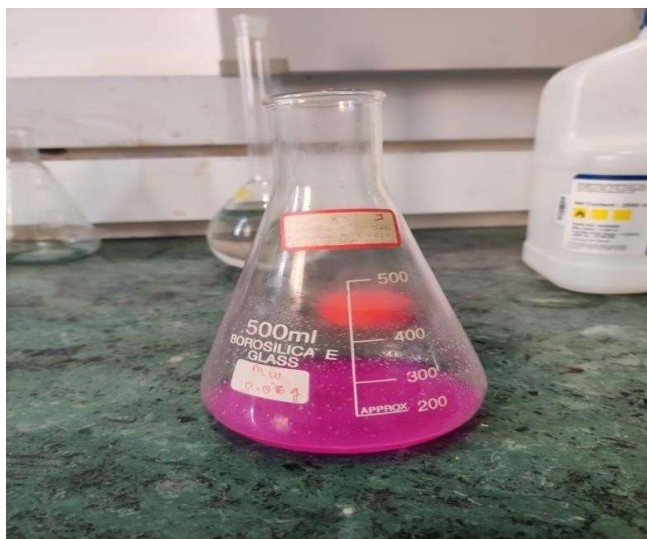


Fig 4.3 Change of colour after adding indicator

4.2.2 Density:

Density of the oil is important to find its biofuel characteristics as the burning condition of the biofuel depends upon the density of the oil used. The density of the oils varies with each type and temperature. Comparing to water, whose density is 1.00 g/ml, cooking oil is less dense. Density is found by taking

the weight of the oil in the specific gravity bottle and is found using the density formula as mentioned.

Density = empty gravity bottle + sample – empty gravity bottle / 25ml

Its unit is g/cm³.



Fig 4.4 Density calculation process

4.2.3 Viscosity:

Viscosity of the oil is important to find its biofuel characteristics as the burning condition of the biofuel depends upon the viscosity of the oil used. Viscosity is the resistance of a fluid (liquid or gas) to a change in shape or movement of neighbouring portions relative to one another. Viscosity denotes opposition to flow. Its unit is cSt (Centi stoke). Viscosity is found using the kinematic viscometer bath machine in which the oil is placed in a glass flask tube and is heated to 41-degree Celsius and the oil is expanded using the sucker and it is allowed to settle down and the time is noted for the oil to settle down. The formula for viscosity is

Viscosity = Time taken x 0.014



Fig 4.5 Kinematic viscometer

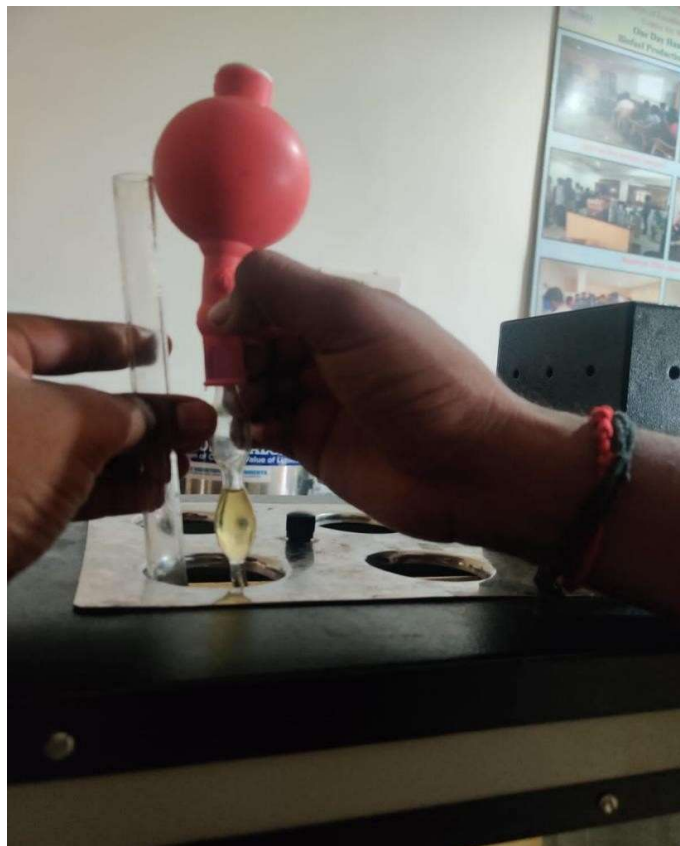


Fig 4.6 Kinematic viscosity calculation process

4.3 Gas Chromatography:

Tsvett Early 1900s, Gas chromatography (GC) is discovered by Mikhail Semenovitch. This method used to do a separation technique to separate compounds. In this GC method, stationary phase is a column that contains substance is located in the device. The gases filled in carrier phase meanwhile mobile phase is an inert gas, which went through a column under the high pressure. The sample that needs to analysis have to be heated to produce gas before it go to gaseous mobile phase.

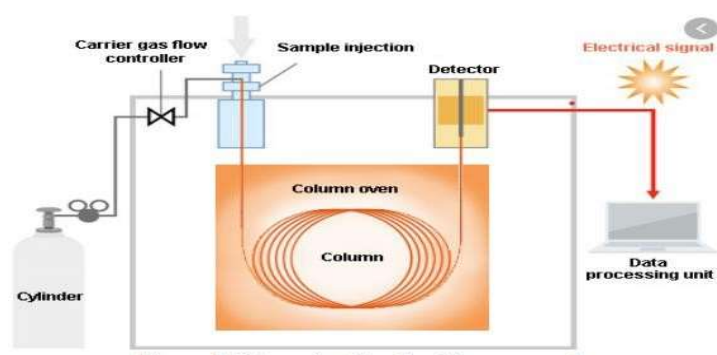


Fig 4.7 Gas Chromatography Flowdiagram

Gas chromatograph analysis commonly used because it is simple, multifaceted and highly sensitive as reported by Ozlem Coskun. Other than that, this method is very convenience, accurate and small budget needed to do an analysis. Normally, GC chromatogram results showed sharp and symmetric peaks. If the stationary phase is being used, the interpreted resolution of GC is highly rely on the column length and polarity. There are two type of detectors that used to determine FFAs which is Mass spectrometry detector (MS) and flame ionization detector (FID). However, as reported by Azeman et al., flame ionization detector (FID) is one of detector that widely and commonly used as a detector to detect existence of FFA . FID is the most appropriate compared to other detector because of its sensitivity and resolution and because it reliable and relatively easy to use. In addition, various compounds can to be detected by using FID. Meanwhile, mass spectrometry detectors tend to thermally degrade the samples before it can be detected. As the result; the entire sample is destroyed by fragmentation as reported by Scott RPW.

The machine used for GC is GYL6500 GC driver developed by YL Instruments company can control YL6500 chromatograph over the LAN connection. Direct control means that the chromatograph can be completely controlled from the Clarity environment. Instrument method controlling the analysis conditions will be saved in the measured chromatograms.

To use more than one YL6500 gas chromatograph on Clarity Station, a minimum YL6500 firmware version 1.7.1 and minimum Clarity version 4.0.3.876 is needed. If older Clarity/firmware is used, it is possible to control just one GC YL6500 on the same computer.

YL Instruments YL6500 Control

Controlled instruments YL6500 gas chromatograph

Data acquisition interface LAN - Digital Data Acquisition

Communication interface LAN

Purchasing GC Control (p/n A23)

Related products Clarity (p/n C50)

Connection cross LAN (p/n SK08)

GC MACHINE SPECIFICATION

With improved APCs (Advanced Pneumatic Control), YL's 5th-generation Gas chromatograph provides fast, reliable and precise data from every single part — from stable inlets to detectors — as well as the from innovative haptic touch pad, which makes the instrument more intuitive and easier to operate while expediting the data-processing cycle. Furthermore, the GC can be configured to perform complex analyses with the installation of inlet and detector components.



Fig 4.8 Gases used in GC



Fig 4.9 GC machine

This is the GC result that is run for one of the samples of the waste cooking oil that is used for biodiesel production

4/18/2022 Chromatogram C:\YLC\arity\WORK1\DATA\NIRMAL\SAMPLES DATA\Mechanical Mini project sample\Waste cooking oil- Project Sample - 3.PRM Page 1 of 2



Sathyabama University
 Center of Excellence for Energy Research
 Ministry HRD (India) Funded Centre

Chromatogram Info:			
File Name	C:\YLC\arity\WORK1\DATA\NIRMAL\SAMPLES DATA\Mechanical Mini project sample\Waste cooking oil- Project Sample - 3.PRM		File Created : 4/18/2022 11:44:34 AM
Origin	Acquired, Acquisition started 4/18/2022 11:06:54 AM		Acquired Date : 4/18/2022 11:43:28 AM
Project	C:\YLC\arity\Projects\Work1.LPRJ		By : Administrator
Sample Info:			
Sample ID	Waste Cooking Oil		Amount : 0
Sample	Project Sample - 3		ISTD Amount : 0
Inj. Volume [mL]	0.2		Dilution : 0.1
Method	C:\YLC\arity\WORK1\DATA\NIRMAL\SAMPLES DATA\FID METHODS\FAME method-2		By : Administrator
Description			
Created	3/17/2018 9:53 AM	Modified	4/18/2022 11:44 AM
Column			
Mobile Phase			
Flow Rate			
Note			
Autostop	None		
Detector 1	Detector 1		
Subtraction Chromatogram	(None)		
External Start	Start - Restart, Down		
Range 1	Bipolar, 2500000 mV, 10 Samp. per Sec.		
Matching	No Change		
Base	Not Used	Calibration File	None
Scale Factor	Not Used	Units After Scaling	Not Used
Unretained Time	0.00 min	Column Length	50.00 mm
Result Table Reports	All Peaks	Hide ISTD Peak	Enabled
Calculation	Uncal		
Uncal. Response	0		
Column Calc.	From Width at 50% of Height		

Fig 4.10 GC Calculation

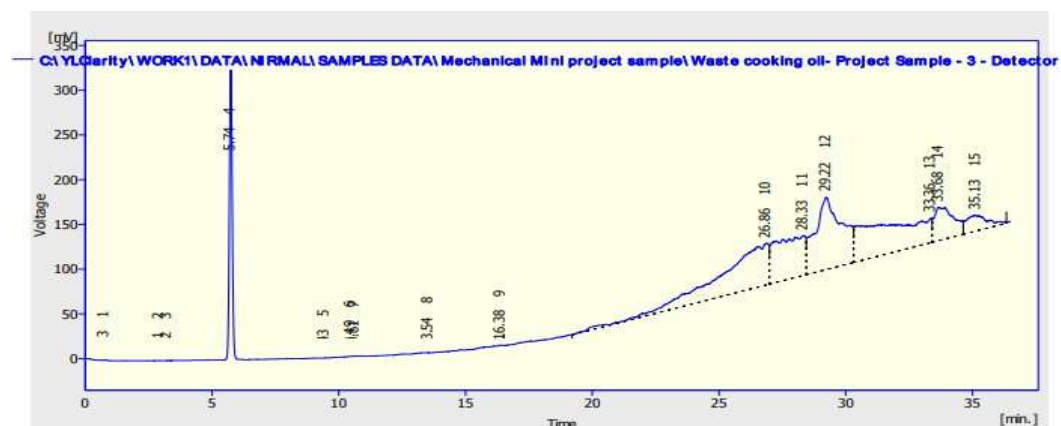


Fig 4.11 GC graph of Waste cooking oil

Table 4.1 GC waste cooking oil table

SI.NO	RETN TIME [min]	COMPOSITION	STRUCTURAL NOMENCLATURE	COMPOUND NAME
1	0.730	0.03076	C16:0	Methyl palmitate
2	2.910	0.01685	C18:0	Methyl stearate
3	3.217	0.007303	C20:0	Methyl arachidate
4	5.737	9.0622	C22:0	Methyl behenate
5	9.433	0.04155	C12:0	Methyl dodecanoate
6	10.487	0.09997	C13:0	Methyl tridecanoate
7	10.613	0.03275	C14:0	Methyl myristate
8	13.537	0.06754	C4:0	Methyl butyrate
9	16.383	0.1724	C17:0	Methyl heptadecanoate
10	26.860	23.8987	C18:2 (c9,c12)	Methyl linoleate
11	28.327	13.7850	C20:0	Methyl arachidate
12	29.223	21.3296	C12:0	Methyl dodecanoate
13	33.360	20.6657	C18:1 (trans-6)	Methyl petroselaidate
14	33.677	7.0322	C21:0	Methyl heneicosanoate
15	35.127	3.7571	C18:1 (trans-6)	Methyl petroselaidate

CALCULATION:

FFA HOME WATSE COOKING OIL:

$$= \frac{0.1 \times 0.8 \times 28.2}{5.12}$$

$$= 0.4408$$

4.4 Moisture Content Measurement:

The moisture content was determined by checking the weight of the crucible before and after heating. The initial weight of the crucible was measure and noted down. The weight on adding 10g of fuel sample was also noted down. The crucible was placed inside a hot air oven for three hours at a temperature of 110°C. The weight of the crucible was measured after the heating and was noted down. The difference between the weight before and after heating determines the moisture content of the sample. The results are displayed Moisture Content = 0.001%.



4.12 Moisture content

4.5 Flash Point and Fire Point Measurement:

Flash point and fire point of fuels in the temperature range from 40°C to 370°C can be measured by using a manual Pensky-Martens closed-cup apparatus. In the test, about 200 ml of biodiesel is poured into a brass cup which is then fit with a cover. The sample is heated and stirred steadily. An ignition source (fire) is introduced into the cup at regular intervals while simultaneously

stirring until it reaches a temperature at which the volatile material ignites and a flash is produced that spreads evenly throughout the inside of the cup.

This temperature was recorded as flash point. On further heating and stirring, an ignition source was introduced at regular intervals until it reaches a temperature where vapour is produced at a rate to sustain the fire for at least 5 seconds. This temperature was noted down as the fire point. The same procedure was carried out for diesel and the biodiesel blends.



Fig 4.13 Flash and fire point value measurement

4.6 Copper Strip Corrosion Test:

Pour some oil to the bomb unit and then put the copper strip inside. Place the bomb in copper strip corrosion bomb. Heat at 60°C for 3 hours and Check for corrosivity of hydro carbon liquids. Copper Strip Corrosion @ 60°C for 3hrs = 1a (slight tarnish).



Fig 4.14 Copper strip corrosion test @60°C for 3 hrs

4.7 Cloud Point and Pour Point Measurement:

Cloud Point (CP) and Pour Point (PP) values were measured according to ASTM D97-17 standards. The fuels were poured into a glass test tube and cooled under given conditions Figure 4.8 and was inspected at intervals of 1°C until tiny wax crystals were formed, giving rise to a cloudy structure. This temperature was noted down as CP.

The biodiesel sample was further cooled in a glass tube and observed at intervals of 3°C until the sample completely lost its fluidity even when it was tilted. The PP is noted as 3°C above the temperature at which the sample lost its fluidity



Fig 4.15 Cloud point and pour point measurement

4.8 Calorific Value Measurement:

The calorific value was determined using a bomb calorimeter Figure 4.9 This was achieved by measuring the amount of heat generated when biodiesel of 10ml was burnt in a closed chamber. The apparatus comprises of bomb, bomb firing unit, illuminator with magnifier, offset stirrer, calorimeter vessel, gas release valve, water jacket, pellet press vibrator, timer, pressure gauge, crucible and ignition wire.



Fig 4.16 Calorific value measurement

CHAPTER 5

RESULTS AND DISCUSSION

RESULT:

The application of the current biofuel production technologies and nanotechnology together delivers steady and tolerant chemical reaction which can minimize unfriendly impacts of different solvents and catalysts utilized during biofuel generation; moreover, it can reduce biofuel's cost of production. It is comprehended that most of the fuel properties can be enhanced by adding NPs. Thus, to determine the consequence of NPs utilization in DICl diesel engine performance, combustion and emission characteristics; critical selection of NPs and their best dosages are crucial. Furthermore, the physicochemical properties of the fuels used and their mixes have to be verified.

In most of the investigations piloted, the diesel engine performance enhances apparent with the NPs addition for most of the diesel, diesel–biodiesel and diesel–biodiesel–ethanol blends utilization in DICl diesel engines. A portion of the experiments demonstrated that increasing the concentration of NPs did not result in performance improvement equivalent to the proportions of NPs added. Thus, In order to find best performance enhancement effects, the main factor in choosing the ideal scope of NPs type, size and concentration ratio. Finally, using NPs in DICl diesel engine has the prospective to boost the performance and reduce the harmful exhaust emissions from the engine considerably.

1. Lower heating value of biodiesel and its blends produces lower net HRR, slightly lower brake thermal efficiency and higher BSFC than those of diesel fuel.
2. The quantitative comparisons reveal that biodiesel and its blends produces lesser exhaust emissions such as CO, HC and particulate matter/smoke and higher NO_x and CO₂ than diesel fuel due to higher cetane number and enriched oxygen content which leads to improved combustion.

3. Biodiesel does not contain any sulphur and aromatic compounds and hence does not emit any sulphurous components.
4. Biodiesel blends up to 20% can be used as alternate fuel in diesel engines with little or no modifications

However, biodiesel have some drawbacks when compared to mineral diesel fuel such as undesirable properties at low temperatures, higher viscosity, higher brake specific fuel consumption, higher NO_x emissions and slightly lower brake thermal efficiency. The higher specific fuel consumption and lower brake thermal efficiency are generally due to lower heating value and higher density of biodiesel. Although encouraging work has been carried out on performance, emission and combustion characteristics of biodiesel in a single cylinder diesel engines with waste cooking oil is observed from literature review that limited amount of work has been done to evaluate multi-cylinder engine characteristics with biodiesel produced from non-traditional vegetable oils. Biodiesel is currently not economically feasible. Improved technological developments and supporting policies are needed to promote the biodiesel research to make their prices competitive with other conventional sources of energy. Hence more investigations on long term issues such as engine deposits, injector choking and contamination of the engine have to be carried out to improve the engine performance with modified engine design based on the biodiesel properties.

CHAPTER 6

SUMMARY AND CONCLUSIONS

The objective of this study was to improve the performance and reduction in exhaust emissions from biodiesel operated diesel engine through CR and catalytic converter.

- According to the results; The use of biodiesel will lead to loss in BTE due to the reduction in heating value of biodiesel compared to diesel, but there exists power recovery of biodiesel operated diesel engine as the result of an increase in biodiesel fuel consumption.
- The majority of studies have shown that HC, CO and Smoke emissions were significantly reduced, compared with diesel. The higher oxygen content of biodiesel and lower aromatic compounds has been regarded as the main reasons. The vast majority of literatures agree that NO_x emission will increase when using biodiesel. This increase is mainly due to higher oxygen content, cetane number and different injection characteristics
- Most of researches showed that increase in BTE was observed at higher CR. Further, the HC, CO and Smoke emissions were reduced but increase of NO_x emission was observed with increase of CR.
- It can be concluded that increase of CR with biodiesel blend improved the performance and reduction in exhaust emissions except NO_x emission. In order to reduce the NO_x emission, the catalytic converter was used

a) The further studies on biodiesel endurance tests should be executed to make clear the reason and mechanism of wears

b) The further studies on the low temperature performance of biodiesel engine should be fulfilled because biodiesel presents higher viscosity than diesel, which could affect the emissions.

c) The further studies on non-regulated emissions of biodiesel should be carried out to obtain conclusive trend, especially for the carbonyl compounds emissions.

d) The methodology used for measurements need be improved to fulfil the expected requirements

CHAPTER 7

REFERENCES

1. Ansolin M, Basso RC, Meirelles AJdA, Batista EAC. Experimental data for liquid–liquid equilibrium of fatty systems with emphasis on the distribution of tocopherols and tocotrienols. *Fluid Phase Equilib.* 2013;338:78–86. doi: 10.1016/j.fluid.2012.09.033. [[CrossRef](#)] [[Google Scholar](#)]
2. Bayne S, Carlin M. *Forensic applications of high performance liquid chromatography*. Boca Raton: CRC Press/Taylor & Francis; 2010. [[Google Scholar](#)]
3. Berezin O, Turyan Y, Kuselman I, Shenhar A. Rapid and complete extraction of free fatty acids from oilseeds for acid value determination. *J Am Oil Chem Soc.* 1996;73:1707–1711. doi: 10.1007/BF02517976. [[CrossRef](#)] [[Google Scholar](#)]
4. Bordin K, Kunitake MT, Aracava KK, Favaro-Trindade CS. Changes in food caused by deep fat frying—a review. *Arch Latinoam Nutr.* 2013;63:5–13. [[PubMed](#)] [[Google Scholar](#)]
5. Bravi E, Benedetti P, Marconi O, Perretti G. Determination of free fatty acids in beer wort. *Food Chem.* 2014;151:374–378. doi: 10.1016/j.foodchem.2013.11.063. [[PubMed](#)] [[CrossRef](#)] [[Google Scholar](#)]
6. Bravi E, Marconi O, Sileoni V, Perretti G. Determination of free fatty acids in beer. *Food Chem.* 2017;215:341–346. doi: 10.1016/j.foodchem.2016.07.153. [[PubMed](#)] [[CrossRef](#)] [[Google Scholar](#)]
7. Cao X, Ito Y. Supercritical fluid extraction of grape seed oil and subsequent separation of free fatty acids by high-speed counter-current chromatography. *J Chromatogr A.* 2003;1021:117–124. doi: 10.1016/j.chroma.2003.09.001. [[PubMed](#)] [[CrossRef](#)] [[Google Scholar](#)]
8. Choe E, Min DB. Chemistry of deep-fat frying oils. *J Food Sci.* 2007;72:R77–R86. doi: 10.1111/j.1750-3841.2007.00352.x. [[PubMed](#)] [[CrossRef](#)] [[Google Scholar](#)]
9. Dawczynski C, Kleber ME, März W, Jahreis G, Lorkowski S. Saturated fatty acids are not off the hook Nutrition. *Nutr Metab Cardiovasc Dis.* 2015;25:1071–1078. doi: 10.1016/j.numecd.2015.09.010. [[PubMed](#)] [[CrossRef](#)] [[Google Scholar](#)]

10. Frega N, Mozzon M, Lercker G. Effects of free fatty acids on oxidative stability of vegetable oil. *J Am Oil Chem Soc.* 1999;76:325–329. doi: 10.1007/s11746-999-0239-4. [[CrossRef](#)] [[Google Scholar](#)]
11. Fritsch CW. Measurements of frying fat deterioration: a brief review. *J Am Oil Chem Soc.* 1981;58:272–274. doi: 10.1007/BF02582355. [[CrossRef](#)] [[Google Scholar](#)]
12. IUPAC . *Standard methods for the analysis of oils, fats and derivatives*. 6. Oxford: Pergamon; 1979. [[Google Scholar](#)]
13. Kail B, Link D, Morreale B. Determination of free fatty acids and triglycerides by gas chromatography using selective esterification reactions. *J Chromatogr Sci.* 2012;50:934–939. doi: 10.1093/chromsci/bms093. [[PubMed](#)] [[CrossRef](#)] [[Google Scholar](#)]
14. Kanicky JR, Shah DO. Effect of degree, type, and position of unsaturation on the pKa of long-chain fatty acids. *J Colloid Interface Sci.* 2002;256:201–207. doi: 10.1006/jcis.2001.8009. [[PubMed](#)] [[CrossRef](#)] [[Google Scholar](#)]
15. Keurentjes JT, Sluijs JT, Franssen RJH, Van't Riet K. Extraction and fractionation of fatty acids from oil using an ultrafiltration membrane. *Ind Eng Chem Res.* 1992;31:581–587. doi: 10.1021/ie00002a020. [[CrossRef](#)] [[Google Scholar](#)]
16. Lalas S. Quality of frying oil. In: Sahin S, Sumnu SG, editors. *Advances in deep-fat frying of foods*. Boca Raton: CRC Press; 2009. [[Google Scholar](#)]
17. Min DB, Boff JM. Lipid oxidation of edible oil. In: Akoh CC, Min DB, editors. *Food lipids: chemistry, nutrition, and biotechnology*. 2. Boca Raton: CRC Press; 2002. [[Google Scholar](#)]
18. ISC2016 CONFERENCE PROCEEDING BOOK final 16 June 2016.pdf
19. Rizwana Khatoon Soomro University of Sindh, January 2013
20. Fatty Acids Composition of Vegetable Oils and Its Contribution to Dietary Energy Intake and Dependence of Cardiovascular Mortality on Dietary Intake of Fatty Acids, *Int. J. Mol. Sci.* 2015

21. Determination of free fatty acids in crude vegetable oil samples obtained by high-pressure processes, Food Chemistry: X, ELSEVIER, 2021
22. Effect of refining on quality and composition of sunflower oil, J Food Sci Technol. 2015 Jul
23. THERMOPHYSICAL PROPERTIES OF CASTOR OIL (RICINUS COMMUNIS L.) BIODIESEL AND ITS BLENDS, un. 2015

CONFERENCE CERTIFICATE



