

**An Investigation on the Tribological Performance
of Carbon nanotube (CNT) as a Lubricant Additive
to Locally Made Engine Oil**

By

Nur Zeenaida Muhd Khiyar (5803)

Dissertation submitted in partial
fulfillment of the requirements for the
Bachelor of Engineering (Hons)
(Mechanical Engineering)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Mechanical Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfilment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(MECHANICAL ENGINEERING)

Approved by:

(How Meng Git)

UNIVERSITY TEKNOLOGI PETRONAS
TRONOH, PERAK
January 2008

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

NUR ZEENaida MUHAMAD KHIYAR

ABSTRACT

Lubricating oil is manufactured with the purpose to minimize friction and to control wear in machine elements such as gears, bearings, springs and beams. The components of machines nowadays become more complex in order to improve the production rate. These machines required lubricant for protection in order to avoid damages and giving stability in performance of machine. With the addition of additives to the lubricant, it makes the lifetime of the machine even longer. The additive would improve the performance of lubricating ability by enhancing the desirable tribology properties. This project is concerning on the usage of Carbon nanotube (CNT) as the potential additive to be added to the locally made lubricant i.e. MACH 5. The purpose of adding the CNT into the lubricant is to enhance the lubricant properties in terms of providing less friction for a sliding contact. Initially, this project starts with gathering information on internal combustion engine, lubricant and the additive candidate; CNT. For this project to prove that CNT might be the next candidate for lubricant additive, there will be two types of experiment to be conducted throughout this project i.e. viscosity test and four balls wear test. Viscosity test is to measures a lubricant's resistance to flow at certain temperature and is considered as oil's most important physical property. Four ball wear test on the other hand is the industry's standard test method for measuring the wear preventive characteristics of a lubricant. The smaller the average wear scar, the better the protection provided by the lubricant. The wear scar surface resulted form the four ball wear test is later to be analyzed with the scanning electron microscope (SEM) machine. At the end of this project CNT is aimed as the solution that could provide less friction to the surface contact and capable to withstand extreme pressure.

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ABBREVIATIONS AND NOMENCLATURES

ASTM	American Society for Testing and Materials
AW	anti-wear
CNT	Carbon nanotube
cSt	centistokes
EP	extreme pressure
rpm	revolution per minute
SEM	scanning electron microscope
UTP	Universiti Teknologi PETRONAS
wt %	weight percent

CHAPTER 1

INTRODUCTION

1.1 Background of study

Lubricant is an oily or slippery substance that is formulated from petroleum-base oil. It is mainly used to reduce the friction of working parts in machinery. Lubricant that being applied on object's surface is to provide the smooth movement of one surface over another and to prevent damages. Fishing reels and lawn mowers are among devices that require lubrication. Automobiles, buses, airplanes and trains are also dependent on lubrication to operate where the lubricant must be effective to maintain the lifetime and to protect the mechanical components of the machine.

The commercial lubricant nowadays is basically formulated from petroleum-base oil with the addition of chemical additives. Additives are chemical materials that are added to base oil which could improve the properties of lubricant. Among the advantages of adding the additives to the base oil are it works as the anti-wear agent, anticorrosion agent, capable to withstand extreme pressure and as friction modifier agent. This improvement of lubricant properties may help prolong the lifetime of machine components.

The addition of additive in lubricant is being concerned because of tribological properties. The study of friction, wear, lubrication and contact mechanics are all important parts of tribology. Quoted from the book of '*Engineering Tribology*' second edition by Gwidon W. Stachowiak and Andrew W. Batchelor;

“**Tribology**’ is derived from the Greek word; ‘**tribos**’ meaning rubbing or sliding.”

In other words, tribology may be defined as interaction of contacting moving surfaces when load is being applied.

1.2 Problem statement

Towards the twenty first century, development on technology has improved efficiency in most forms of machinery. These advances have led to faster machine speed, greater load-handling capability and higher machine temperature. In many lubricated machine elements such as gears, cams and piston rings, the friction and lifetime of the machine are greatly influenced by protection of lubricant. Although the machine elements are lubricated, the movement of the machine elements still causes friction which at the end, it affects the lifetime of the machine. Due to these advances of machine, it motivates an investigation on how to improve the tribological properties of lubricant. An experimental study on Carbon nanotubes (CNT) as additive in lubricant may be proposed as a solution for improvement in lubricant's tribology properties.

1.3 Objective

This experimental study on lubricant additive is to study the effect of CNT as an additive to engine oil in order to improve its tribological properties. By adding the additive to the base oil, it is hoped that it could withstand pressure and minimize the wear in a sliding steel-on-steel application which simulates the operating condition within internal combustion engines.

1.4 Scope of study

This project focuses on tribological performance of MACH 5 after the addition of CNT as the lubricant additive. Areas of study included in this project are tribology, internal combustion engine, lubricant and additive. There are two types of experiment conducted in this project which are viscosity test where it is to measure lubricant's resistance to flow at certain temperature and four ball wear test where it is to evaluate lubricant performance in terms of wear scar. From the experiment's results, the effectiveness of CNT as the lubricant additive can be analyzed.

CHAPTER 2

LITERATURE REVIEW

This literature review explores the three dominant themes of the research: internal combustion engine, lubricant and CNT, tests involved in the project and similar study by other researches. The scope in this experimental study will explain on the internal combustion engine works where it relates to how wear is resulted in an engine, information regarding to lubricant where it mostly concern about the types and its properties and lastly about the additive i.e. CNT.

2.1 Internal combustion engine

2.1.1 Four stroke engine

Internal combustion engines are most commonly used for mobile propulsion in automobiles, equipment, and other portable machinery [1]. With reference to Figure 1, here's what happens on a four strokes engine as it goes through its cycle:

I. Intake stroke

The piston starts at the top, the intake valve opens, and the piston moves down to let the engine take in a cylinder-full of air and gasoline (1a).

II. Compression

Then the piston moves back up to compress this fuel / air mixture. Compression makes the explosion more powerful (1b).

III. Explosion

When the piston reaches the top of its stroke, the spark plug emits a spark to ignite the gasoline (1ci). The gasoline charge in the cylinder explodes, driving the piston down (1cii).

IV. Exhaust

Once the piston hits the bottom of its stroke, the exhaust valve opens. The used air (smoke) from the explosion escapes through the outlet valve. As the piston reaches the top, the outlet valve closes (1d).

The cycle is then repeated.

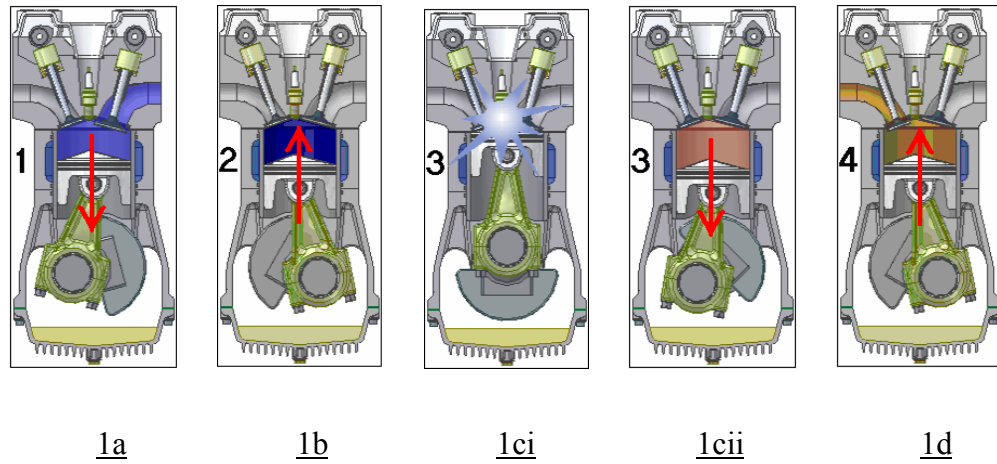


Figure 1: Schematic of an operational four stroke engine [1]

2.1.2 Operating consideration – Wear

Normally, engine wear is detected mostly at the wall of the cylinder and piston rings shown in Figure 2 [2]. Bearings and camshafts also experienced wear but barely.

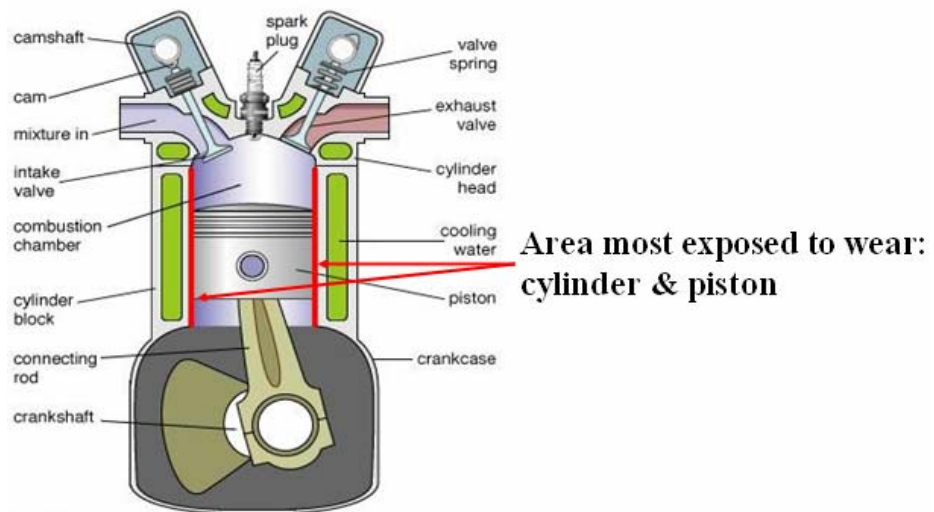


Figure 2: Most detected wear scar area in an engine

There are three common causes of wear; abrasion, metal-to-metal contact and corrosion. When the engine is at the air intake stage, the dust and dirt carried into the cylinders is hard and abrasive. Abrasive particles carried by the oil onto load-supporting surface of the cylinder walls and other areas can cause wear to the ring and cylinder. The rate of wear from this cause depends to the suitability of the lubricating oil. When the oil is with the correct viscosity and has sufficient antiwear characteristics, wear due to metal-to-metal contact can be controlled. A poor distribution of oil into the engine can contribute to increased rates of metallic wear.

Corrosion on the other hand happens when the rate of condensation is increased at the time the operating temperature in the engine is low. This condition is either during the warm-up period or stop-and-go operation where this can lead to corrosive wear. The corrosion wear is a result from the existence of water or combination of water, severe oil degradation products and corrosive end products of combustion.

2.2 Lubricant

Lubricants are used to reduce friction and wear between contacting surfaces. Fluids like oil and grease are the most common types of lubricant. It is mostly applied to any machinery parts such as gears, fishing reels, lawn mowers and automobiles. The purpose of applying lubricant to such components is to reduce friction in order to maintain the lifetime of the machine and to protect mechanical component of the machine.

The oil in an engine does more than just reduce friction between its moving parts. It also helps to seal the high-pressure combustion gases inside the cylinders, to slow down the corrosion of metal parts, to absorb some of the harmful by-products of combustion, and to transfer heat from one part of the engine to another.

2.2.1 Types of lubricants

There are two basic types of engine oil which are the conventional mineral-based oils and synthetic-based oils [3]. These two types of oil are different in terms of grade and weight of oil. Mineral-based oil has petroleum oil as its base stock while synthetic-based oil on the other hand consisting of chemical compounds which were not originally present in petroleum. Synthetic-based oil is superior to mineral-based oil in many ways such as high resistance to heat and higher film strength. Purpose of this project using the mineral-based oil is basically that this type of engine oil is not fully modified compared to the synthetic-based oil.

2.2.2 Viscosity

Viscosity is one of the most important physical properties of lubricating oil [2]. It is one factor responsible for the formation of lubricating films under both thick and thin film conditions. Viscosity affects heat generation in bearings, cylinders and gears due to internal fluid friction. It affects the sealing properties of oils and the rate of oil consumption. It determines the ease with which machines can be started at various temperatures, particularly cold temperatures.

Lubricant oils have long chain hydrocarbon structures, and viscosity increases with chain length. Viscosity is also a major factor in predicting the performance and fatigue life of rolling element such as bearings, cylinders and gears. The viscosity of oils is dependent upon temperature and pressure [4]. Viscosity decreases as temperature increases because the molecules vibrate more, and interact less. As for pressure, viscosity increases with pressure because the molecules are forced together causing greater interaction.

Hydrodynamic Lubrication

Hydrodynamic lubrication is a condition where the sliding surfaces are separated by relatively thick film of lubricant [5]. The hydrodynamic lubrication is to prevent wear in moving parts or contacted surface. The essential element in

hydrodynamic lubrication is the viscous properties of the lubricant because the higher the viscosity, the higher the friction of the lubricant [6]. Since the only friction present in a hydrodynamic lubrication system is the friction of the lubricant itself [7], therefore lubricant with the most appropriate viscosity is a must in order to minimize the friction.

2.2.3 MACH 5

The base oil that will be used for the experiment in this project is from a PETRONAS brand; MACH 5 (SAE 10W-30). Table 1 shows the product typical of MACH 5. PETRONAS Mach 5 SG is high-performance multi-grade engine oil specially designed and formulated to provide a high level of engine protection and performance in engines running under very severe driving conditions [8]. It is formulated with premium quality base oils and a carefully selected high-performance additive system to provide superior thermal and oxidation stability that resist oil thickening at very high operating temperatures, and to ensure superior deposits control and minimized engine wear and tear.

Table 1: Product typical and specifications of MACH 5 [8]

SAE Grade	MACH 5
Characteristics	10W-30
Density @ 15 °C, kg/l	0.8636
Pour Point, °C	-33
Flash Point, °C	228
Kinematic Viscosity, cSt	
@ 40 °C	72.9
@ 100 °C	11.2
Viscosity Index	146
Cold Cranking Visc, cP	
@ - 15 °C	-
@ - 25 °C	4,130
Sulphated Ash, %wt	0.81
TBN, mg KOH/g	6
ASTM Colour	3.5

2.2.4 Disposal of used engine oil

After certain miles as the car goes running, the engine oil needs to be changed. The used engine oil must never be disposed on the ground i.e. in the river or in the garbage [9]. The right thing to dispose used engine oil is to transfer to a clean leak-proof plastic bottle container or disposed it in the disposable barrels.

The used engine oil never wears out. It just gets dirty and it can be recycled, cleaned, and be used again. Currently, issue on the shortage of conventional hydrocarbon that is left on earth is being concerned. By recycling used engine oil, it could reduce the percentage of decreasing conventional hydrocarbon. Besides concerning about shortage conventional hydrocarbon, recycling could help to protect the environment rather than tossed the used engine oil into trash or in the river.

2.3 **Additive – Carbon nanotubes (CNT)**

Additive is a chemical substance added to a petroleum product to improve certain properties. Common petroleum product additives are, antifoam agent, anti-wear additive, corrosion inhibitor, detergent, dispersant, emulsifier, Extreme Pressure additive, oiliness agent, oxidation inhibitor, pour point depressant, rust inhibitor, tackiness agent, viscosity index improver [10]. Adding additives to the lubricant provides some improvements to the lubricant's properties in order to protect the engine. Some examples of improvement that has made to the engine oil are by adding anti-oxidant agents that could help to reduce the ability of oxygen from reacting with combustion by product, adding anti-wear agents which helps to reduce friction, and by adding detergent to the oil in order to clean the deposits of carbon and ash are that believed could increase engine wear.

2.3.1 Properties of Carbon nanotubes (CNT)

Carbon nanotubes (CNT) is a molecular-scale tube of graphitic carbon is which known as the stiffest and strongest material in terms of tensile strength and elastic modulus respectively [11]. The structure of a CNT is actually in a hexagonal network of carbon atoms that has been rolled up to make a seamless cylinder. Generally, there are two types of CNTs: single-walled carbon nanotubes (SWNTs) and multi-walled carbon nanotubes (MWNTs) where they are different in terms of cylindrical graphene layer [12]. For this project, MWNTs is the chosen additive material to be added to the lubricant due to the availability of the CNT here in UTP.

CNT applications include energy storage where CNT is being used as electrodes in batteries and capacitors [13]. Because CNT have high surface area, this makes it good in electrical conductivity. Field emission-based flat panel displays, novel semi conducting devices, chemical sensors, and ultra-sensitive electromechanical sensors are other application of CNT in electronic base. The properties of CNT that could provide in electronics are conductors, semiconductors and insulators [12].

The superior properties of CNTs are not limited to electrical but it also provides good mechanical properties, such as stiffness, toughness, and strength [13]. The tiny tubes can be used to produce extremely robust plastics that are constructed in the same way as reinforced concrete. This application is being applied in sports equipment such as baseball bat or ice hockey.

In fuel application, CNT works as the anti-knock additive which it helps to reduce the tendency of gasoline to detonate [14]. CNT also works as catalyst in accelerating burning rate for diesel fuel where it is to promote clean burning. Applying CNT to lubricant alternatively helps to increase the viscosity. Viscosity is important properties of lubricant because it determines the ease of engine to start at various temperatures. Since there are applications of CNT in fuel and lubricant,

it is hoped that by adding the CNT into lubricant, it also could help in reducing friction in an engine. Figure 3 shows the structure of a CNT.

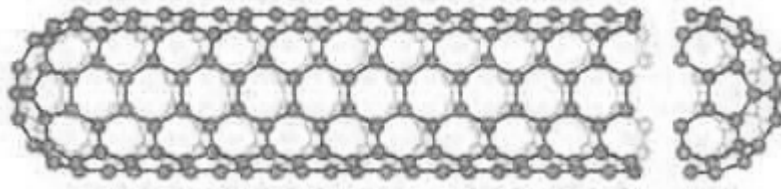


Figure 3: Structure of CNT [15]

2.4 Tests involved in this project

There are two types of test being conducted in this project, which are the viscosity test (ASTM D-445) and the four ball wear test (ASTM D-4172).

2.4.1 Viscosity test (ASTM D-445)

Purpose: Measures a lubricant's resistance to flow under gravity through a calibrated glass capillary viscometer at certain temperature [16].

Selecting ubbelohde tube:

Depending on lube grade, viscosity is tested at 40 °C and 100 °C. Referring to Table 1, selection of ubbelohde tube is base on the viscosity value of MACH 5 i.e. at 40 °C, the viscosity value is 72.9 cSt and at 100 °C the viscosity value is 11.2 cSt. Types of ubbelohde tube used in this test are as highlighted in Appendix 1.

The viscosity of the sample can be obtained by multiplying viscosity constant with the time taken for the fluid to flow through the calibrated region. The calculation on finding the viscosity for each sample is included on the 'Result and Discussion' part.

2.4.2 Four ball wear test (ASTM D-4172)

Four ball wear test is the common wear tester used by the oil industry to study lubricant chemistry. It has been used widely to study the lubricating properties of oils and to study chemical interaction of wearing contact [17].

The four ball wear test consists of three balls being clamped in a ball pot plus a fourth ball rotating on top of them with pressure pressed upon it. The balls are 12.7 mm in diameter. While the fourth ball rotating at the desired speed, load is being applied to the fourth ball which presses into the centre of the bottom three balls. The schematic of the four ball can be shown in Figure 4.

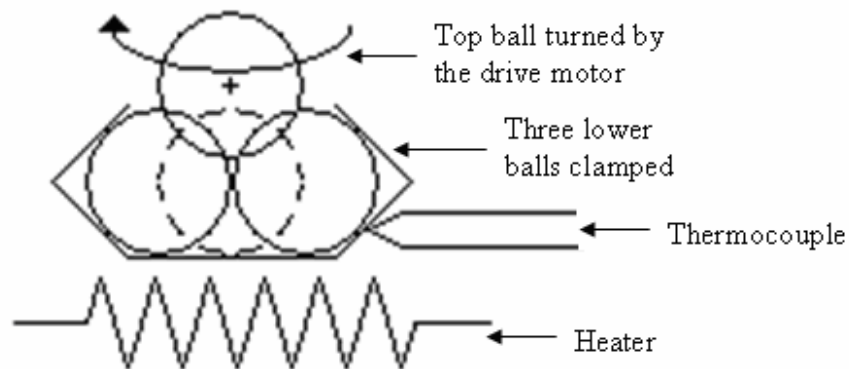


Figure 4: Schematic of four ball wear test [18]

The primary measurement resulted from the four ball wear test is the wear scar. The wear scar produced on the bottom three balls is measured by using optical microscope.

2.5 **Similar study by other researchers**

Figure 5 is an example result taken from *Oil, Rheology, Tribology and Driveline Fluids, SEA International* for a lubricant that being added with Zinc dialkyl dithiophosphates (ZDDP) as the additive [19]. From Figure 5, comparison can be made between the wear scar diameter on a ball lubricated with base oil (i.e. 500SN

type) and a ball lubricated with addition of ZDDP to the base oil at a load of 588 N. Figure 5a and 5b it is being captured by SEM from the same ball at the different magnification; i.e. at 100X and 500X. Same goes to Figure 5c and 5d.

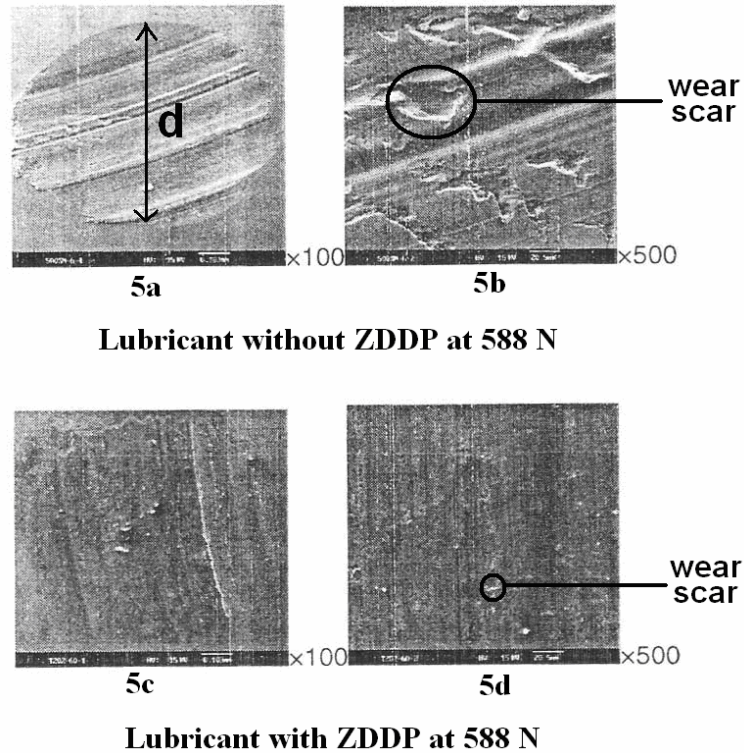


Figure 5: SEM of wear scar [19]

At magnification of 100X, the diameter of wear scar on 5a can be measured easily compared to 5c. The ball later is being zoomed at magnification of 500X to analyze the surface of the wear scar. From here, the wear scar on 5b easily detected compared to 5d. The effectiveness of a lubricant substance is measured by the wear scar diameter. The summary based on this study can be simplified by referring to Table 2.

Table 2: Summary of previous study on the usage of ZDDP as the lubricant additive

Figure	Magnification	ZDDP additive (wt %)	Description of wear scar
5a	100 X	0.00	The diameter of the wear scar can easily being measured
5b	500 X	0.00	The wear scars can easily being detected
5c	100 X	2.00	Hardly can measure the diameter of the wear scar
5d	500 X	2.00	Hardly can detect the wear scars

This experiment concluded that with the addition of ZDDP to the base oil, it helps to reduce friction. Through the research that has been made, there is no yet study on CNT as the lubricant additive that could help reduce in friction. Thus, this motivates undertaking the project to investigate on CNT as the lubricant additive.

CHAPTER 3

METHODOLOGY

3.1 Project flow

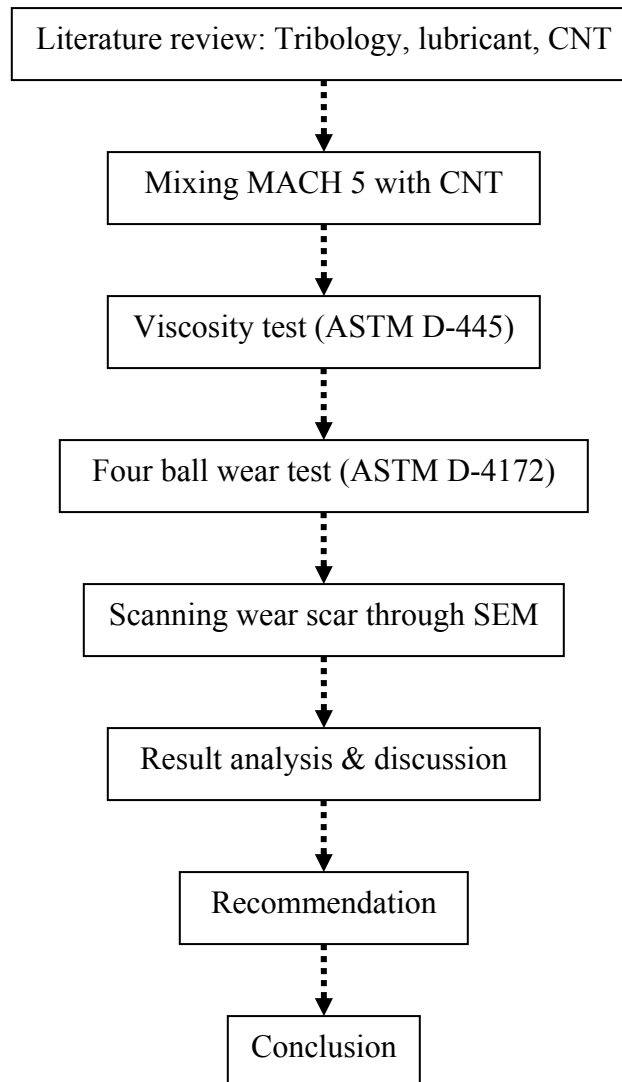


Figure 6: Project flow chart

The project flow chart is shown in Figure 6. Initially, literature review on the topic is carried out at the beginning of the project and also done continuously throughout the project. Literature review basically covers up topic on tribology, lubricant and the candidate additive, i.e. CNT. This project then continues with the first experiment which is mixing the MACH 5 with CNT by using the ultrasonic bath. The mixed samples are later to be tested with viscometer (ASTM D-445) which it is to analyze the difference in viscosity for each sample. After done gathering data form the viscosity test, this project continues with the four ball wear test (ASTM D-4172). Four ball wear test is actually to study the tribological properties of the lubricant after being added with CNT. The wear scar diameter resulted form the four ball wear test is later to be scan through Scanning Electron Microscope (SEM). All of the results from each experiment and tests need to be analyzed and this will provide a discussion. At the end of this project, recommendation will be included which it is for improving this project for future research.

3.2 Preparation on mixing the additive to the engine oil

On preparing the mixed sample of CNT with MACH 5, there will be two stages which are finding the weight percent (wt %) of CNT based on MACH 5 weight and mixing them by using the ultrasonic bath.

3.2.1 Stage 1: Determination on wt % of CNT

There are five samples of mixed lubricant to be tested in this project. Each of them, which contain of 50ml MACH 5 need to be different in terms of CNT concentration. In order to get the right amount of CNT to be added into MACH 5, below is the formula on finding the wt % of CNT:

$$\frac{\text{weight CNT (g)}}{\text{total weight MACH 5 (g)}} \times 100 = X \text{ wt \% CNT} \quad (1)$$

Table 3 is to show the amount of CNT that need to be added to 50ml of MACH 5. After setting up all of the five samples, it is ready for the next stage which is mixing the sample using ultrasonic bath.

Table 3: Calculation on finding the wt % of CNT

	Beaker + 50 ml MACH 5	Weight MACH 5 (g)	Weight CNT (g)
0.02 wt % CNT Beaker = 92.4081 g	134.4949	42.0868	$CNT(g) = \frac{0.02 \text{ wt}\%}{100} \times 42.0865$ $CNT = 0.0084 \text{ g}$
	134.4949	42.0868	
	134.4940	42.0859	
	Avg:	42.0865	
0.04 wt % CNT Beaker = 104.1959 g	142.2408	38.0449	$CNT(g) = \frac{0.04 \text{ wt}\%}{100} \times 38.0451$ $CNT = 0.0152 \text{ g}$
	142.2411	38.0452	
	142.2411	38.0452	
	Avg:	38.0451	
0.06 wt % CNT Beaker = 96.0143 g	129.9313	33.9170	$CNT(g) = \frac{0.06 \text{ wt}\%}{100} \times 33.9161$ $CNT = 0.0203 \text{ g}$
	129.9303	33.9160	
	129.9297	33.9154	
	Avg:	33.9161	
0.08 wt % CNT Beaker = 96.1105 g	133.5014	37.3909	$CNT(g) = \frac{0.08 \text{ wt}\%}{100} \times 33.3906$ $CNT = 0.0299 \text{ g}$
	133.5008	37.3903	
	133.5010	37.3905	
	Avg:	33.3906	
0.10 wt % CNT Beaker = 97.7424 g	136.9974	39.2550	$CNT(g) = \frac{0.10 \text{ wt}\%}{100} \times 39.2516$ $CNT = 0.0393 \text{ g}$
	136.9930	39.2506	
	136.9917	39.2493	
	Avg:	39.2516	

3.2.2 Stage 2: Mixing of CNT to MACH 5 using ultrasonic bath

Ultrasonic bath is a device for mixing chemical liquid which it uses vibration that being sent through water to mix them [20]. The beaker that is filled with the sample liquid is partially immersed in the tub that contains water for it to be mixed. This mixing operation is conducted for an hour. Figure 7 shows the device of an ultrasonic bath.

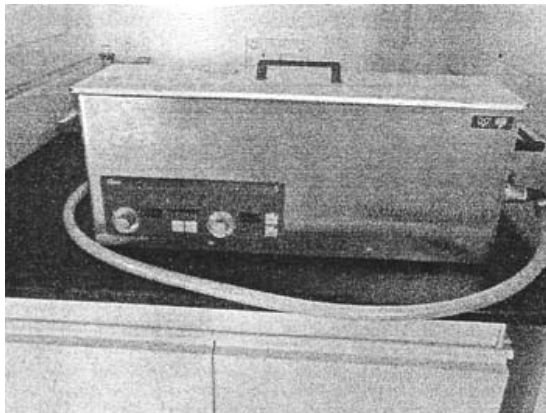







Figure 7: Ultrasonic bath [21]

Apparatus that required for the mixing samples are:

- Ultrasonic bath device
- Beaker
- Weight electric scales
- Spatula

Details on preparing the samples are shown on table 4.

Table 4: The mixing process using the ultrasonic bath

1	 <p>(a)</p>	<p>Pour 50 ml of MACH 5 into 200 ml beaker.</p>
2	 <p>(b)</p>	<p>Weight the lubricant by using the weight electric scale for three times to get the average weight of the lubricant.</p>
3	 <p>(c)</p>	<ul style="list-style-type: none"> - Use Equation 1 to get the ideal weight percent of CNT that need to be added into the lubricant. Use spatula to scoop the CNT. - Figure (c) shows the condition of the sample with the addition of CNT before the mixing process.
4	 <p>(d)</p>	<ul style="list-style-type: none"> - Place all the samples that need be mixed into the ultrasonic bath and immerse them partially. - This mixing process will take about an hour for the samples to be fully mixed together.
5	 <p>(e)</p>	<p>Figure (e) shows the condition of the mixture sample after the mixing process.</p>

3.3 Tests

There will be two types of tests to be conducted through out this project. The first test to be conducted is the viscosity test where it is to analyze the lubricant's viscosity properties. It is then followed by four ball wear test which the result will be analyzed with optical microscope and SEM. The samples that have been tested with four ball wear test needs to go through viscosity test again. The second running test for the viscosity is to analyze if there is any difference in viscosity compared to the previous test. Below are the details on the methodology:

3.3.1 Viscosity test (ASTM D-445)

Viscosity test is to measure lubricant's resistance to flow at a specific temperature by using a viscometer. The viscosity of a lubricant is being measured on how long does it takes for a volume liquid to flow under gravity through a calibrated glass capillary [22]. The lubricant will be tested at two different temperatures which are 40 °C and 100 °C. Viscosity is measured at 40 °C for industrial applications and 100 °C for engine oil applications. When the oil is being heated with the desired temperature, it will flow down the tube and up to the other side. The number of seconds the oil takes to flow through the calibrated region is measured and the unit for viscosity is centistokes (cSt).

Apparatus that required for the viscosity test are:

- Viscometer device (Figure 8)
- Ubbelohde tube (Figure 9)
- Stopwatch



Figure 8

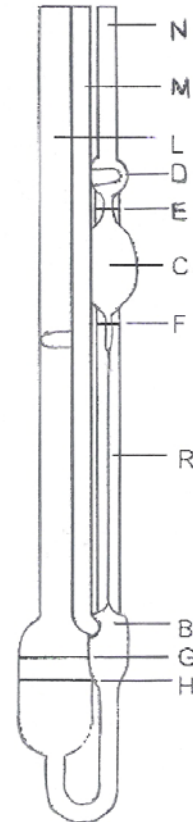


Figure 9

For this project, ubbelohde tube type 2 and 1C is chosen based on the MACH 5's viscosity range as discussed on the literature review part. Tube 2 is use for temperature at 40 °C while tube 1C is for 100 °C respectively. Both tubes have their own viscometer constant where for tube 2, the value is $0.1\text{mm}^2/\text{s}^2$ and as for tube 1C is $0.03\text{mm}^2/\text{s}^2$. The calculation on finding the viscosity for each sample is included in 'Result and Discussion' part. With reference to Figure 9, procedure on how the viscosity test is conducted as follows [23].

1. Set the viscometer temperature to be at 40 °C.
2. Pour the lubricant sample through tube L until the sample brings to the level between lines G and H.
3. Place the ubbelohde tube into the temperature bath and allow approximately 20 minutes for the sample's temperature to be stable with the temperature bath.

4. Place finger over tube M and apply suction by using the vacuum pressure unit to tube N until the liquid reaches the center of bulb D.
5. Remove suction from tube N as well as finger from tube M.
6. Measure efflux time starting the liquid reaches line E until line F.
7. Repeat steps 2 to 6 for other samples.

3.3.2 Four Ball Wear Test (ASTM D-4172)


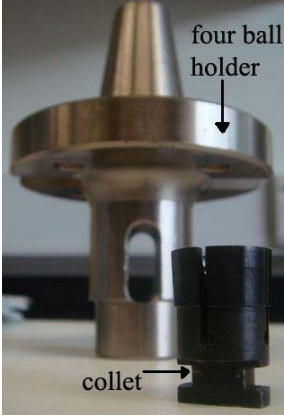
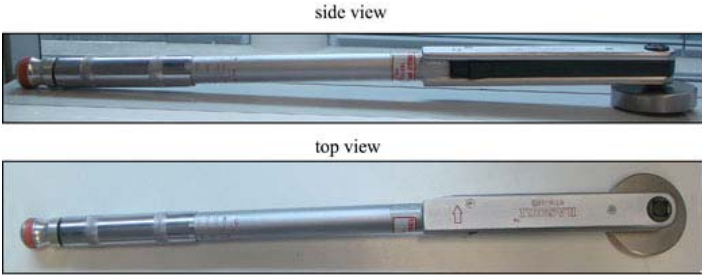
The ASTM D-4172; Four Ball Wear Test is a device that being used to test the antiwear and extreme pressure characteristics of lubricant oils in sliding steel-on-steel applications [24]. Figure 10 is to show the four ball wear test equipment.



Figure 10: Four ball wear test equipment

Apparatus that required for the ASTM D-4172 test are shown in table 5.

Table 5: ASTM D-4172 apparatus and their function

No	Apparatus	Function
1	 <p data-bbox="581 646 943 680"><u>Figure 11: Ball pot and ring</u></p>	To clamp the three bottom balls
2	 <p data-bbox="521 1119 1003 1152"><u>Figure 12: Four ball holder and collet</u></p>	To hold the fourth ball
3	 <p data-bbox="558 1455 894 1488"><u>Figure 13: Torque wrench</u></p>	To tighten the ball pot

Four ball wear test consist of three steel balls which covered with the lubricant sample that will be evaluated are clamped together and placed in a triangular pattern [17] shown in Figure 14. The fourth ball will be place on the top of the rest three balls and force will be applied to it. After setting up these four balls, it is later to be placed in the four ball wear test equipment.

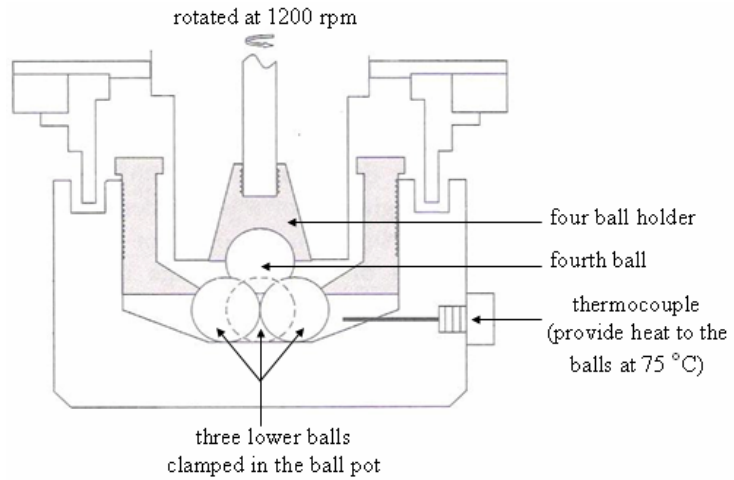



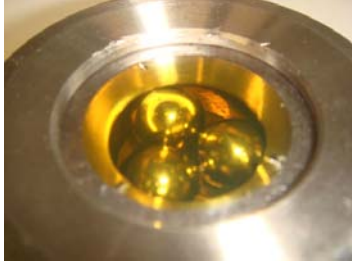
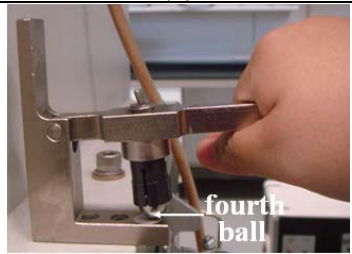
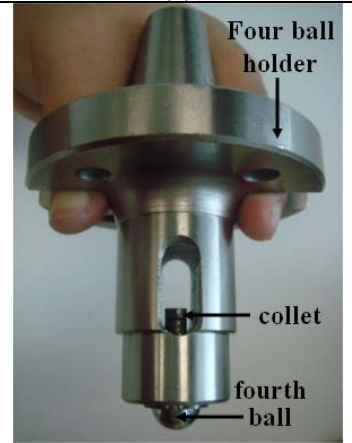
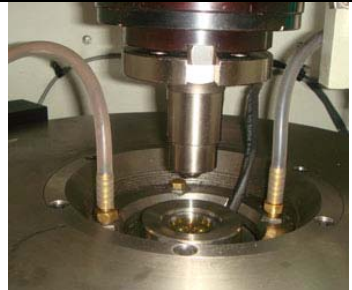


Figure 14: Schematic of four ball wear test equipment [17]

Details on preparing the apparatus are shown in table 6.

Table 6: Setting up the apparatus for the ASTM D-4172 test

1	 <p>(a)</p>	<ul style="list-style-type: none"> - Place the three balls inside the ball pot as shown in (a).
1	 <p>(b)</p>	<ul style="list-style-type: none"> - Place ring inside the ball pot before clamping it (b).
2	 <p>(c)</p>	<p>Tighten the ball pot with a torque wrench.</p>

3	 <p style="text-align: center;">(d)</p>	<p>Pour the lubricant sample inside the ball pot. The balls inside the ball pot must be fully submerged in lubricant as shown in (d).</p>
4	 <p style="text-align: center;">(e)</p>	<p>Lock the fourth ball inside the collet by using the collet extractor which is located next to the machine.</p>
5	 <p style="text-align: center;">(f)</p>	<p>Place the collet that already contains the fourth ball into the four ball holder as shown in (f).</p>
6	 <p style="text-align: center;">(g)</p>	<ul style="list-style-type: none"> - After setting up the apparatus, position the apparatus according to Figure (g) i.e. ball pot to be place at bottom while the fourth ball holder is at top. - Close the lid of the machine before starting the experiment.

Before starting the experiment, the computer must be logged in to the WinDUCOM application software in order to set up the experiment's parameters i.e. temperature, speed, load and time taken for the experiment to be conducted. Figure 15 shows the setting up for the WinDUCOM software.

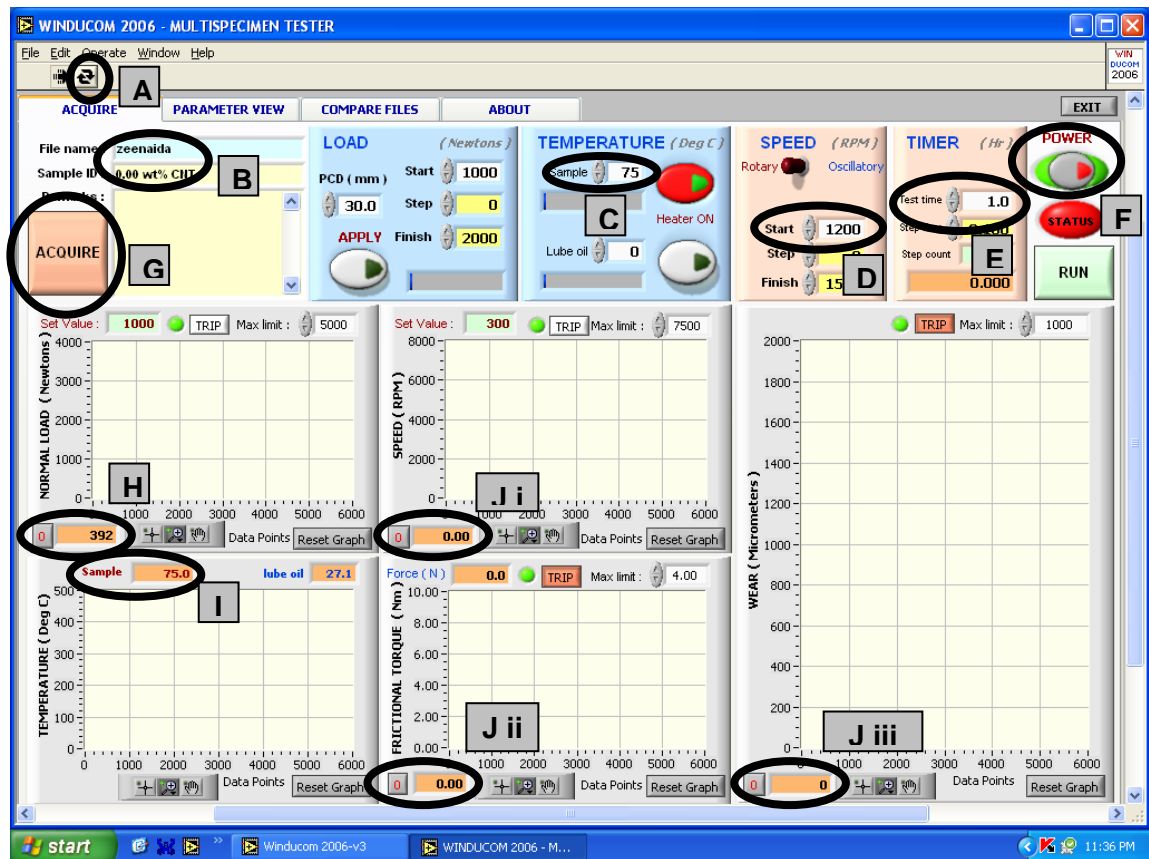


Figure 15: Setting up the WinDUCOM application software

The procedure is as follows [25]:

1. Click on the mode run continuously {A}.
2. Enter the file name and set all the parameters accordingly. For this experiment, the temperature is to be set at 75 °C and speed is to be running at 1200 rpm for about an hour {B - E}.
3. Press the power button; {F} and this followed by the acquire button; {G}.
4. To set the load for the experiment; {H}, adjust the load located on the right side of the machine shown in Figure 16.

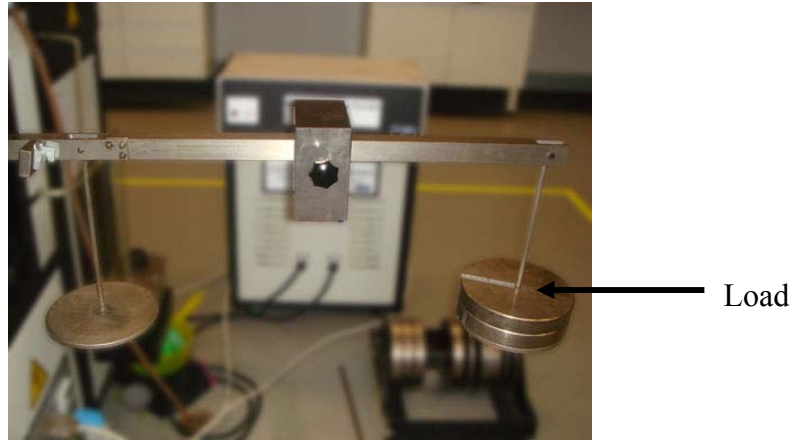


Figure 16: Load alignment.

5. Allow the machine to reach the temperature stability; {I} for about 30 minutes before starting the experiment.
6. When the machine is ready to run, set the speed, frictional torque and wear to zero; {Ji, Jii, Jiii}.
7. Click the run button.

As for the load, there are two types of weight which are 392 N and 588 N. The reason to have different loads is to observe, which lubricant sample could withstand extreme pressure. Therefore, each lubricant sample shall be running two tests at two different loads.

3.4 Evaluating the results from ASTM D-4172

Evaluating results from the ASTM D-4172 require two types of assessment which are optical microscope and the SEM machine. The optical microscope is to measure the diameter of the wear scar while the SEM machine is to capture the surface condition of the wear scar. Below are the details on the evaluation.

i. Optical microscope

The use of optical microscope as indicated in Figure 17 is to measure the wear scar on the three lower clamped balls. To assure the accuracy of the wear scar measurement, three data of measurement based from the balls that

have wear scar should be taken. From the three measurements, the average scar diameter is determined.

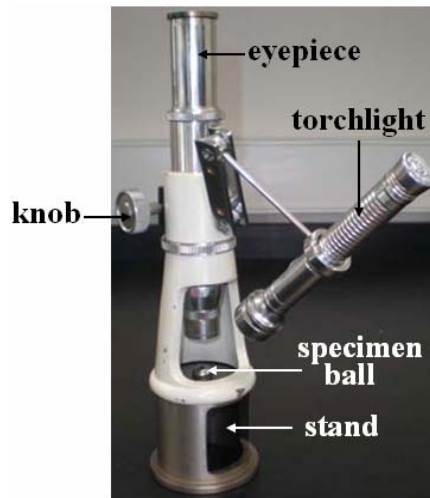


Figure 17: Optical microscope

ii. SEM – Scanning Electron Microscope

The scanning electron microscope (SEM) as shown in Figure 18 is a type of electron microscope capable of producing high-resolution images of a sample surface [26]. With SEM, the surface of the steel balls that has wear scar can be analyzed. Figure 19 shows an example of wear scar being captured using the SEM machine.



Figure 18: SEM machine [27]

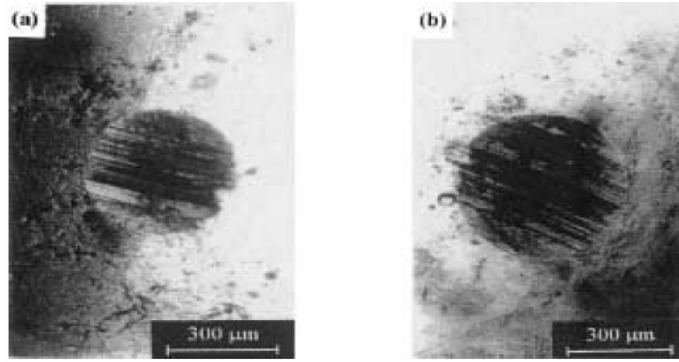


Figure 19: Example topographies of the wear scar on the ball by SEM [28]

In conclusion, the lower the average wear scar diameter, the better the wear protection provided by the lubricant. This same goes to the condition of the wear scar surface; i.e. the smoother the surface, the better the protection provided by the lubricant.

CHAPTER 4

RESULT AND DISCUSSION

This chapter discusses on the result from the experiments that has been conducted which are the viscosity test as well as the four balls wear test. After analyzing the result, few recommendations are presented for improvement.

4.1 Viscosity test (ASTM D-445) result

Shown in Table 7, there are six samples with different concentration of CNT being tested with viscometer at the temperature of 40 °C and 100 °C. From the viscosity test, result obtained is plotted in Graph 1.

Table 7: Viscosity test result on different concentration of CNT

Sample contain CNT (wt %)	Temp. (°C)	Viscometer constant (cSt/s)	Efflux time (s)			Average time (s)	Viscosity (cSt)
			1	2	3		
0.00	40	0.10	653	654	653	653.33	65.33
	100	0.03	351	350	352	351.00	10.53
0.02	40	0.10	658	658	657	657.67	65.76
	100	0.03	357	353	354	354.67	10.64
0.04	40	0.10	670	669	670	669.67	66.96
	100	0.03	362	360	361	361.00	10.83
0.06	40	0.10	672	673	672	672.33	67.23
	100	0.03	363	369	363	365.00	10.95
0.08	40	0.10	685	686	684	685.00	68.50
	100	0.03	377	374	376	375.67	11.27
0.10	40	0.10	707	710	702	706.33	70.63
	100	0.03	385	378	383	382.00	11.46

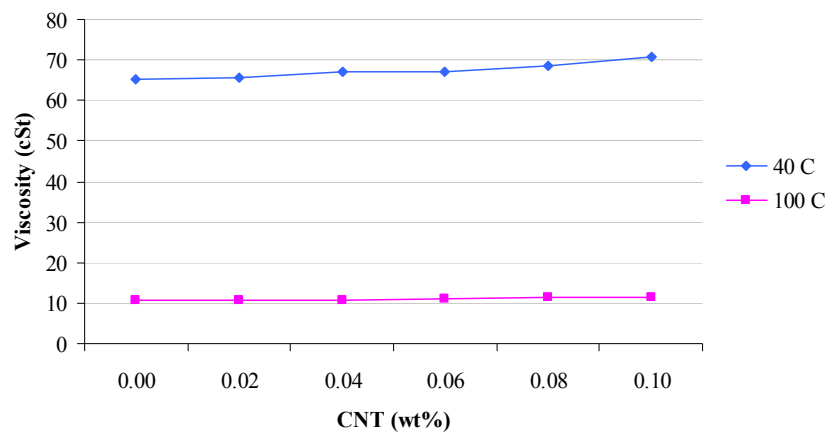
The average viscosity value of MACH 5 with addition of CNT at 40 °C is 67.40 cSt while at 100 °C is 10.95 cSt as shown in Table 8. This is to show that although with addition of CNT into MACH 5, the viscosity value is still acceptable as it doesn't exceed the original viscosity value. The original viscosity value of MACH 5 is taken from Table 1.

Table 8: Comparison viscosity value of MACH 5 without and with addition of CNT

Temperature	MACH 5	MACH 5 + CNT
40 °C	72.90	67.40
100 °C	11.20	10.95

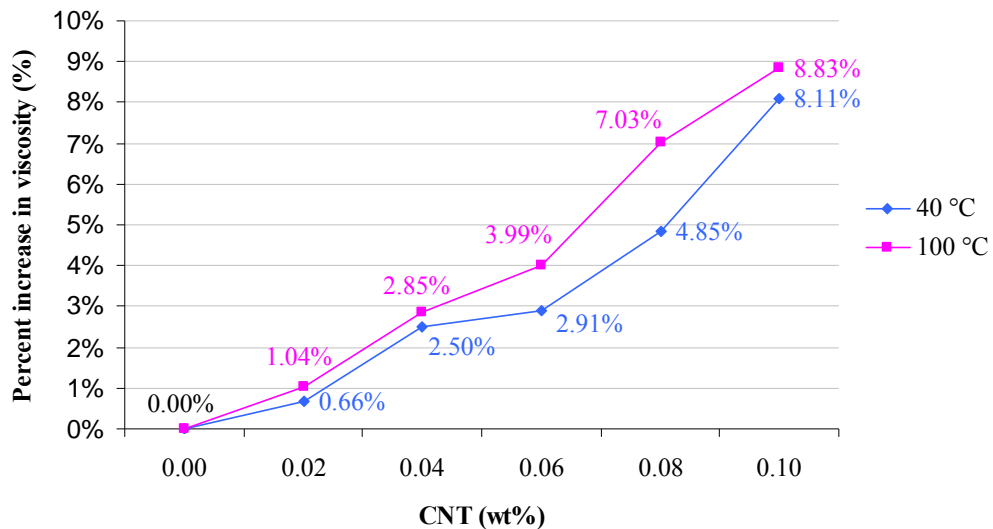
Result from the viscosity test shown in Graph 1 indicates there is increment in viscosity as the number of wt % CNT increases in the lubricant sample. For the same CNT concentration, the viscosity at 40 °C has greater value compared to at 100 °C. This implies that, at lower temperature, the fluid takes more time to flow through the calibrated region hence, the fluid is more viscous. When the temperature is low, the molecules of the sample travel at the average speed due to less kinetic energy compared to at higher temperature [29].

Besides temperature, the viscosity increase as the wt % of CNT in the sample increases. When CNT added into the lubricant, the sample tends to be thicker. Great concentration of CNT in the sample causes intermolecular forces between MACH 5 molecules with CNT molecules. Intermolecular forces are the forces of attractions that exist between molecules in a mixture [30]. Therefore, these two types of molecules have more interactions among themselves. This is the reason why the sample that has a higher wt % of CNT tends to flow slower.



Graph 1: Viscosity (cSt) vs. wt % of CNT

The trend shown in Graph 1 may not be noticeable. In order to amplify the increase in viscosity, Graph 2 shows the percent increase in viscosity with 0.00 wt % CNT as the base line. Both temperatures result in increase of viscosity percentage as the CNT concentration increases. From Graph 2, it is noted that the line exhibit a similar trend. By using simple graphical methods, this trend can be established to be used for predicting the viscosity of samples with varying wt % of CNT.



Graph 2: Percent increase in viscosity (%) vs. wt % of CNT

During the experiment few precautions shall be considered. In order to get an accurate result for the viscosity test, the viscosity devise should be given sufficient time to allow the temperature bath to stabilize, which is normally at least 20 minutes. Besides temperature, user needs to be alert during the whole time taken for the oil to flow at the calibrated region. Another thing that needs to be considered while handling this test is to make sure that the ubbelohde viscometer tube is clean and free from other substance. Such substances are like water or small particles that could affect the lubricant's flow at the calibrated region inside the ubbelohde tube. In order to reuse the ubbelohde tube again for other samples, it needs to be rinsed with petrol and fully dried.

4.2 Four ball wear test (ASTM D-4172) result

Four ball wear test is conducted to determine the antiwear and extreme pressure characteristics of lubricant oil in sliding steel-on-steel application. All samples which contain different concentration of CNT were evaluated based on two different weights i.e. 392 N and 588 N. This project runs four ball wear test twice in order to determine the repeatability of the result. Table 9 shows the result based on the first run while Table 10 is for the second run.

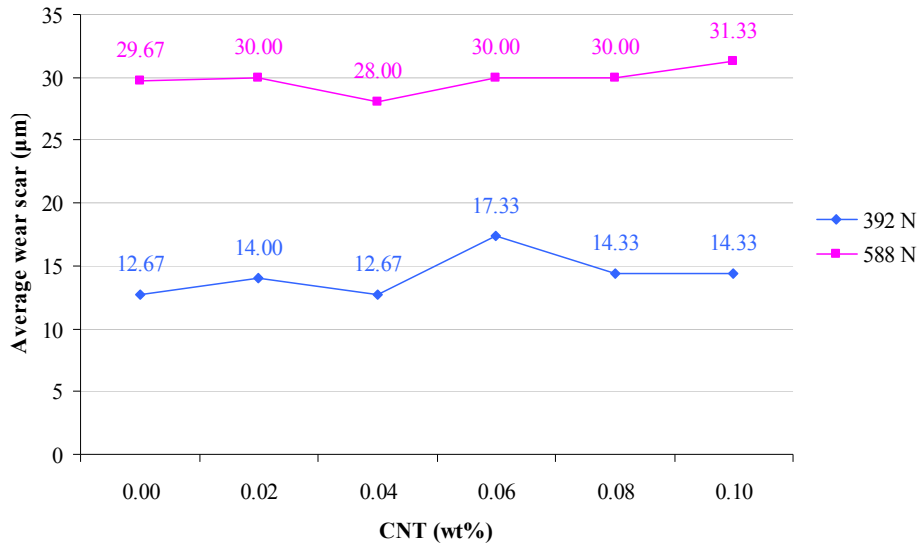
Table9: Four ball wear test result on different concentration of CNT (first run)

Sample (CNT wt %)	Load (N)	Wear scar (μm)			Average wear scar (μm)
		1	2	3	
0.00	392	11	13	14	12.67
	588	30	25	34	29.67
0.02	392	15	11	16	14.00
	588	33	26	31	30.00
0.04	392	10	13	15	12.67
	588	31	23	30	28.00
0.06	392	16	15	21	17.33
	588	36	26	28	30.00
0.08	392	15	11	17	14.33
	588	35	30	25	30.00
0.10	392	12	15	16	14.33
	588	34	33	27	31.33

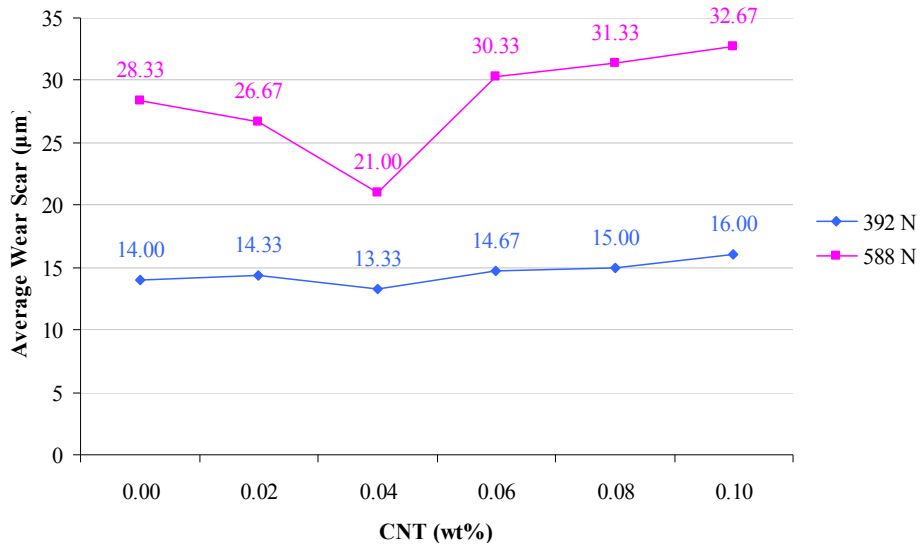
Table10: Four ball wear test result on different concentration of CNT (second run)

Sample (CNT wt %)	Load (N)	Wear scar (μm)			Average wear scar (μm)
		1	2	3	
0.00	392	15	13	14	14.00
	588	28	27	30	28.33
0.02	392	12	15	16	14.33
	588	27	25	28	26.67
0.04	392	12	13	15	13.33
	588	23	22	18	21.00
0.06	392	14	14	16	14.67
	588	30	32	29	30.33
0.08	392	12	17	16	15.00
	588	31	34	29	31.33
0.10	392	16	15	17	16.00
	588	32	32	34	32.67

Graph 3 is plotted based on data from Table 9 which resulted from the first run of the test. Graph 4 is based on the second run test. Referring to Graph 3 and 4, at lower load the wear scar diameter is smaller compared to at higher load. Both graphs also show that sample 0.04 wt % of CNT shows reduction in wear scar.

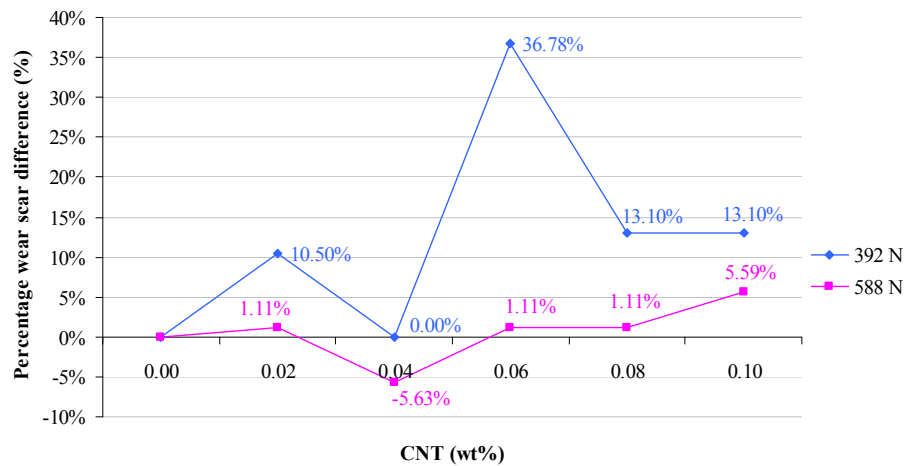


Graph 3: Average Wear Scar (μm) vs. wt % of CNT (first run)



Graph 4: Average Wear Scar (µm) vs. wt % of CNT (second run)

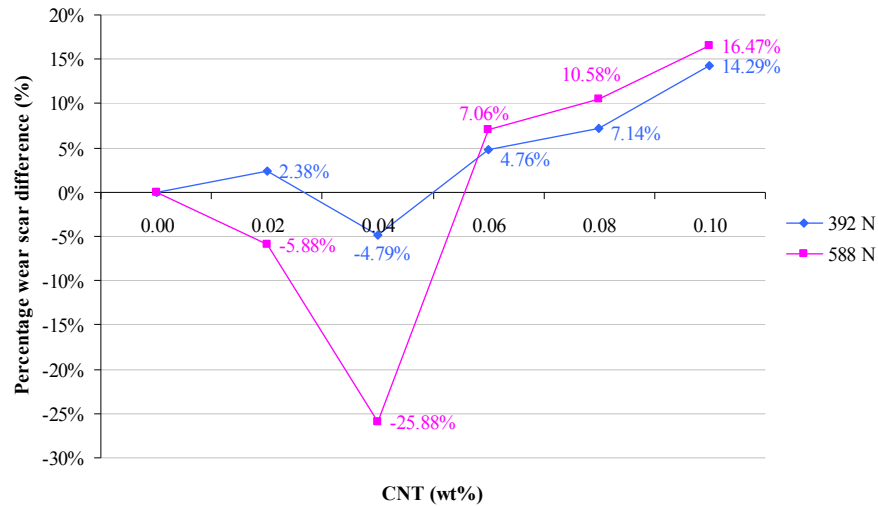
Graph 5 shows the percentage difference in wear scar for the first run of the test with sample 0.00 wt % of CNT as the baseline. From the first run, only sample 0.04 wt % of CNT shows reduction in wear scar which is by 5.63% at the load of 588 N. As for other samples, they do not provide wear protection to the balls, as indicated by increment in wear scar.



Graph 5: Percentage difference in wear scar (%) vs. wt % of CNT (first run)

Graph 6 shows the percentage difference in wear scar for the second run of the test. The second run test shows two samples that have reduction in wear scar i.e. 0.02

wt % and 0.04 wt % of CNT. At load of 392 N, sample 0.04 wt % of CNT shows reduction in wear scar by 4.79%. At load of 588 N, sample 0.02 wt % reduced by 5.88% while sample 0.04 wt % reduced by 25.88%.



Graph 6: Average wear scar percent (%) vs. wt % of CNT (second run)

Both Graph 5 and 6 show wear scar diameter increases as the concentration of CNT increase from 0.06 wt % of CNT. When the additive is excessive, this will result in worse lubricating effectiveness, and the wear scar diameter will become bigger. At this point, it can be said that the optimum concentration of CNT in providing improvement to the lubricant tribological properties is at the range of 0.02 wt % up to 0.04 wt % of CNT.

Every object's surface is uneven though it looks smooth. This is described as the surface roughness. Real rock surfaces can be described as similar models of surface roughness. Surface roughness is an important factor when dealing with issues such as friction, lubrication and wear. Reduction in wear scar diameter is caused by the protection provided by the lubricant. Assumption can be made that lubrication is primarily controlled by the hydrodynamic fluid mechanism [17]. If the diameter of the wear scar is small resulted from the four ball wear test, this is due to the high thickness of lubricant film where it provides protection to the mating wear surfaces. The film thickness could serve as spacers or gap in

preventing rough contact between the two mating wear surfaces. This mechanism can be described as illustrated in Figure 20.

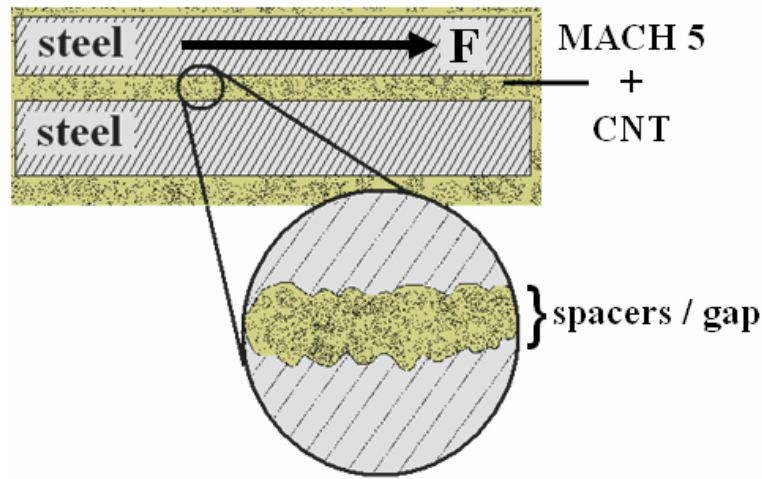


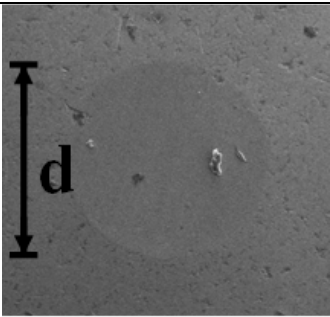
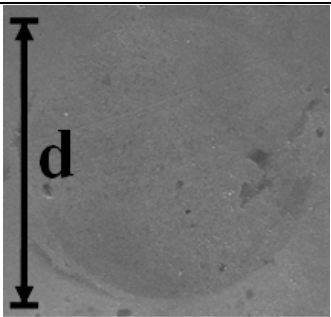
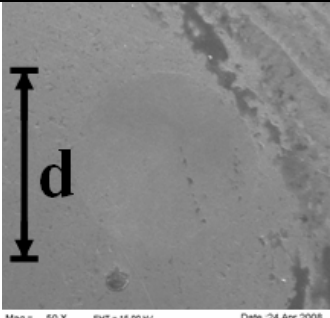
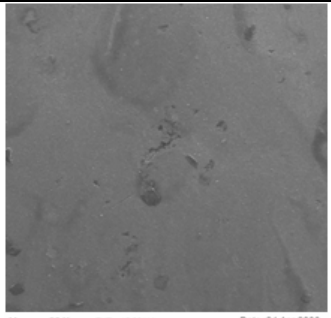
Figure 20: Schematic of the surface roughness being coated with lubricant sample

When handling this test, it also required few precautions that need to be considered in order to obtain reliable result. The first thing that need to be considered is to make sure that all apparatus is clean and free from any other substance. This is to ensure the integrity of the lubricant sample and prevent contamination. Before commencing the test, the lower three balls must be fully submerged in the lubricant sample. This ensures a realistic stimulation of the working condition within an engine. The parameters need to be set up accurately and the device should be given ample time to allow the temperature to reach at stable value. Normally, the stabilizing time is about 30 minutes.

4.3 Scanning Electron Microscope (SEM) result

Scanning Electron Microscope (SEM) is a type of microscope used to capture images of an object for surface condition analysis. The selection of balls is base on the balls which has been tested with sample 0.00 wt % and 0.04 wt % of CNT. These two types of concentration are selected because sample 0.00 wt % of CNT used as the base line for comparison with sample 0.04 wt % of CNT. Table 10 shows the image captured by using the SEM at the magnification of 50X.

Table 11: SEM image of wear scar at magnification of 50X

Load \ Sample	392 N	588 N
0.00 wt % CNT	 <p>(a)</p>	 <p>(b)</p>
0.040 wt % CNT	 <p>(c)</p>	 <p>(d)</p>

The summary based on Table 11 can be simplified by referring to Table12.

Table12: Summary of diameter wear scar base on Table 11

Figure	CNT additive (wt %)	Load (N)	Description of wear scar diameter
a	0.00	392	The diameter of the wear scar can easily be measured
b	0.00	588	The diameter of the wear scar can easily be measured and its bigger due to greater load
c	0.04	392	Slightly hard to measure the diameter of the wear scar
d	0.04	588	Hardly to measure the diameter of the wear scar

CHAPTER 5

CONCLUSION AND RECOMMENDATION

CONCLUSION

The purpose of applying lubricant to an engine is to provide smooth movement in order to minimize wear and to prevent it from damages. Although the engine has lubricated with engine oil, it still causes friction which at the end, it affects the performance and the lifetime of the engine. Thus, this project proposed that by adding CNT to locally made engine oil i.e. MACH 5, it could reduce friction in the engine. To analyze the effectiveness of a lubricant that has addition of CNT, two experiments have been conducted through out this project which are the viscosity test and the four ball wear test. Viscosity test is to measure lubricant's resistance to flow at certain temperature. According to ASTM D-445, viscosity test will measure the lubricant resistant at the temperature of 40 °C and 100 °C. The common wear tester used by the oil industry to study the lubricant tribological properties is by test the lubricant using the four ball wear test.

Based on the result of the experiment, it was concluded that by adding the additive to the base oil the lubricant could withstand pressure and minimize the wear in a sliding steel-on-steel application which simulates the operating condition within internal combustion engines. From the viscosity test, the greater the wt % of CNT in the lubricant, the higher is the viscosity. This is indicated by the time the lubricant takes to flow through the calibrated region. At high concentration of CNT in the lubricant, intermolecular forces between MACH 5 molecules with CNT molecules is greater. The viscosity values of the samples are also acceptable since it doesn't exceed the original viscosity value of MACH 5.

For the four ball wear test, the first run test shows a reduction in wear scar by 5.63% for sample 0.04 wt % of CNT at the load of 588 N. The second run test has two samples with reduced wear scar i.e. 0.02 wt % and 0.04 wt % of CNT. At load of 392 N, sample 0.04 wt % of CNT shows reduction in wear scar by 4.79%. At

load of 588 N sample 0.02 wt % reduced by 5.88% while sample 0.04 wt % reduced by 25.88%. The experimental outcome indicates a probable optimum range of CNT concentration i.e. 0.02 wt % up to 0.04 wt % of CNT. At this optimum range, it shows that by adding CNT to MACH 5 it could enhance wear protection. This result can motivate further investigation to determine the optimum concentration of CNT and to promote CNT as an additive candidate for enhanced tribological performance.

RECOMMENDATIONS

There are still many aspects and areas in lubricant additive that need to be studied to a greater extent. The following are some recommendations that can be considered in the future for further improvement.

1. *Further investigation on other additives that could improve lubricant properties.*

This project has proposed CNT as the lubricant additive and the experimental result supports the feasibility of candidate for antiwear agent and extreme pressure agent. For future investigation, other chemical substances can be experimented on to improve lubricant properties.

2. *Study on other chemical substance that could enhance the effectiveness of CNT as the lubricant additive.*

This experimental study on improving the lubricant properties is being tested with only addition of CNT into the lubricant. For a wider scope of investigation, experiments can be conducted by adding other chemical substance as the catalyst in enhancing the effectiveness of CNT.

3. *Four ball wear test to be repeated to gauge the repeatability of result obtain.*

This project has only managed to run the four ball wear test for two times only. The reason by repeating the test for few runs is to determine the repeatability result pattern. By having similar result pattern, effectiveness of the additive can be confirmed.

4. *Improve the color of the product.*

After the addition of CNT into the MACH 5, the color of the lubricant has changed from light yellowish to black color. Most consumers would prefer a light color of lubricant compared to darker color. This is due to misconception on lubricant's color as an indication of how dirty it is. In order to fulfill the consumer satisfaction i.e. preferable on light lubricant color, further investigation on chemical substance is another recommendation that could improve the color of the product.

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APPENDIXES

Appendix 1:

Selection of ubbelohde tube

Size	Viscometer constant mm^2/s^2 (cSt/s)	Kinematics viscosity range mm^2/s (cSt)	
0	0.001	0.3 to 1	
0C	0.003	0.6 to 3	
0B	0.005	1 to 5	
1	0.01	2 to 10	
1C	0.03	6 to 30	→ 100 °C
1B	0.05	10 to 50	
2	0.1	20 to 100	→ 40 °C
2C	0.3	60 to 300	
2B	0.5	100 to 500	
3	1.0	200 to 1000	
3C	3.0	600 to 3000	
3B	5.0	1000 to 5000	
4	10	2000 to 10000	
4C	30	6000 to 30000	
4B	50	10000 to 50000	
5	100	20000 to 100000	

Appendix 2: Gantt chart for the Final Year Project I

No.	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14	
1	Selection of Project Topic								Mid-Semester Break								
2	Preliminary Research Work																
3	Submission of Preliminary Report																
4	Seminar I																
5	Project Work - Reference / literature - Practical / laboratory work - Preparing on progress report and seminar II																
6	Submission of Progress Report																
7	Seminar II																
8	Project work continues - Reference / literature - Practical / laboratory work - Preparing on Interim Report Final Draft and oral presentation																
9	Submission of Interim Report Final Draft																
10	Oral Presentation																

Appendix 3: Gantt chart for the Final Year Project II

No.	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14	
1	<u>Project Work Continue</u> - Practical / laboratory work - Preparing on Progress Report I								Mid-Semester Break								
2	Submission of Progress Report I																
3	<u>Project Work Continue</u> - Practical / laboratory work - Preparing on Progress Report II and seminar I																
4	Submission of Progress Report II																
5	Seminar I																
5	<u>Project work continue</u> - Practical / laboratory work - Preparing on poster, dissertation and final presentation																
6	Poster Exhibition																
7	<u>Project work continue</u> - Practical / laboratory work																
8	Submission of Dissertation (soft bound)																
9	Oral Presentation																