CHARACTERIZATION AND PRODUCT IDENTIFICATION OF SLUDGE OBTAINED FROM THE RECYCLING OF LUBRICANT INTO BASE OIL

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BORANG PENGESAHAN STATUS TESIS

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SESI PENGAJIAN: **2005/2006**

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CHARACTERIZATION AND PRODUCT IDENTIFICATION OF SLUDGE OBTAINED FROM THE RECYCLING OF LUBRICANT INTO BASE OIL

LOSHBINAN A/L THAVASIGAMANI

Thesis submitted to the Faculty of Chemical and Natural Resources Engineering in
Partial Fulfillment of the Requirement for the
Degree of Bachelor Engineering in Chemical Engineering

Faculty of Chemical & Natural Resources Engineering
Universiti Malaysia Pahang

APRIL 2008

I declare that this thesis entitled "*Characterization And Product Identification Of Sludge Obtained From The Recycling Of Lubricant Into Base Oil*" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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To my beloved father and mother

ACKNOWLEDGEMENT

I would like to express my humble thanks to God for the strength, inspiration and encouragement given to me throughout the completion of this thesis without any obstacle. A lot of experiences and knowledge were gained along the way.

I wish to express my sincere appreciation to my supervisor, Professor.Dr. Rosli Mohd Yunus for his critics, advices and input of ideas as well as relentless support, guidance and endless encouragement for this research project to be completed. I would like to express my heartfelt thanks to Pn. Sazwani Suhaimi as the co supervisor and also Cik Aini the coordinator for Industrial Project 4BKC for helping me to stay on the tasks concerning the preparation and the completion of the research and thesis.

I am also very thankful to my father, Mr.T.Thavasigamani, my mother, Mrs.P.Andaal Devi, family members, and all my friends for their advice and motivation. Without their endless support and encouragement, this thesis would not have been presented here. Unfortunately, it is not possible for me to list all involved in this limited space. I am grateful to everybody that is involved directly or indirectly in helping me to complete this thesis.

ABSTRACT

This study describes the characterization and product identification of sludge that is obtained from the recycling of spent lubricant into base oil. Sludge is produced from the recuperation of spent lubricant which involves two main processes. The first process is to produce the solvent that will be used. The solvent consists of Hexane, Isopropanol (IPA) and Potassium hydroxide (KOH). The next process is the recuperation of used lubricant using the solvent produced. The ratio of spent lubricant and solvent used is 1:4. Physical mixing is then used to produce a natural reaction between the solvent and spent lubricant. The mixture is stirred continuously for 30 minutes. The stirred mixture is then filtered. There are two ways to filter the mixture. One is by centrifugation and the other is filtration with thin filter papers. The residue that is left behind is the sludge that is going to be used for the characterization process. Many parameters of the sludge is investigated and characterized before product identification is done. Both physical and chemical parameters are investigated. The parameters investigated are pH, density, viscosity, heavy metal content and chemical composition of the sludge. The pH of sludge on average is 6.59 with an average density of 0.9041 g/ml or 904.13 kg/m^3. The average viscosity is 1419 cP. There is a fair amount of heavy metals present in the sludge and its chemical composition consists of paraffin and olefins. From the results obtained, the sludge can be singled out for its major components and the contents can be derived into useful products. Three different products are identified. The sludge can be used as raw material for pyrolysis where the end product obtained is a diesel like liquid and also as concrete or cement additive. The sludge can also be used as grease.

ABSTRAK

Kajian yang dijalankan adalah tentang analisis dan pengenalpastian produk daripada sisa mendapan minyak yang terhasil daripada proses kitar semula minyak pelincir yang telah digunakan. Minyak pelincir yang telah digunakan dikitar semula menjadi minyak asas untuk penghasilan minyak pelincir. Sisa mendapan minyak yang dihasilkan melibatkan dua proses iaitu proses penyediaan pelarut dan seterusnya proses memisahkan sisa mendapan minyak dari pelarut. Pelarut yang digunakan dihasilkan dengan mencampurkan Heksana, Isopropanol (IPA) dan Kalium hidroksida (KOH). Pelarut yang terhasil ditambah kepada minyak pelincir terpakai dengan nisbah 4 bahagian pelarut kepada 1 bahagian minyak pelincir. Terdapat 2 cara untuk mengasingkan larutan minyak asas daripada sisa mendapan minyak yang terhasil. Proses pengasingan ialah dengan proses sentrifugasi dan juga dengan penapisan menggunakan kertas turas yang nipis. Sisa yang tertinggal ialah sisa mendapan minyak yang akan dianalisis. Parameter yang dikaji ialah dari segi fizikal dan kimia sisa mendapan minyak yang terhasil. Parameter yang dikaji ialah pH, kelikatan, ketumpatan, kandungan logam berat dan juga komposisi kimia sisa mendapan minyak. pH sisa mendapan minyak ialah 6.59 dengan ketumpatan sebanyak 0.9041 g/ml atau 904.13 kg/m^3. Kelikatan sisa mendapan minyak ialah 1419 cP. Sisa mendapan miyak yang terhasil mengandungi sisa logam berat dan komposisi kimia yang dikenalpasti ialah ia terdiri daripada parafin dan olefin. Dari keputusan kajian yang dibuat,produk daripada sisa mendapan minyak boleh dikenal pasti. Tiga produk yang boleh terhasil daripada sisa mendapan minyak. Sisa mendapan minyak boleh digunakan sebagai bahan mentah untuk proses pirolisis untuk penghasilan cecair seperti diesel dan juga sebagai penambah untuk simen dan konkrit. Sisa mendapan minyak juga boleh digunakan sebagai gris.

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LIST OF ABBREVIATIONS

KOH - Sodium Hydroxide

IPA - Isopropanol

RPM - Revolution Per Minute

HC - Hydrocarbon

AAS - Atomic Absorption Spectrometer

FAAS - Flame Atomic Absorption Spectrometer

IR - Infrared

FTIR - Fourier Transform Infrared

cP - Centi Poise

ppm - Particles Per Million

µm - Micrometer

MSDS - Material Safety Data Sheet

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CHAPTER 1

INTRODUCTION

1.1 Research Background

A lubricant is a substance introduced between two moving surfaces to reduce the friction and wear between them. A lubricant provides a protective film which allows for two touching surfaces to be separated and "smoothed," thus lessening the friction between them. The lubricant must be replaced when it has dissolved to its saturation point, because the inability to dissolve additional abrasive debris allows abrasive particles to scrape against or become lodged in the working surfaces, thus introducing a margin for physical contact between them.

One of the single largest applications for lubricants, in the form of motor oil, is to protect the internal combustion engines in motor vehicles and powered equipment (Boughton and Horvath, 2003). Typically lubricants contain 90% base oil and less than 10% additives. Vegetable oils or synthetic liquids such as hydrogenated polyolefin, esters, silicone, fluorocarbons and many others are sometimes used as base oils. Additives deliver reduced friction and wear, increased viscosity, improved viscosity index, resistance to corrosion and oxidation (El-Fadel and Khoury R. M., 2004).

Base oil is nowadays produced by recycling spent lubricants. The recycling of lubricants to get base oil produces sludge as a by-product. Sludge is the residual semi-solid material left from industrial treatment processes (Norris S. and McCalmont M., 2005).

Oil sludge or black sludge is a solid or gel in motor oil caused by the oil gelling or solidifying. Sludge can be a major contributor to engine problems, and can require the engine to be replaced if the damage is severe. Sludge is usually caused by the presence of water in the oil, and can accumulate with use. Ways to minimize sludge production and accumulation include performing frequent oil changes, using synthetic oil, and following the manufacturer's engine maintenance routine (Synthetic Performance Solutions, 2007-2008).

Base oil and sludge separation covers a broad spectrum of separation processes. There are many ways employed for the separation depending on the situation. The base oil and sludge exist as an emulsion naturally. The base oil and sludge also contains the solvent that is used for the recycling process. Therefore further separation like evaporator is needed to produce base oil and sludge. If allowed to stand the emulsion will separate because base oil is lighter than the oil sludge. This process however is very slow and needs a lot of time for the separation to occur. The separation will also not be complete by natural gravitation force.

There are several ways for separation of sludge such as centrifuge and also through traditional method of filtration using thin filter paper. Thin filter paper is used as the oil sludge is in its semi solid state and it is easily separated.

1.2 Problem statement

It is estimated that 40 percent of all lubricants are released into the environment regarding its state whether it is in solid, semi-solid or liquid state. One of the lubricant wastes that are disposed is the sludge which is formed from the long usage of lubricants and also as a by-product from the recycling process. This sludge cannot be reused and this makes it to be disposed. The sludge contains a fair amount of oil and also heavy metals in it.

With today's knowledge, understanding and laws it is clear that to prevent soil and water contamination, we must properly dispose of oil wastes. Lubricants both fresh and used can cause considerable damage to the environment, mainly due to their high potential of serious water pollution. Further, the additives typically contained in lubricant can be toxic to flora and fauna.

There are ways to dispose used lubricants but it is not a safe way as it possesses its own hazards after the used lubricants are treated. Recycling, burning, landfill and discharge into water may achieve disposal of used lubricant. There are typically strict regulations in most countries regarding disposal in landfill and discharge into water as even a small amount of lubricant can contaminate a large amount of water. The lubricant can also be burned as fuel to generate electricity. Burning generates both airborne pollutants and ash rich in toxic materials, mainly heavy metal compounds.

Unfortunately, most lubricant that ends up directly in the environment is due to general public discharging it onto the ground, into drains and directly into landfills as trash. Other direct contamination sources include runoff from roadways, accidental spillages, natural or manmade disasters and pipeline leakages.

Improvement in filtration technologies and processes has now made recycling a viable option now with the rising price of base stock and crude oil. This recycling process produces sludge as a by-product. Typically various filtration systems remove particulates, additives and oxidation products and recover the base oil. The oil may get refined during the process. This base oil is then treated much the same as virgin base oil. However there is considerable reluctance to use recycled oils as they are generally considered inferior. The cause for this consideration by the public is because the lack of public awareness towards the usage of recycled base oil. More and more public awareness programs should be carried out to educate the people about the advantage of recycling. This will create more user friendly recyclers among the public thus recycling will be a viable option and there will be an increase in the usage of recycled base oil.

1.3 Objective

1. To obtain oil sludge in a lab scale.
2. To determine the suitable sludge filtration method in order to obtain large quantity of sludge.
3. To analyze the contents of oil sludge from the recycling process of lubricant into base oil
4. To identify the uses of oil sludge as a useful product.

1.4 Scope of Research

The scope of this experiment is to obtain oil sludge and to analyse the contents of it from the recycling process of lubricant into base oil. To achieve the objectives of this experiment, the following aspects have been identified:

i. Production of oil sludge by performing small scale base oil recuperation from used lubricant
ii. Determination of oil sludge characteristic using pH meter, Viscometer, Atomic Absorption Spectrometer (AAS) and Fourier Transform Infrared (FTIR).
iii. To identify the uses of oil sludge as a useful product.

1.5 Rationale and significance

Used oil is one of the most common wastes produced. In fact, approximately 4 billion gallons are generated every year worldwide. Used oil is generated by vehicle maintenance and repair facilities as well as various industrial areas. It is illegal to dispose of used oil in sewers, storm drains, waterways, on the ground or in the trash. Used oil can contain harmful contaminants that can work their way into our ground and surface waters and cause serious pollution. Just one gallon of used oil can contaminate a million gallons of drinking water. To cope with the polluting factor, used lubricant oil is recycled into base oil. The process produces oil sludge as a by-product and this creates another problem as the sludge contains a fair amount of oil and also heavy metals in it. The use of base oil from the recycled lubricant is also far cheaper if compared to the virgin base oil that is produced. Therefore it is a better way to recycle used lubricant and find a useful product for the sludge that is obtained as a by-product.

CHAPTER 2

LITERATURE REVIEW

It is estimated that less than 45% of used engine oil is being collected worldwide while the remaining 55% is thrown by the end user in the environment (Boughton and Horvath, 2003).

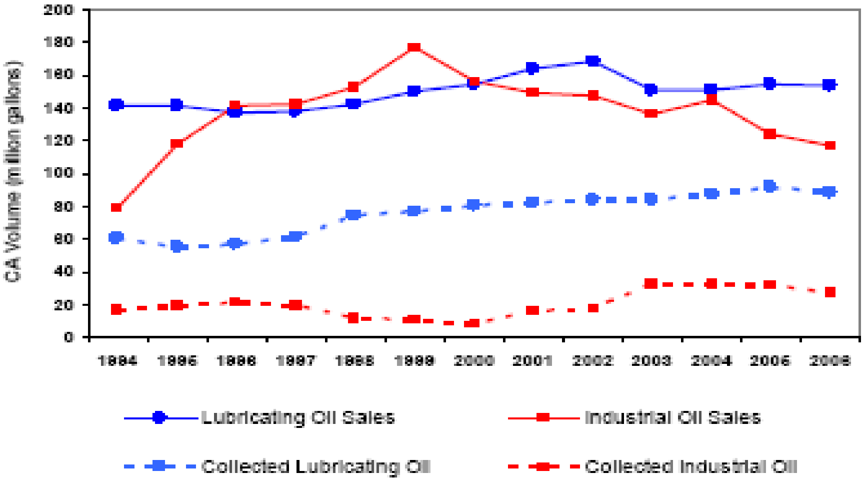


Figure 2.1: Oil sales and used oil collection trends, 1994-2006 (Tocci and Lisa, 2008).

Oil sludge or black sludge is a solid or gel in motor oil caused by the oil gelling or solidifying, usually at temperatures lower than 100^0C. Sludge can be a major contributor to internal combustion engine problems, and can require the engine to be replaced, if the damage is severe. Sludge is usually caused by the presence of water in the oil, and can accumulate with use. Ways to minimize sludge production and accumulation includes performing frequent oil changes, using synthetic oil and following the manufacturer's engine maintenance routine (Norris S. and McCalmont M., 2005).

Sludge formation begins when suspended particles and other contaminants begin to settle out of the oil. Most oils contain additives to manage the contaminants, but if the oil is not changed often enough the additives will become overwhelmed. This happens when there are too many contaminants to handle or when the oils chemical properties are weakened from over use. Additives deplete over time from oxidation and heat resulting in changes to the chemical makeup of the oil itself (Kaufman M., 2002).

This sludge then begins to deposit on engine parts, obstructing the flow of oil and the proper transfer of heat away from metal parts. Oil sludge will begin to blanket the surfaces of the engine and clog oil channels eventually resulting in oil starvation to parts of the engine. When an engine runs, it takes in air from its current environment, mixes it with fuel and ignites it. The air and fuel mixture undergoes a chemical reaction and several by-products are produced. These by-products make their way into the engine oil through cylinder blow-by and exhaust gas recirculation and further contaminating the oil (Kaufman M., 2002).

Engine oils often contain detergents and dispersants as part of their additive packages which suspend and in some cases neutralize these contaminants. Even in such cases, the additive package can only handle so much contamination before these by-products begin depositing on engine parts. Sludge formation begins when the chemically suspended particles of contaminants, including the metal particles from engine wear, begin to settle out of the oil depositing anywhere the oil flows (Kaufman M., 2002).

Another cause of sludge is running the engine low on oil for a prolonged period of time. The detergent and oxidation inhibitors are important components of the additive package that prevent sludge from forming. When an engine is run low on oil, there are less additive available to protect the engine, suspending particles and preventing oxidation (thickening) of the oil. Running an engine just 15-20% low for about 55 hours can increase the viscosity, or the thickness, of the oil by over 1000%. When engine oil gets this thick, it's a safe bet that the additive packet is shot and the oil is no longer actively suspending contaminants or neutralizing corrosives. This causes your oil to effectively damage your engine and not protecting it (Kaufman M., 2002).

Oil sludge from the internal combustion engine is the breakdown product of over-stressed oil. Oil that is stressed by contaminants and oxidation has to work thousands of miles longer than it was designed to and it will break down into a gel that sticks to the engine parts. As the sludge sticks, there is less good oil to circulate and do its protective job. This coating of gel also stores heat instead of releasing it which stresses the radiator and cooling system (Synthetic Performance Solutions, 2007-2008).

Every year, more of my customers suffer through unnecessary and very expensive engine re-builds due to oil sludge. The causes are complex but sludge damage can be prevented. The problem has been controlled by the type of oil used and also the invention of different engine designs (Norris S. and McCalmont M., 2005).

2.1 Advantages and Disadvantages of Recycling Spent Lubricant into Base Oil

2.1.1 Advantages

Cars, trucks, farm machines and boats all need regular lubricating oil changes. While some engines such as two-stroke engines burn oil completely, others like motor vehicle engines and machinery produce large volumes of used oil that can be reclaimed and reused. Used oil should not be thrown away. Although it gets dirty, used oil can still be cleaned and re-used. In fact, recycled used oil can be used as an industrial burner fuel, hydraulic oil, incorporated into other products or re-refined back into new lubricating oil (El-Fadel and Khoury R. M., 2004).

Used oil is hazardous. Any spill or mismanagement in the disposal of the used lubricating oil can pose as a severe hazard to the environment and the surroundings. Lubricating oil picks up a variety of hazardous contaminants when used in engines and transmissions including lead, dioxins, benzene and polycyclic aromatics (Boughton and Horvath, 2003).

Mankind, the proud creator of wastes is anxiously watching today the harmful effects of its own creation on the environment and ultimately on its existence in this beautiful world. Scientists have developed different technologies like incineration with power production, pyrolysis, pelletization, dumping in landfill area, recycling of non-biodegradable wastes and composting of biodegradable wastes (Diaz L. F. and Golueke C. G., 1985). The first four are destructive approach while last two are constructive approach with value addition to wastes.

The challenge is to combine this with continuing economic growth in a way which is sustainable over the long term (Odum HT., 1996). The technologies of recycling have been developing in order to cope with increasing prices of oil. It is now more viable for the recycling process to take place as there is severe depletion in the natural resources of crude petroleum. It is because there are a lot of advantages that can be gained by recycling spent lubricant into base oil. The advantages are discussed below:

2.1.1.1 Slows the consumption of natural resources

We know that recycling involves the processing and usage of the core elements of an old product for the production of new products. This helps in saving our natural resources to a great extent. Making a brand-new product without any recycled material causes natural resources to deplete in the manufacturing process. For example, less crude petroleum is used to produce lubricant if more and more used lubricant is recycled and reused. This way, proper recycling can help us preserve our natural resources for our future generations and maintain the balance of the nature (Chian *et al.*, 1976).

2.1.1.2 Recycling Saves Space for Waste Disposal

Most of the landfill sites are filled up with a lot of waste products that could have been recycled effectively. Some of these waste materials belong to non-biodegradable category which takes a long time to decompose. Recycling enables proper usage of these waste products and saves space for landfills. The pace with which landfills are getting filled up, soon we might run short of landfills unless we start following recycling at our own home and spread the word to others. One of the main reasons for recycling is to reduce the amount of garbage sent to landfills. Even though modern sanitary landfills are safer and less of a nuisance than the open dumps of the past, no one likes having a landfill around. In heavily populated areas, landfill space is scarce. Where space is plentiful, filling it with garbage isn't a very good solution to the problem (Diaz L. F. and Golueke C. G., 1985).

2.1.1.3 Recycling uses less energy

There is room for debate on this aspect of recycling, but many recycling processes require less energy than it would take to manufacture the same item brand-new. When new products are manufactured from the raw material obtained from recycled products, it saves a lot of energy which is consumed for the production. When new products are manufactured from ‘virgin materials’ the amount of energy consumed is much higher. Besides, the energy required to acquire and transport the ‘virgin’ raw materials from their origins or natural sources is also saved. Add to that the energy which is required to clean and protect the environment from the pollutant waste products, especially those which are non-biodegradable (plastic) and fill up the landfill areas. Energy use is a factor weighed on a case-by-case basis (Odum HT., 1996).

2.1.1.4 Recycling saves environmental conditions and reduces pollution

Recycling helps in preventing global climate change to a great extent. By minimizing the energy spent on industrial production, recycling also helps in reducing greenhouse gas emission. All these emit harmful gases such as methane, sulfur dioxide, carbon dioxide to the environment. The processing of fresh raw material also creates toxic materials which pollute the environment. By reducing the energy used, recycling also minimizes the amount of fuel usage which in turn reduces the amount of harmful pollutants in the environment (Shie J. L. *et al.,* 2003).

Recycling also reduces pollution from landfill leachate. Landfills cause other problems in addition to taking up lots of space. The assortments of chemicals thrown into landfills causes leachate as a result of garbage break down. Used oil is a known source to cause leachate as dumping of used oils will create an easier way for the leachate to form. This is because the oil is in liquid form and blends with other toxic material which later seeps into the ground easily. Leachate can drain out of the landfill and contaminate groundwater supplies. Today, impermeable clay caps and plastic sheeting prevent much of this run off, making the landfills much safer than they were just a few decades ago. Still there are leachate that seep pass this barriers and pollute the environment (Albers H. and Mennerich A., 1986).

2.1.1.5 Economic Benefits

Similar to energy and natural resource, recycling also helps in saving a lot of expense, demanded for the production of new products from ‘virgin’ materials.

These expenses include the entire production cycle starting from acquiring the raw materials, transferring them from their origin to production places, processing and manufacturing costs (Bartone C. R., 1990).

Recycling process creates employment opportunities for a lot of people, involved in the various stages of the process. This in turn contributes to the economic development of the state or country.

Recycling has a variety of economic impacts. For the companies that buy used goods, recycle them and resell new products, recycling is the source of all their income. For cities in densely populated areas that have to pay by the ton for their landfill usage, recycling can shave millions of dollars off municipal budgets. The recycling industry can have an even broader impact. Economic analysis shows that recycling can generate three times more revenue per ton as landfill disposal and almost six times as many jobs (Diaz L. F. and Golueke C. G., 1985).

2.1.2 Disadvantages

The reprocessing of recovered materials is not always pollution free. Certain reprocessing technologies create residues which are difficult to treat. The acid-clay process for re-refining waste oil is one example where the residual sludge has contaminated land. Whether the use of recycled material is less polluting can only be assessed on a project by project basis (Boughton and Horvath. 2003).

The costs of collection transport and reprocessing may be a disadvantage. This also results in higher costs for recycled materials.

The emphasis upon packaging means that a great deal of effort goes into extending the recycling of materials which account for only one tenth of total urban waste by weight. It also means that the focus is upon products such as used lubricant which are conspicuous in litter. Even a small part of used lubricant in the waste stream will have major adverse environmental effects. A drop of oil can spread on the surface of water in a big scale causing the lack of oxygen transfer in the water thus killing organisms in it. Some of these recycling schemes may bring no net gain to the community (Shie J. L. *et al.,* 2003).

2.1.2.1 No hassle free collection centre

A number of different systems have been implemented to collect recyclates from the general waste stream. These systems tend to lie along the spectrum of trade-off between public convenience and government ease and expense. The three main categories of collection are "drop-off centers", "buy-back centers" and "curbside collection" (Bartone C. R., 1990).

Drop-off centers require the waste producer to carry the recyclates to a central location, either an installed or mobile collection station or the reprocessing plant itself. They are the easiest type of collection to establish, but suffer from low and unpredictable throughput. Buy-back centers differ in that the cleaned recyclates are purchased, thus providing a clear incentive for use and creating a stable supply. The post-processed material can then be sold on, hopefully creating a profit. Curbside collection encompasses many subtly different systems, which differ mostly on where in the process the recyclates are sorted and cleaned. A waste collection vehicle generally picks up the waste (Bartone C. R., 1990).

There is still no specific way of collecting or distributing used oil in order to recycle it. This poses as a hassle to automobile repair shops as well as to other sources where the used lubricant can be obtained. Therefore the government should come up with a sound and collective ruling that states the way of disposal for used oils. The implementation of new rules will increase the awareness of people in the benefits of recycling thus increasing the recycling rate (Bartone C. R., 1990).

2.1.2.2 Mixed waste collection causes further problem

At one end of the spectrum is mixed waste collection, in which all recyclate are collected, mixed in with the rest of the waste, and the desired material is then sorted out and cleaned at a central sorting facility. This results in large amounts of recyclable waste being too soiled to reprocess. Public education is needed to change the mindset of people (Shie J. L. *et al.*, 2003).

Source separation is the other extreme, where each material is cleaned and sorted prior to collection. This method requires the least post-collection sorting and produces the purest recyclate, but incurs additional operating costs for collection of each separate material. This sort of processes requires a lot of cleaning process. Therefore it is more expensive and sometimes not feasible (Bartone C. R., 1990).

2.2 Machine and apparatus utilized

Many equipments and apparatus were utilized during the experiment. The machines used are discussed more below.

2.2.1 Centrifuge

Centrifugation is a process that involves the use of centrifugal force for the separation of mixtures, used in laboratory settings. More-dense components of the mixture migrate away from the axis of the centrifuge, while less-dense components of the mixture migrate towards the axis. Chemists may increase the effective gravitational force on a test tube so as to more rapidly and completely cause the precipitate to gather on the bottom of the tube. The remaining solution is called the supernatant liquid. The supernatant liquid is then either quickly decanted from the tube without disturbing the precipitate (Harrison *et al.*, 2003).

The rate of centrifugation is specified by the acceleration applied to the sample, typically measured in revolutions per minute (RPM). The particles' settling velocity in centrifugation is a function of their size and shape, centrifugal acceleration, the volume fraction of solids present, the density difference between the particle and the liquid, and the viscosity (Ng C. W. W. *et al*., 2006).

The centrifuge uses large horsepower motor and because of the number of moving parts in it, it is subject to high maintenance. While centrifuges are effective at removing suspended solids, they do not account for dissolved solids and heavy metal species in solution. The effluent from a centrifuge would need further treatment prior to disposal (Ng C. W. W. *et al*., 2006).

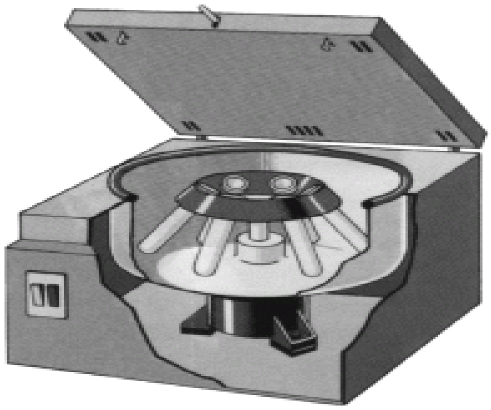


Figure 2.2: Centrifuge (Ainul Axiila Bt.Ahmad, 1994)

2.2.2 Filtration

Filtration is a mechanical or physical operation which is used for the separation of solids from fluids by interposing a medium to fluid flow through which the fluid can pass, but the solids in the fluid are retained. It has to be emphasized that the separation is not complete, and it will depend on the pore size and the thickness of the medium as well as the mechanisms that occur during filtration (Hulanicki A., 1995).

Filtration, as a physical operation is very important in chemistry for the separation of materials of different chemical composition in solution by first using a reagent to precipitate one of the materials and then use a filter to separate the solid from the other materials. Filtration is also important and widely used as one of the unit operations of chemical engineering (Hulanicki A., 1995).

It is important not to confuse filtration with sieving. In sieving there is only a single layer of medium where size separation occurs purely by the fact that the fraction of the particulate solid matter which is too large to be able to pass through the holes of the sieve, scientifically called oversize are retained. In filtration a multilayer medium is involved, where other mechanisms are included as well, for instance direct interception, diffusion and centrifugal action, where in this latter those particles, which are unable to follow the tortuous channels of the filter will also adhere to the structure of the medium and are retained (Cunie *et al.*, 1988).

The filtration process separates particles and fluid from a suspension, and the fluid can be either a liquid or a gas. To separate a mixture of chemical compounds, a solvent is chosen which dissolves one component, while not dissolving the other. By dissolving the mixture in the chosen solvent, one component will go into the solution and pass through the filter, while the other will be retained. This is one of the most important techniques used by chemists to purify compounds (Cunie *et al.*, 1988).

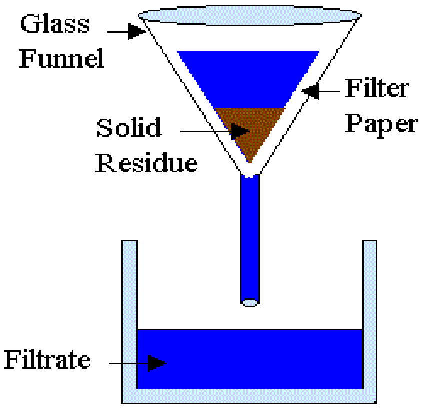


Figure 2.3: Typical filtration system (Dr. Collin France, 2008)

There are many different methods of filtration; all aimed to attain the separation of substances. This is achieved by some form of interaction between the substance or objects to be removed and the filter. In addition the substance that is to pass through the filter must be a fluid (Crosby *et al.*, 1995)

The simplest method of filtration is to pass a solution of a solid and fluid through a porous interface so that the solid is trapped, while the fluid passes through. This principle relies upon the size difference between the particles making up the fluid, and the particles making up the solid. In the laboratory, a Büchner funnel is often used, with a filter paper serving as the porous barrier (Pierre Gy., 1998).

2.2.3 Atomic absorption spectroscopy

Atomic absorption spectrometer (AAS) is designed to determine the amount of an object element in a sample by utilizing the phenomenon that the atoms in the ground state absorb the light of characteristic wavelength passing through an atomic vapor layer of the element (Hulanicki A., 1995).

The apparatus used usually consists of a light source, a sample-atomizer, a spectroscope, a photometer, and a recording system. Some are equipped with a background compensation system. For the light source, a hollow cathode lamp and a discharge lamp are mainly used. To the sample-atomizer, the flame type, electro thermal type, and the cold-vapor type are applied (Haswell S. J., 1991).

The cold-vapor flameless type is categorized as the two methods: reduction vaporizing method and heat vaporizing method. The flame type is composed of a burner and a gas-flow regulator. The electro thermal type is composed of an electric furnace and a power source and the cold-vapor type is composed of a mercury generator by chemical reduction-vaporization and thermal reduction-vaporization and an absorption cell. For the spectroscope, a grating for light diffraction or an interference filter prism is used. The photometer mainly consists of a detector and a signal treatment system. A recording system is composed of a display and a recording device. A background compensation system is employed for the correction of matrix effects on the measuring system. Several principles can be utilized for background compensation, using the continuous spectrum sources; the Zeeman split spectrum, the non resonance spectrum, or the self-inversion phenomena (Haswell S. J., 1991).

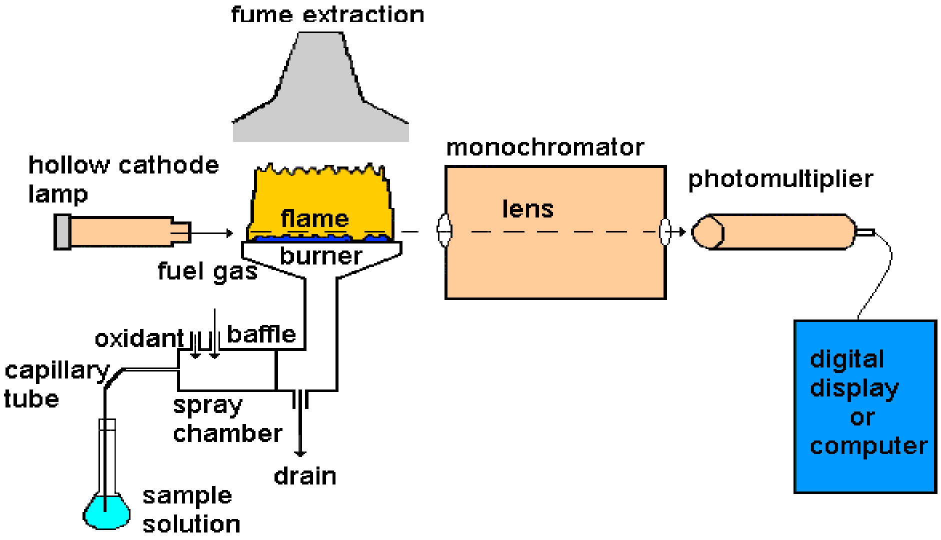


Figure 2.4.: Schematic diagram of Atomic absorption spectrophotometer (Baker *et al.*, 1982)

The types of test available for atomic absorption spectrophotometry are as below:

a) Flame Type of flame atomic absorption spectroscopy (FAAS) requires a liquid sample to be aspirated, aerosolized, and mixed with combustible gases, such as acetylene and air or acetylene and nitrous oxide. The mixture is ignited in a flame whose temperature ranges from 2100 to 2800 ^{o}C. During combustion, atoms of the element of interest in the sample are reduced to free, unexcited ground state atoms, which absorb light at characteristic wavelengths. The characteristic wavelengths are element specific and accurate to 0.01-0.1nm. To provide element specific wavelengths, a light beam from a lamp whose cathode is made of the element being determined is passed through the flame. A device such as photon multiplier can detect the amount of reduction of the light intensity due to absorption by the analyte and this can be directly related to the amount of the element in the sample (Haswell S. J., 1991).

b) Electro thermal type works when the specific light source is fitted to the lamp housing. After lighting the lamp and selecting the analytical wavelength specified in the individual monograph an appropriate electric current and slit-width is set. A suitable amount of sample solution which is also known as standard solution, or control solution is prepared as specified in the individual monograph. The prepared sample is then injected to the furnace and an appropriate stream of inert gas is made to flow through the furnace. The specimen is dried and ashed. The element included is atomized where it is heated at appropriate temperature for an appropriate time. The atomic absorption specified is observed and the intensity of absorption is measured (Haswell S. J., 1991).

c) Cold-vapor type works when there is a specified light source lamp on the photometer. The source lamp is lit and the wavelength dial of the spectroscope is adjusted to the wavelength of the analytical line specified. An appropriate current value and a slit-width are set. Then, in the reduction vaporizing method, transfer the test solution or the standard solution to the closed vessel. The element is reduced by addition of a proper reducing agent, and vaporized. In the heat vaporizing method, the sample is vaporized by heating. The absorbance of the atomic vapor generated is measured by these methods (Haswell, S.J., 1991).

Usually, the determination can be done by an appropriate one of the methods given below. In the determination, the interference and background should be considered. Below are few methods that are discussed:

i) Calibration Curve Method is done by preparing standard solutions of at least three different concentrations. The absorbance of these standard solutions is measured and a calibration curve from the obtained values is prepared. Then measure the absorbance for the test solution concentration to a measurable range and determine the amount (concentration) of the object element from the calibration curve (Hulanicki A., 1995).

ii) Standard Addition Method is to equal volumes of more than two different test solutions. The standard solution is added so that there are stepwise increasing amounts of the object element in the solutions. The solvent is added to make a definite volume. The absorbance for each solution is measured. The amounts (concentration) of added standard object element on the abscissa and the absorbance is plotted. Extend the calibration curve obtained by linking the plots and determine the amount (concentration) of object element from the distance between the origin and the intersecting point of the calibration curve on the abscissa (Hulanicki A., 1995).

2.2.4 pH meter

pH as a term was coined by Sorensen in 1909. He described the concentration of hydrogen ion as a logarithmic scale which he called “Pondus Hydrogeni” or the exponent of hydrogen. pH is a quantitative unit of measure which describes the degree of acidity or alkalinity of a substance. It is measured on a scale of 0 to 14. The term pH is derived from “p” the mathematical symbol of the negative logarithm, and “H” the chemical symbol of Hydrogen (Kristensen H. B. *et al.*, 1991).

The pH for pure water at 25 °C (77 °F) is close to 7.0. Solutions with a pH less than 7 are said to be acidic and solutions with a pH greater than 7 are said to be basic or alkaline. pH measurements are important in medicine, biology, chemistry, food science, environmental science, oceanography and many other applications (Kristensen H. B. *et al.*, 1991).

A pH measurement loop is made up of three components, the pH sensor, which includes a measuring electrode, a reference electrode, and a temperature sensor; a preamplifier; and an analyzer or transmitter. A pH measurement loop is essentially a battery where the positive terminal is the measuring electrode and the negative terminal is the reference electrode. The measuring electrode, which is sensitive to the hydrogen ion, develops a potential (voltage) directly related to the hydrogen ion concentration of the solution. The reference electrode provides a stable potential against which the measuring electrode can be compared (Cunie *et al.*, 1988).

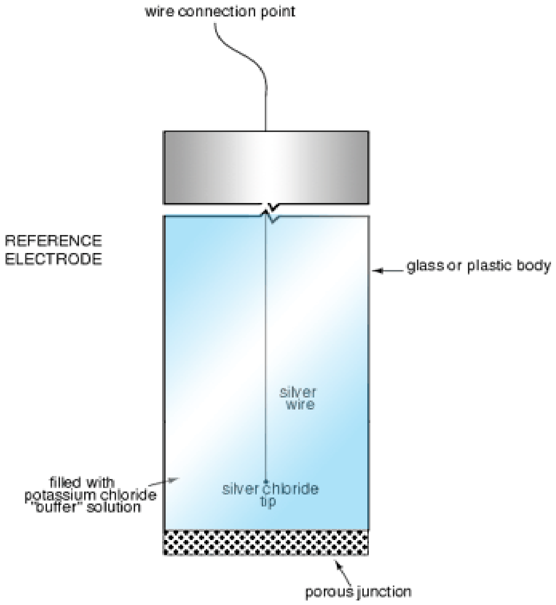


Figure 2.5: Illustration of the reference electrode's construction

(Zemaitis *et al*., 1996)

pH is a potentiometric measurement where the electrode used produces an electrical signal which the pH meter converts to a pH reading. The signal produced and measured is a voltage and since voltage is a potential difference, two potentials are needed to make a measurement: The potential difference between the reference and sensing electrode is measured by the meter and converted into a pH value (Cunie *et al.*, 1988)**.**

2.2.5 Viscometer

A viscometer (also called viscosimeter) is an instrument used to measure the viscosity of a fluid. For liquids with viscosities which vary with flow conditions, an instrument called a rheometer is used. Viscometers only measure less than one flow condition (Baucke F. G. K. *et al.*, 1993).

In general, either the fluid remains stationary and an object moves through it, or the object is stationary and the fluid moves past it. The drag caused by relative motion of the fluid and a surface is a measure of the viscosity. The flow conditions must have a sufficiently small value of Reynolds number for there to be laminar flow (Baucke F. G. K. *et al*., 1993).

At 20.00 degrees Celsius the viscosity of water is 1.002 mPa·s and its kinematic viscosity (ratio of viscosity to density) is 1.0038 mm^2/s. These values are used for calibrating certain types of viscometer (Cunie *et al.,* 1988).

In this experiment, the Brookfield rotational viscometer is used. It measures viscosity by sensing the torque required to rotate a spindle at constant speed while immersed in fluid. The torque is proportional to the viscous drag on the spindle; thus the sample viscosity (Hemphill T. *et al.,* 1993).

Rotational viscometers offer several advantages:

- The continuous rotation of the spindle allows measurements to be made over time, permitting analysis of time-dependent fluids
- The rate of shear is constant, so both Newtonian and non-Newtonian fluids can be tested
- By rotating the spindle at several different speeds, shear dependent behavior can be analyzed

Brookfield viscometers are the industry standard in determining absolute viscosity of all types of liquids with viscosities as high as 320 million centipoises (cP). There are a variety of spindles that are used to accurately determine the viscosity of a solution. The type of spindle used varies for different type of liquids with different type of viscosity (Hemphill T. *et al.*, 1993).

Figure 2.6: Brookfield viscometer

2.2.6 Fourier Transform Infrared spectroscopy

Fourier Transform Infrared (FTIR) is a method of infrared spectroscopy. In infrared spectroscopy, IR radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some of it is passed through or transmitted. The resulting spectrum represents the molecular absorption and transmission, creating a molecular fingerprint of the sample. Like a fingerprint no two unique molecular structures produce the same infrared spectrum. This makes infrared spectroscopy useful for several types of analysis (Hulanicki A., 1995).

FTIR can provide information as it can be used to identify unknown materials. The quality and consistency of a sample can also be determined. The

FTIR is also capable of determining the amount if components in a mixture (Hulanicki A., 1995).

Fourier transform infrared spectroscopy is preferred over dispersive or filter methods of infrared spectral analysis for several reasons (Kristensen H. B. *et al.*, 1991):

- It is a non-destructive technique
- It provides a precise measurement method which requires no external calibration
- It can increase speed, collecting a scan every second
- It can increase sensitivity – one second scans can be co-added together to ratio out random noise
- It has greater optical throughput
- It is mechanically simple with only one moving part

The normal instrumental process is as follows:

1. The Source: Infrared energy is emitted from a glowing black-body source. This beam passes through an aperture which controls the amount of energy presented to the sample and, ultimately to the detector (Baucke F. G. K. *et al.*, 1993).
2. The Interferometer: The beam enters the interferometer where the "spectral encoding" takes place. The resulting interferogram signal then exits the interferometer (Baucke F. G. K. *et al.*, 1993).
3. The Sample: The beam enters the sample compartment where it is transmitted through or reflected off of the surface of the sample, depending on the type of analysis being accomplished. This is where specific

frequencies of energy, which are uniquely characteristic of the sample, are absorbed (Baucke F. G. K. *et al.*, 1993).

4. The Detector: The beam finally passes to the detector for final measurement. The detectors used are specially designed to measure the special interferogram signal (Baucke F. G. K. *et al.*, 1993).

5. The Computer: The measured signal is digitized and sent to the computer where the Fourier transformation takes place. The final infrared spectrum is then presented to the user for interpretation and any further manipulation (Baucke F. G. K. *et al.*, 1993).

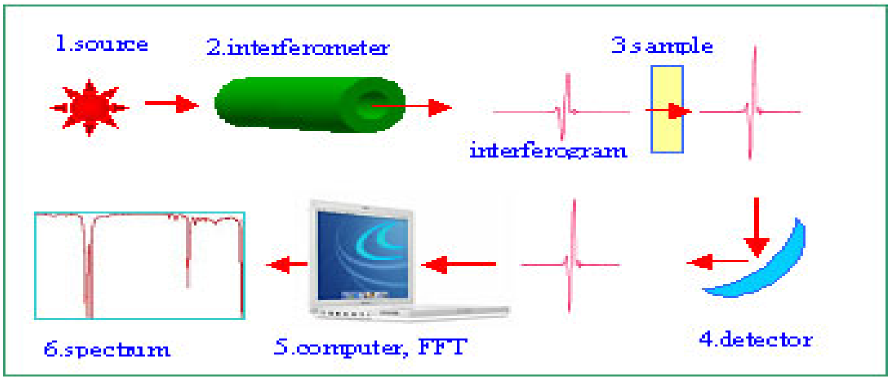


Figure 2.7: Schematic diagram of Fourier Transform Infrared (FTIR)

Some of the major advantages of FTIR over the dispersive technique include:

•Speed: Because all of the frequencies are measured simultaneously, most measurements by FTIR are made in a matter of seconds rather than several minutes. This is sometimes referred to as the Felgett Advantage (Hulanicki A., 1995).

•Sensitivity: Sensitivity is dramatically improved with FTIR for many reasons. The detectors employed are much more sensitive, the optical throughput is much higher (referred to as the Jacquinot Advantage) which results in much lower noise levels, and the fast scans enable the co-addition of several scans in order to reduce the random measurement noise to any desired level (referred to as signal averaging) (Hulanicki A., 1995).

•Simplicity: The moving mirror in the interferometer is the only continuously moving part in the instrument. Thus, there is very little possibility of mechanical breakdown (Hulanicki A., 1995).

•Calibration: These instruments employ a HeNe laser as an internal wavelength calibration standard (referred to as the Connes Advantage). These instruments are self-calibrating and never need to be calibrated by the user (Hulanicki A., 1995).

These advantages, along with several others measurements made by FTIR are extremely accurate and reproducible. Thus it is a very reliable technique for positive identification of virtually any sample. The sensitivity benefits enable identification of even the smallest of contaminants. This makes FTIR an invaluable tool for quality control or quality assurance applications whether it is batch-to-batch comparisons to quality standards or analysis of an unknown contaminant. In addition, the sensitivity and accuracy of FTIR detectors, along with a wide variety of software algorithms, have dramatically increased the practical use of infrared for quantitative analysis. Quantitative method scan be easily developed and calibrated and can be incorporated into simple procedures for routine analysis. Thus, the Fourier Transform Infrared (FTIR) technique has brought significant practical advantages to infrared spectroscopy. It has made possible the development of many new sampling techniques which were designed to tackle challenging problems which were impossible by older technology. It has made the use of infrared analysis virtually limitless (Hulanicki A., 1995).

CHAPTER 3

METHODOLOGY

3.1 Chemicals

The chemicals used for the recycling of spent lubricant are Hexane, Isopropanol (IPA) and Potassium Hydroxide (KOH).

3.1.1 Hexane

Hexane's molecular formula is $CH_3(CH_2)_4CH_3$ or C_6H_{14}. Hexane isomers are largely unreactive, and are frequently used as an inert solvent in organic reactions because they are very non-polar. The Hexane used in the experiment is the analytical grade chemical with a purity of 99.5-99.9 % purity. The brand of Hexane used is R&M Chemicals.

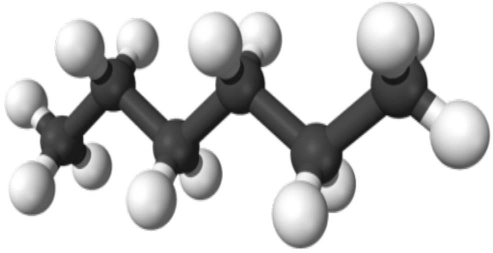


Figure 3.1: Ball-and-stick model of the hexane molecule

3.1.2 Isopropanol

Isopropyl alcohol which is also known as propan-2-ol, 2-propanol, or the abbreviation IPA is a common name for Isopropanol. It is a colorless, flammable chemical compound with a strong odor. It has the molecular formula C_3H_7OH and is the simplest example of a secondary alcohol, where the alcohol carbon is attached to two other carbons. It is an isomer of propanol. IPA is completely miscible with water, ethanol, acetone, chloroform, and benzene. The brand of IPA used is MERCK.

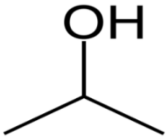


Figure 3.2: Skeletal formula of Isopropanol

3.1.3 Potassium hydroxide

Potassium hydroxide is the inorganic compound with the formula KOH. This colorless solid is a prototypical strong base. It has many industrial and niche applications. Potassium hydroxide is usually sold as translucent pellets, which will become tacky in air because KOH is hygroscopic. Its dissolution in water is strongly exothermic, meaning the process gives off significant heat. The KOH used in the experiment was also in its solid pellet form. The brand of KOH used is SYSTERM.

Figure 3.3: Pellets of Potassium hydroxide (KOH)

3.2 Equipments

There were a variety of equipments used for the recycling of spent lubricant. They are magnetic stirrer, overhead stirrer and also aluminum foil.

3.2.1 Magnetic stirrer

A magnetic stirrer is used during the experiment of solvent preparation. It is use for dissolving KOH in IPA. The dissolution process takes time and constant stirring facilitates the process.

Figure 3.4: Magnetic stirrer

3.2.2 Overhead Stirrer

The overhead stirrer that was used in the experiment is the RW 11 basic stirrer. This stirrer is suitable for stirring and mixing fluids with low and medium viscosity. It is designed for use in laboratories for stirring quantity of approximately 2 liter of liquid. For proper usage, the device must be secured by means of the guide in the plastic housing and on a stable support stand with a shaft diameter of 10 mm using the rotary handle. In this experiment, the stirrer is used to facilitate the extraction process when the solvent is added to the used lubricant. Physical mixing is induced and there will be a faster process to produce base oil and also sludge.

Figure 3.5: RW 11 Overhead stirrer

3.2.3 Aluminium foil

Aluminum foil is use to cover the beaker which contains the sludge and base oil. This is because the usage of hexane makes this experiment dangerous. Hexane is a very toxic substance and should be handled with care. Therefore aluminium foil is used to cover the beakers containing both the sludge and base oil. This is a preventive measure to avoid hexane vaporizing into the surrounding.

Figure 3.6: Aluminium foil

3.3 Recuperation of spent lubricant into base oil and sludge

Sludge and base oil from the recuperation of spent lubricant involves three main processes. The first process is to prepare the solvent that will be used. The following process is the recuperation of spent lubricant using the solvent produced. Finally the substance obtained from the recuperation process is filtered to separate sludge from base oil.

3.3.1 Solvent preparation

For the solvent preparation, first Potassium hydroxide (KOH) is weighed using a digital weighing scale. The weighed KOH is added to Isopropanol (IPA). The ratio of KOH used is 3 gram of KOH for every liter of IPA used. In this experiment 1200 ml of IPA is used therefore 3.6 grams of KOH is used. The mixed chemicals is put in the conical flask and stirred with a magnetic stirrer. The stirring is done to facilitate KOH to dissolve in IPA. The KOH is in solid pellet form and thus making the dissolution process slow. The KOH is made sure that it has fully dissolved in IPA before proceeding to the next process. Next hexane is added to the mixture of IPA and KOH. The ratio of hexane and the mixture used is 2 part of hexane to 3 part of the mixture. In this experiment, 800 ml hexane is added to 1200 ml of the mixture. The mixed chemicals is then stirred using an overhead stirrer for 30 minutes. The solvent is obtained after the stirring is completed.

3.3.2 Recuperation of spent lubricant

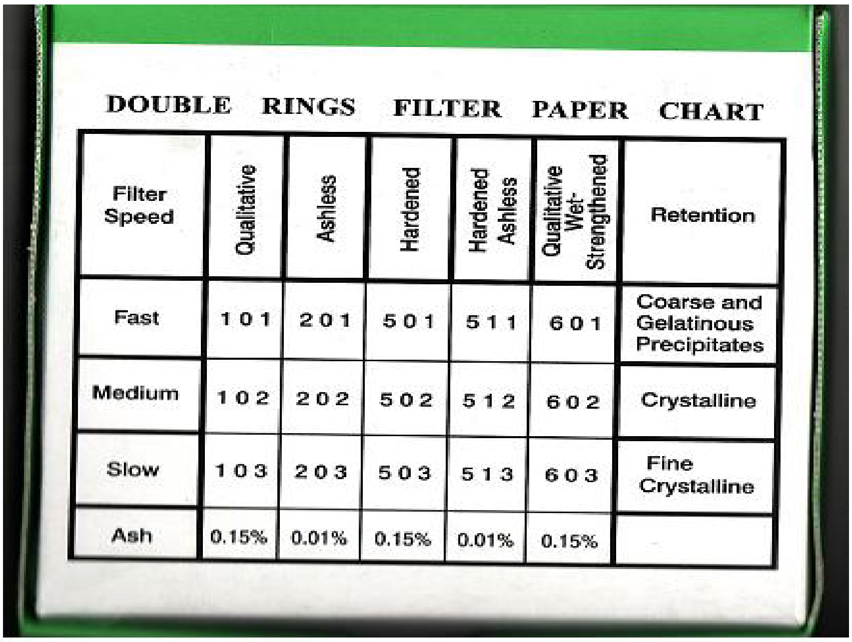
The next stage is the recuperation process. Spent lubricant is added to the solvent that is obtained. The ratio of spent lubricant and solvent used is 1:4. In this experiment, 500 ml of spent lubricant and 2000ml of the solvent prepared is used. The mixed substances are stirred using an overhead stirrer. Physical mixing is used to produce a natural reaction between the solvent and spent lubricant. The mixture is stirred continuously for 30 minutes. There will be a dark colloidal mixture at the end of the mixing process.

3.3.3 Filtration to separate sludge from base oil

The stirred mixture that is obtained from the recuperation of spent lubricant is then filtered. There are two ways to filter the mixture. One is by centrifugation and the other is filtration with thin filter papers. The residue that is left behind is the sludge that is going to be used for the characterization process.

3.3.3.1 Filtration using thin filter paper

The dark colloidal mixture that is produced at the end of the mixing process is filtered using a thin filter paper. Filter paper is folded and put into a funnel. The funnel is then placed on a conical flask. Next the colloidal mixture is poured into the funnel through the filter paper. The filter paper is picked based on the figure below.



DOUBLE RINGS FILTER PAPER CHART

Filter Speed	Qualitative	Ashless	Hardened	Hardened Ashless	Qualitative Wet-Strengthened	Retention
Fast	101	201	501	511	601	Coarse and Gelatinous Precipitates
Medium	102	202	502	512	602	Crystalline
Slow	103	203	503	513	603	Fine Crystalline
Ash	0.15%	0.01%	0.15%	0.01%	0.15%	

Figure 3.7: Filter paper chart

The filter paper chosen is the 201 filter paper. This is because the filter paper retention and type matches the characteristic of the sludge that is to be separated.

This process is done in a repetitive manner as the filter paper gets saturated with sludge and there will be less filtration done. The filter paper needs regular changing to increase the efficiency of this process.

Figure 3.8: Filtration using thin filter paper carried out in the lab

Figure 3.9: Deposition of sludge on filter paper

3.3.3.2 Centrifugation

The dark colloidal mixture that is produced at the end of the mixing process is filtered using a centrifuge. First the mixture is filled in a centrifuge tube. The centrifuge tube used is the 50 ml tubes. First the centrifuge is set for 5 minutes of centrifugation. Then the centrifuge speed is set to 1000 RPM and the centrifugation process is started. At that specific speed, the experiment is repeated 4 times to determine the average sludge accumulation (Crosby *et al.*, 1995). The following runs are made by varying the speed of centrifugation to 2000, 3000, 5000, 7000, 10000, 13000 and 15000 RPM. Next the centrifuge speed is set constant and the time is varied. The centrifuge is set for 1 minutes of centrifugation. Then the centrifuge speed is set to 5000 RPM and the centrifugation process is started. This experiment is also repeated 4 times to obtain the average sludge accumulation (Crosby *et al.*, 1995). The following runs are made by varying the time at 2, 3, 4, 5, 8 and 10 minutes.

Figure 3.10: Sludge deposition at the bottom of the centrifuge tube

Figure 3.11: Base oil produced after centrifugation

3.4 Characterization of oil sludge

The characterization of oil sludge is the investigation of the pH, viscosity, density, heavy metal composition, and chemical compounds contents.

3.4.1 pH

The pH of the sludge is determined by using a pH meter. 4 different readings are taken to determine the average pH for the sludge (Crosby *et al.*, 1995).

3.4.2 Density

The determination of density is done by weighing a clean and dry 50ml graduated cylinder. Approximately 30ml of sludge is added to the weighed 50ml graduated cylinder. Then the amount of sludge there is in the cylinder is carefully read and recorded. The cylinder and sludge weight is weighed. Finally the density of the sludge is calculated. The procedure is repeated 4 times to find the average density of the sludge (Crosby *et al.*, 1995).

3.4.3 Heavy metal content analysis

The composition of heavy metal in sludge is determined using the Atomic Absorption Spectrophotometer (AAS). The sample and also standard solution has to be prepared before the testing using the machine can take place. For sample preparation, the sludge which is in its semi-solid state is diluted with hexane. 4.5250 grams of oil sludge is measured and diluted with 100ml hexane in a volumetric flask. The dilution factor that is used is 20 times dilution. The mixture that is obtained is filtered using a micro filter. The filter used is 25µm. The reason this is done is to avoid the AAS sample suction tube from getting clogged as it has a very small nozzle pipe. Next is the preparation of the standard solutions. The standard solution is prepared at 1, 2 and 4 PPM. There is also a blank sample prepared. For solvent preparation the amount of the metal reagent that needs to be added is calculated. Then the calculated amount of reagent is added to ultra pure water. The prepared sample and standards are then put aside till the AAS is set. The AAS is started by switching on the power switches that power the AAS and the computer as well as the chiller. The chiller contains the cooling fluid that will cool the AAS system. Then compressed air and acetylene is regulated by opening the closed valves. The computer and AAS are then switched on. The flame is then fired up by pressing the Flame On switch. A distinctive blue flame can be seen. The computer is then set up and the AAS software is opened. The system is offline at this point and the online button is clicked and the AAS will respond to the computer after it is online. First for the setting, the element to be analyzed is selected and the lamp of the corresponding metal analysis that is going to be identified is selected. Then the working curve is set up by keying in the information about the number of standards and the concentrations that are used are registered. Then the AAS is set to the run mode. The AAS then gives instruction on which standard to be dipped in the suction tube. It usually starts from the blank sample moving to the least concentrated standard to the most concentrated standard. After all that has finished, finally the sample will be dipped into the suction tube. The AAS will then automatically end the experiment and the results that are obtained consist of a calibration curve as well as the metal composition in the sample.

Figure 3.12: Flame used in AAS

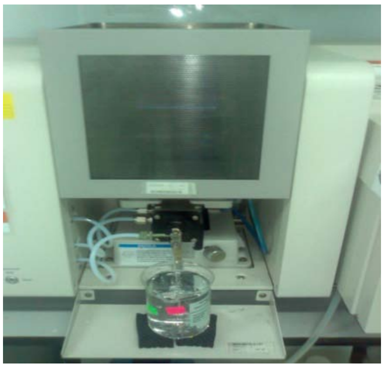


Figure 3.13: Sample analysis

3.4.4 Viscosity

The viscosity of sludge is determined by using a viscometer. The sludge is a very viscous substance. From previous similar works to that of viscosity determination of sludge, the T-type spindle is chose for the analysis. The sludge is put in a beaker. The viscometer is switched on and the machine starts up by performing automatic calibration. After the startup the machine is set to analyze the viscosity of sludge and give the readings in centiPoise (cP). The spindle number is also selected in the menu function. Next the spindle is dipped into the beaker containing the sludge. The position of the spindle is made sure to be upright and not slanting in any direction as it will affect the readings of the results. The analysis is started by pressing the enter button. The spindle will start revolving and the viscosity of sludge is determined. The readings can be taken form the digital screen that is on the viscometer. The determination of the viscosity is run about 4 times to obtain the average viscosity of sludge (Crosby *et al.*, 1995).

Figure 3.14: T-type spindle

Figure 3.15: Configuration of viscosity analysis using viscometer

3.4.5 Chemical composition analysis

The chemical composition in the sludge is determined using the Fourier Transform Infrared (FTIR) machine. The FTIR analyzes the sludge readily so there is no sample preparation for this experiment. The FTIR however has to be assembled before it can be used. The main console is detached during the last run of experiment and desiccant (Silica gel) is placed in the compartment to inhibit moisture built up. Therefore the FTIR main lid is opened and the silica gel is taken out. Next the main console is put in place. After this is done, the FTIR and computer switches are switched on. Next the EZ Omnic software is clicked open. The FTIR responds to the computer and then the next step is to perform calibration of the FTIR. The sample tablet is placed on the FTIR main console. Before that the sample tablet is cleaned using acetone to remove any residues left behind from previous experiments. Then the FTIR is run to collect the background sample when the tablet is blank. This will take approximately 32 seconds to finish. A graph reading is obtained which is called the background sample reading. This background reading is taken to differentiate the results that is obtained later form the original resulting readings. Next a small amount of sludge is smudged over the tablet glass orifice. This process is gently done as rough handling might scratch the glass orifice and later affecting the experiment. The tablet with the sludge in place is put in the FTIR console. Next the experiment is started by clicking the collect sample key.

The experiment is fast and ends in a few minutes. The reading obtained is in graphical form with peaks with specific Wavenumbers (cm^{-1}) of the chemicals markers in the sludge. The obtained peaks are then selected and are compared to the sample library that is in the computer. The sample library has a wide range of chemical markers that are known and it differentiate the peaks and specify the chemicals that are present in the sludge.

Figure 3.16: Fourier Transform Infrared (FTIR) Machine

3.5 Research design

The flow of the experiment is shown as the figure below:

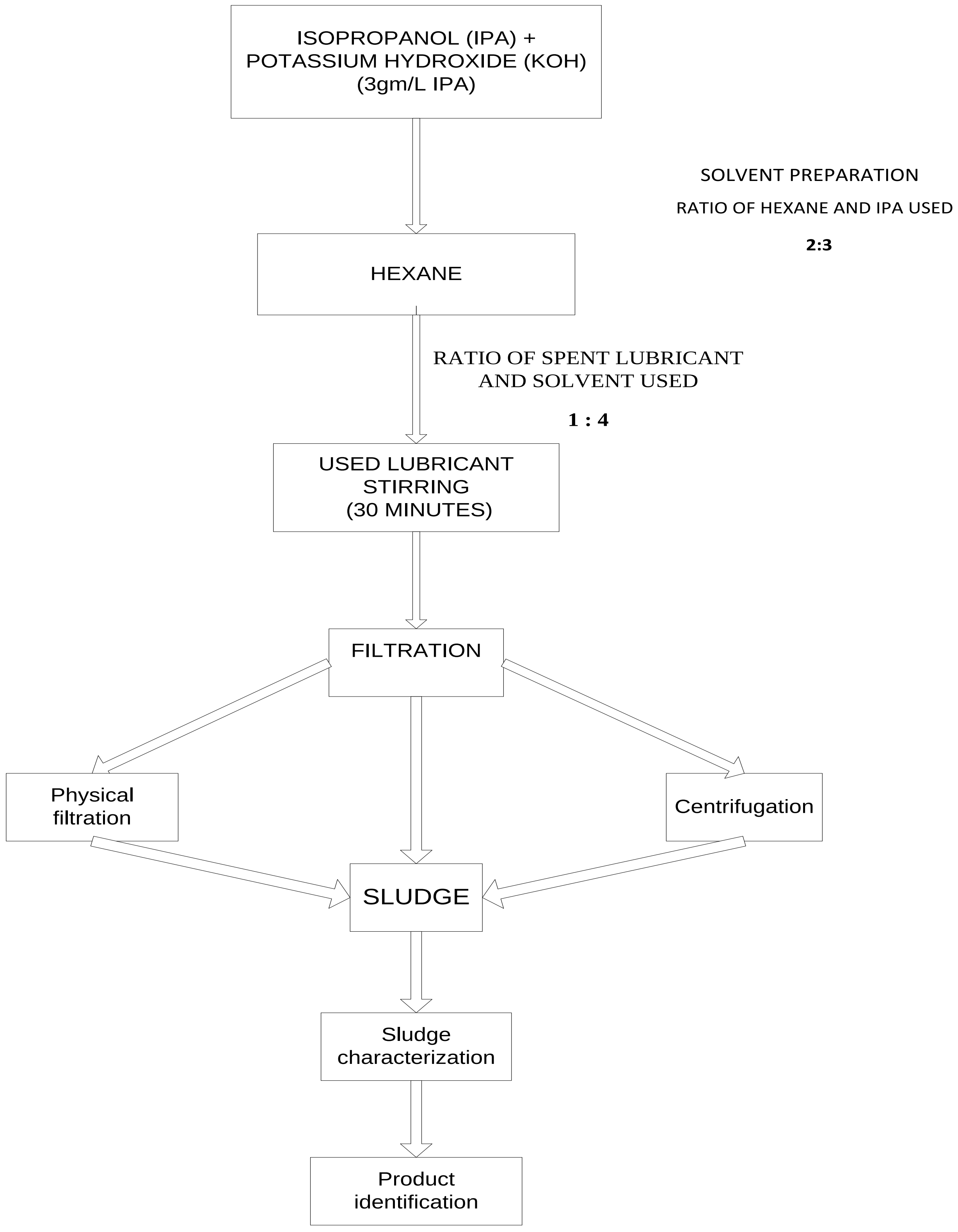


Figure 3.17: Research design

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Recycling of used lubricant

Waste oil can be a very valuable resource, if it is managed properly. It has very refined fractions of petroleum and its recovery possibilities are extremely high. Average crude oils have 3-8% base-oil, whereas lube crudes typically have 12- 16% base-oil. This compares with 65-75% recoverable base-oil content in used automotive oils, which if burnt or dumped would mean the loss of a valuable natural resource (Rahman M. M. *et al.*, 2008). Recycling of waste oil is energy efficient; less energy (about one third) is used to produce a gallon of base stock from waste oil than to produce the same gallon from crude oil. It is also environment friendly (Siddiquee *et al.*, 2008).

There are basically three options to deal with waste oil in the world which are:

- dumping the waste oil on land, garbage heap and sewerage system
- regeneration of base oil from waste oil
- extracting of heat value of waste oil through combustion process

Waste oil creates enormous problems if it is improperly disposed in the environment such as on lands, garbage heaps, surface waters and sewer systems. Simply one gallon of waste oil can ruin the taste of a million gallons of drinking water. Films of oils on the surface of water prevent the replenishment of dissolved oxygen thereby hamper aquatic life, impair photosynthetic processes and block sunlight (Kajdas C., 2000). This causes the destruction of a natural ecology in rivers and streams thus killing all organisms in it. The major problem arises when the water flows to sea and the same destructive path is lead to the sea. Therefore proper management of used lubricants is vital to keep the environmental issues at bay (Boughton and Horvath, 2003). Recycling is a viable option as it replenishes the use of base oil that is usually obtained from crude oil. The recycling process on the whole saves energy, natural resources and also prevents further pollution to the environment.

4.2 Filtration and centrifugation

Filtration is performed in the experiment to separate sludge from base oil. The dark colloidal mixture that is produced at the end of the recuperation process is filtered using a thin filter paper. The solution at the end of the recuperation process is of medium viscosity and is easily separated to form sludge on the filter paper and base oil at the catchment conical flask. This process is faster than natural settling as the colloidal mixture takes a lot of time to separate and the separation that occurs is incomplete. This process was carried out in a repetitive manner as the filter paper gets saturated with sludge and there will be less filtration done. The filter paper needs regular changing to increase the efficiency of this process. This cause excessive usage of filter paper as well as the time taken for this process is longer if compared to the centrifugation process.

The dark colloidal mixture that is produced at the end of the recuperation process is filtered using a centrifuge. However, there are no single set of standard operating conditions in terms of centrifuge time and speed that have been defined for the oil sludge separation. This experiment investigated centrifuge conditions on the effect of speed and time variation on the separation of sludge that is obtained from the recycling process of spent lubricant. Two parameters are studied. The parameters set are variable centrifugal speed with fixed time and also variable centrifugation time with fixed speed. This is done to determine the optimum parameter for centrifugation. Each of the experiment run is repeated 4 times to obtain the average value of sludge separation (Crosby *et al.*, 1995).

The first parameter experimented is when the time is set to 5 minutes and the centrifugal speed is varied.

CENTRIFUGAL SPEED (RPM)	SLUDGE DEPOSITED (mm)				
	1ST	2ND	3RD	4TH	AVERAGE
1000	9	7	7	8	8
2000	9	9	8	9	9
3000	10	9	9	8	9
5000	11	11	11	10	11
7000	11	10	10	11	11
10000	11	11	12	11	11
13000	10	11	11	13	11
15000	12	11	11	11	11

Table 4.1: Variable centrifugal speed with fixed time

From the table above, it can be concluded that as the centrifugal speed increases, the sludge get deposited and complete separation is achieved. The centrifugation reaches its maximum deposition at about 11 mm.

The reason there is an increase in the sludge deposition when the speed is increased is because there is a force which is at work when an object (oil sludge) is in a fluid. This force is the centripetal force. This force is caused by an object that is whirled about an axis is constantly changing direction and thus accelerating. There is acceleration for this process even though the rotational speed is constant. When rotated in a centrifuge, the content of the fluid and solid exerts an equal and opposite force on each other. This force is called the centrifugal force. Furthermore, gravity attracts an object toward the center of the earth. The result of gravity and the action of a fluid on a stationary object is a force that we often call the apparent weight of an object in which it is the sludge in this case. It is the combination of gravity acting downward and the buoyant force acting upward. The force of gravity is proportional to the mass of an object. When an object moves through a fluid there is a force that resists the motion of the object that depends on the viscosity of the fluid and on the area the object presents to the fluid. This accentuates the difference between the apparent weight and true weight of an object moving through a fluid (Cunie *et al.*, 1988). Therefore when there is an increase in the speed of centrifugation, the net forces that is created for separation also increases. When the speed is increased, the centrifugal force also increases thus causing more separation to occur.

The figure shown below depicts the results in a graphical form.

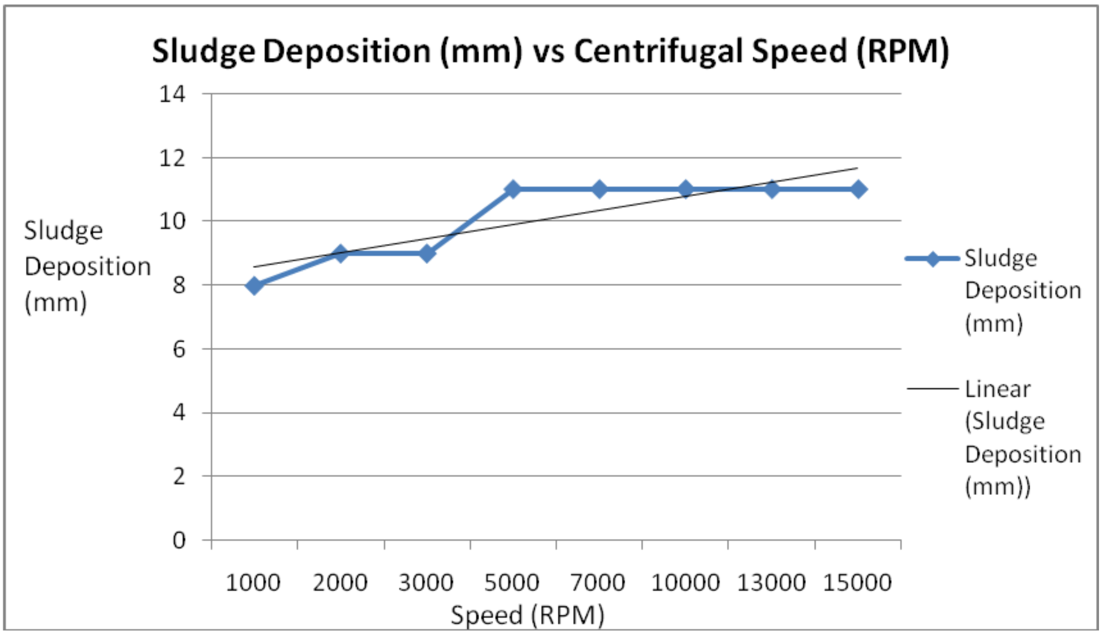


Figure 4.1: Average Deposition (mm) vs Centrifugal Speed (RPM)

From the results obtained, the optimum parameter for centrifugation can be determined. At constant time with variable speed, the sludge deposition shows an increase in thickness but it reaches the maximum deposition at 5000 RPM. The following speeds at 7000, 10000, 13000 and 15000 RPM gives the same deposition values. Therefore it can be concluded that the optimum speed for complete separation to take place is at 5000 RPM.

The second parameter experimented is when the centrifugal speed is set to 5000 RPM and the time is varied.

TIME (min)	SLUDGE DEPOSITED (mm)				
	1ST	2ND	3RD	4TH	AVERAGE
1	6	7	7	5	6
2	7	7	7	8	7
3	8	8	7	9	8
4	8	9	9	9	9
5	9	8	8	10	9
8	9	7	9	10	9
10	9	8	9	9	9

Table 4.2: Variable time with fixed speed

From the table above, it can be concluded that as the centrifugal time increases, the sludge get deposited and complete separation is achieved. The centrifugation reaches its maximum deposition at about 9 mm.

The force involved in the centrifugation process is the centripetal force. This force is caused by an object that is whirled about an axis is constantly changing direction and thus accelerating. There is acceleration for this process even though the rotational speed is constant. Therefore the speed does not play a significant role in this parameter that is studied. Similar to the previous study, the content of the centrifuge which are fluid and solid exerts an equal and opposite force on each other.

This force is called the centrifugal force. There will be more centrifugal force that will be exerted on the dark colloidal mixture as the time increases. The extra time that is available will enable complete separation to occur (Cunie *et al.*, 1988). From the experiment that has been carried out, it can be said that when the speed is set to constant and the time for centrifugation is increased, the centrifugal force also increases thus causing more separation to occur.

The figure shown below depicts the results in a graphical form.

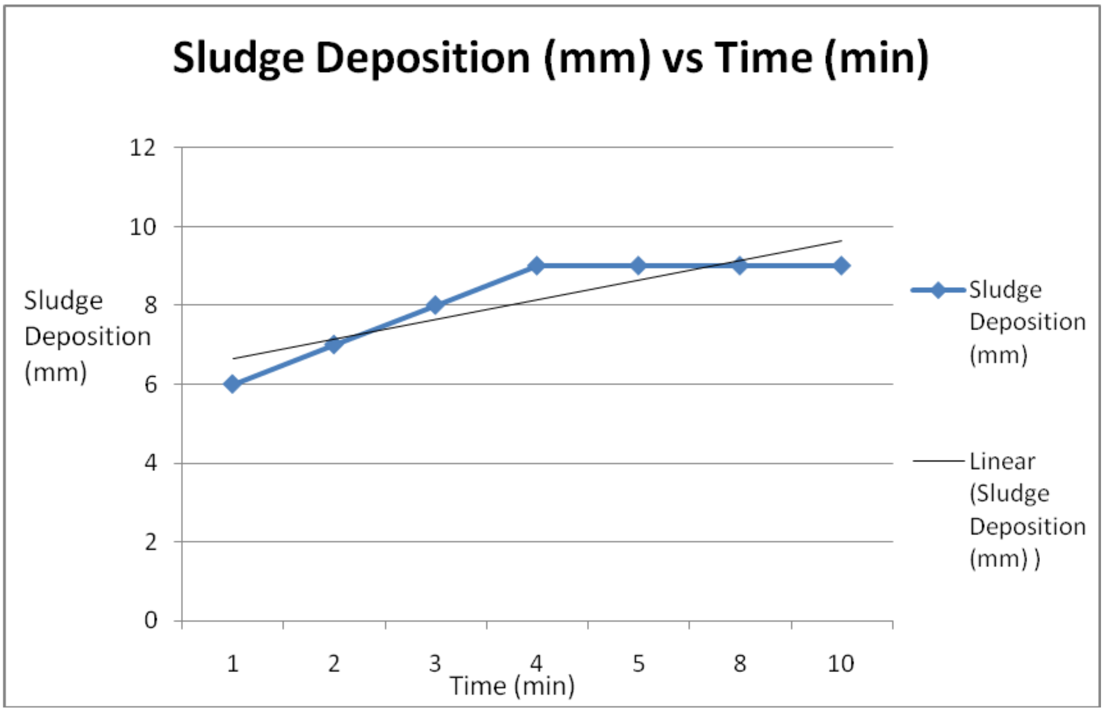


Figure 4.2: Sludge Deposition (mm) vs Time (min)

At constant speed with variable time, the sludge depositions show the same characteristic as the parameter before. There is an increase in the deposition of sludge till it reaches the 4th minute where the deposition is 9mm. The following deposition at 5, 8 and 10 minutes are constant. This shows that optimum separation occurs in the 4th minute with a speed of 5000 RPM.

4.3 Physical properties

The physical property of the sludge obtained is evaluated. The sludge produced is black in color. It has a moderate viscosity when it is first separated from base oil. This is because there is still solvent content in it. The solvent is made up of hexane and IPA which is an alcohol so the sludge is soluble in it. The sludge is dried more to remove the solvent content in it. This causes the sludge to thicken further and become very viscous. The sludge produced an odor which is similar to oily grease. The texture is very coarse as of high viscosity liquids. The sludge forms a very sticky substance. It is also very sensitive to heat. It forms carbonaceous matter when heated directly. The sludge is not soluble in water but soluble in alcohol and hexane.

4.4 pH analysis

The pH of the sludge is tested. The pH testing was done 4 times and the average was taken. The pH varies because of discrepancies in pH measurements are found from reading to reading with the same method, same sample and same lab; and from one lab to another even when they use the same method and paper from the same source. They can be reduced by careful attention to method, but they can never be eliminated entirely (Crosby *et al.*, 1995). Therefore an average reading is taken out of 2 different samples of sludge.

The sludge is slightly basic. The pH range that is obtained is from 6.30 to 6.60 with an average reading of 6.59. This reading is almost similar to the pH of oil sludge that is obtained from the oil refinery. From the Material Safety Data Sheet (MSDS) for oil sludge from refineries, the pH is known to vary from 6.50 to 6.90 based on different type of oil that is processed (Hercules, 2006). The pH of oil sludge from the recycling of spent lubricant has the similar range of pH of the oil sludge from the refinery.

SAMPLE NO.	TRIAL	pH
SAMPLE 1	1	6.76
	2	6.31
	3	6.54
	4	6.57
	AVERAGE	6.54
SAMPLE 2	1	6.58
	2	6.89
	3	6.57
	4	6.54
	AVERAGE	6.64
	∑ AVE.	6.59

Table 4.3: Average pH determination

4.5 Density determination

Density is a measure of the "compactness" of matter within a substance and is defined by the equation:

$$\text{Density} = \text{mass}/\text{volume}$$

The standard metric units in use for mass and volume respectively are grams or kilogram and milliters or cubic meters. Thus, density has the unit grams/milliter (g/ml) or kilogram/cubic meter (kg/m^3). The literature values are usually given in this unit. Density may be calculated from a separate mass and volume measurement.

By using equation below, the density is determined in kg/m^3

$$\frac{g}{ml} X \frac{1\ kg}{1000g} X \frac{1000\ ml}{1\ l} X \frac{1000\ l}{1\ m^3}$$

The density of sludge is tested for 3 different samples of sludge (Crosby *et al.*, 1995). The average density is taken. The density obtained is 0.9041 g/ml or 904.13 kg/m^3. Based on the MSDS, the density of oil sludge from the refinery is in the range of 800 kg/m^3 to 945 kg/m^3 (Hercules, 2006). This reading shows the similarity between the oil sludge from the recycling of spent lubricant and the oil sludge form the oil refinery.

The sludge that is obtained from the recycling of spent lubricant has a density that is less than water density. This causes it to float in water. To test whether the sludge is lighter than water, it is put in the water and the whether the sludge floats or sinks is observed. The sludge was found to be floating on the surface of the water thus proving that the oil sludge is lighter than water.

Figure 4.3: Oil sludge floating on the water surface

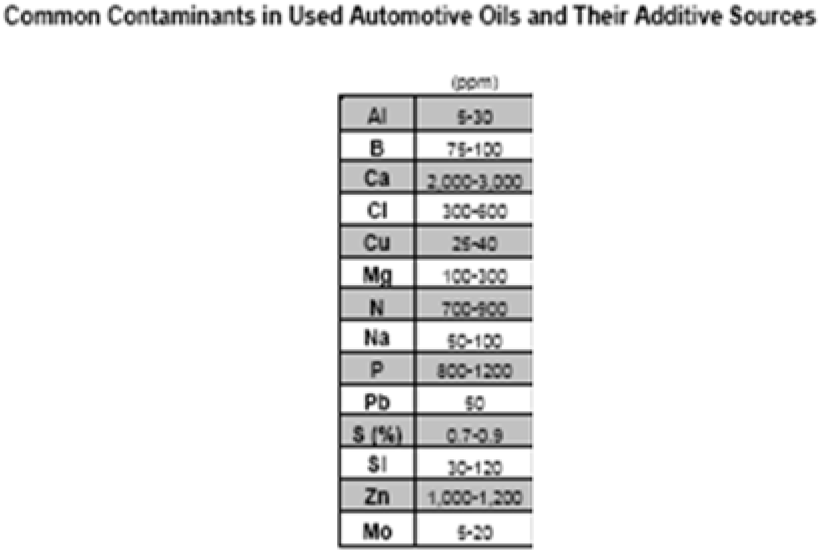
4.6 Heavy metal content analysis

Heavy metal content in the sludge was analyzed using Atomic Absorption Spectrometer (AAS). The sludge sample was tested for heavy metals such as Ferum, Cadmium, Aluminium, Copper, Lead and Zinc. The dilution factor used was 20 times. The results of the heavy metal content in sludge are shown in the table.

HEAVY METAL	CONCENTRATION (PPM)
FERUM (Fe)	10
CADMIUM (Cd)	15.4
ALUMINIUM (Al)	TRACE
COPPER (Cu)	8.2
LEAD (Pb)	0.6
ZINC (Zn)	25.2

Table 4.4: Heavy metal content in sludge

From the results obtained, it is concluded that there is a fair amount of the heavy metals that are present in the sludge. The heavy metal present in 5 grams of sludge is measured in parts per million. This shows that in 5 grams of sludge that has been used in the analysis, there are fair amounts of metal the sludge that is analyzed except for aluminium where only trace amounts were present. Most of the metal that are present served as an antiwear agent an also as an antioxidant in the engine crankshaft (Kaufman M., 2002).



Common Contaminants in Used Automotive Oils and Their Additive Sources

	(ppm)
Al	5-30
B	75-100
Ca	2,000-3,000
Cl	300-600
Cu	25-40
Mg	100-300
N	700-900
Na	50-100
P	800-1200
Pb	50
S (%)	0.7-0.9
Si	30-120
Zn	1,000-1,200
Mo	5-20

Figure 4.4: Heavy metal content in used lubricant (Audibert, 2006)

The figure shows the general heavy metal content in lubricants. This figure shows that the level of heavy metal contaminants in the oil sludge is less if compared to the original content of lubricant. The content of heavy metal present in the used lubricant varies based on the type of lubricant that is used and the ways it was manufactured (Kaufman M., 2002).

The heavy metals that are present are added as an antiwear agent and also as an antioxidant. The amount present is based on the type of lubricant that is used in the recycling process. The metals usually are not present in its basic form. It is usually added as phosphate, phenolates and sulphonates of metal in the lubricant. Examples of such metal sulphonantes, phenolates and phosphates are Zinc Dithiophosphate, Lead phenolate and Ferum sulphonate. The formations of metals are because of the degradation of the lubricant. The metals loose its chemical configuration and loose its properties to protect the lubricant as well causing damage to the engines (Kaufman M., 2002). From the analysis, it can be said that the cause of the presence of metal in the sludge after the recycling process is because of the breakdown of this additives in the used lubricant (Athanasios K. K. and Evangelos, 2001).

4.7 Viscosity

The viscosity of sludge is determined using viscometer. The sludge for this analysis was first dried to remove hexane and IPA. This is done because sludge dissolves in hexane and also IPA so this will affect the analysis by causing the viscosity to be low. Once the hexane and the IPA are removed, the sludge forms to be a high viscous substance.

The table shows the viscosity of sludge obtained in the analysis. The units used were Centi Poise (cP).

SAMPLE NO.	TRIAL	VISCOSITY (cP)
SAMPLE 1	1	1312
	2	1431
	3	1453
	4	1298
	AVERAGE	1374
SAMPLE 2	1	1472
	2	1576
	3	1346
	4	1457
	AVERAGE	1463
	∑ AVE.	1419

Table 4.5: Viscosity of sludge

The result shows the high viscosity of sludge. In average, the viscosity of sludge is 1419 cP. There are no significant findings on the viscosity of sludge in previous studies. The sludge obtained form the recycling process of used lubricant is compared to that of from oil refineries. The sludge from oil refineries has a wide range of viscosity which is from 750 cP to 1650 cP (Hercules, 2006). The range is big because of the wide range of oil being used in the refinery (Athanasios K. K. and Evangelos, 2001). From the results obtained, the similarity between the oil sludge from the recycling process and from the oil refinery can be seen from the viscosity obtained. The viscosity of oil sludge from the recycling process falls in the range of the viscosity that are present from the oil sludge form refineries.

4.8 Chemical content analysis

The chemical contents of sludge were analyzed using Fourier Transform Infrared (FTIR) machine. Like a fingerprint no two unique molecular structures produce the same infrared spectrum. This makes infrared spectroscopy useful for the chemical content identification of sludge. There is a big and comprehensive library of chemicals and materials present for comparison.

The result from the analysis is shown in the table below.

Compound Name	% Match
Poly(Ethylene:Propylene:Diene)	86.92
Triacontane	79.08
Poly(Ethylene:Propylene)	77.64
1-Bromo Adamantane	66.57
Poly(Ethylene)	67.89

Table 4.6: Chemical content in sludge

The active chemical found in the analysis is Triacontane. From the MSDS for Triacontane, it is known to have a chemical formula of $C_{30}H_{62}$. Triacontane exists as a white waxy solid in its natural form but in the sludge it is dark colored as it undergoes reaction and binds with other matter that changes its color. It has a melting point of 66 ^{0}C and its boiling point is 450 ^{0}C. Triacontane is a stable compound with a density of 807 kg/m^{3}. Triacontane is soluble in alcohol bit is insoluble in water (Hercules, 2006).

The next active compound is a mixture of polymers that act as additive. Poly(Ethylene:Propylene:Diene), Poly(Ethylene:Propylene), Poly(Ethylene) are all additives that are added to increase the viscosity of lubricant. These polymers are also known as Polymer Additives. This addition increases the life span of the lubricants as it slows down thickening of lubricant (Kaufman M., 2002).

Heptane is added to the lubricant also. It acts as a good ‘knocking’ inhibitor. Knocking in spark-ignition internal combustion engines occurs when combustion of the air and fuel mixture in the cylinder starts off correctly in response to ignition by the spark plug, but one or more pockets of air and fuel mixture explode outside the envelope of the normal combustion front. The fuel-air charge is meant to be ignited by the spark plug only, and at a precise time in the piston's stroke cycle. The peak of the combustion process no longer occurs at the optimum moment for the four-stroke cycle. The shock wave creates the characteristic metallic "pinging" sound, and cylinder pressure increases dramatically. Effects of engine knocking range from inconsequential to completely destructive effects (Kaufman M., 2002).

1-Bromoadamantane also exists as an additive in the lubricant. It reduces knocking and is widely used in heavy duty machineries (Athanasios K. K. and Evangelos, 2001).

4.9 Product identification

4.9.1 Pyrolysis of oil sludge

Oil sludge, if unused, is one of the major industrial wastes that need to be treated for the refinery or petrochemical industry. It contains a large portion of combustible components with high heating values. Obviously, the conversion of oil sludge to various useful materials such as lower molecular weight organic compounds and carbonaceous residue via pyrolysis not only solves the disposal problem but also matches the appeal of resource utilization (Sakata Y. *et al*., 1997). This characteristic is used for the sludge that is obtained from the recycling of spent lubricant. The sludge consists of lower molecular matters that resemble refinery oil sludge.

The oil sludge from the oil storage tank of a typical petroleum refinery plant located in northern Taiwan is used as the raw material of pyrolysis (Shie J. L. *et al*., 2002). Its heating value of dry basis and low heating value of wet basis are about 10681 and 5870 kcal/kg, respectively. The pyrolysis of oil sludge is conducted by using nitrogen as carrier gas in the temperature range 378-873 K. The pyrolytic reaction is very complex and significant in the range 450-800 K. The residues of pyrolysis of oil sludge exhibit very high viscous form below 623 K while low viscous or solid form above 713 K (Je L. S.*et al*., 2004). The major gaseous products (non condensable gases at 298 K) excluding N_2 are CO_2 (50.88 wt %), Hydrocarbons (HC's, 25.23 wt %), H_2O (17.78 wt %), and CO (6.11 wt %). The HC's mainly consist of a mixture of low and high molecular weight paraffin and olefins (C_1-C_{35}, 51.61 wt % of HC's). The temperature corresponding to the maximum production rate of HC's is 713 K. The distillation characteristic of liquid product (condensate of gas at 298 K) from the pyrolysis of oil sludge is close to diesel oil (Chang C. Y. *et al.*, 2000).

From the chemical analysis it is already determined that sludge consists of olefins as it originated from petrochemicals. The viscosity is high and resembles the oil sludge that is obtained from the refinery. The similarity of both the oil sludge shows that the oil sludge from the recycling process can also be used in pyrolysis to produce liquid product that resembles diesel oil.

4.9.2 Sludge as concrete or cement additive

Wastes can be defined as not readily avoidable by-products for which there is no economical demand and for which disposal is required. Processed or unprocessed industrial by-products or wastes can be used as raw materials in cement manufacturing, as components of concrete binder, as aggregates, a portion of aggregate, or ingredients in manufactured aggregates (Rixom R., Mailvaganam, 1999). Some wastes can be used as chemical mixtures and additives, which can alter and enhance selected properties of fresh and hardened concrete. The successful use of industrial by-products wastes in concrete depends on the required properties of the end product. Economical factors would ultimately determine if potentially beneficial waste could be used as an ingredient in concrete. These factors are generally influenced by the cost of waste disposal, the cost of transportation of waste to a manufacturing site, and existing environmental regulations (Kosmatka S., Panarese W., 1990).

The leakage of oil into the cement or concrete in older grinding units has been reported to result in concrete with greater resistance to freezing and thawing

(Kosmatka S., Panarese W., 1990). As the water in moist concrete freezes, it produces osmotic and hydraulic pressures in the capillaries and pores of the cement paste and aggregate. If the pressure exceeds the tensile strength of the paste or aggregate, the cavity will dilate and rupture. The accumulative effect of successive freeze–thaw cycles and disruption of paste and aggregate, eventually cause significant expansion and deterioration of the concrete. Deterioration is visible in the form of cracking, scaling and crumbling. Entrained air voids act as empty chambers in the paste for the freezing and migrating water to enter, thus relieving the pressures described above and preventing damage to the concrete. There is a hypothesis that adding used engine oil sludge to the fresh concrete mix could be similar to adding an air entraining chemical admixture, thus enhancing some durability properties of concrete while serving as a technique of disposing the oil waste (Rixom R., Mailvaganam, 1999).

The adding of oil sludge into concrete as additive to strengthen it is feasible. This is said because the sludge has the characteristic of seeping into the void spaces in the concrete mixture. The reduction of the void spaces will give extra strength and durability to the concrete. This will also lead to better management of the oil sludge as it can be used as the additive rather than being discarded which will be a hassle.

4.9.3 Oil sludge as grease

Grease is a semi fluid to solid mixture of a fluid lubricant, a thickener, and additives. The fluid lubricant that performs the actual lubrication can be petroleum (mineral) oil, synthetic oil, or vegetable oil. The thickener gives grease its characteristic consistency and is sometimes thought of as a “three-dimensional fibrous network” or “sponge” that holds the oil in place. Additives enhance performance and protect the grease and lubricated surfaces.

Texture is observed when a small sample of grease is pressed between thumb and index finger and slowly drawn apart. Texture can be described as:

- Buttery: the grease separates in short peaks with no visible fibers.
- Resilient: the grease can withstand moderate compression without permanent deformation or rupture
- Stringy: the grease stretches or strings out into long, fine threads, but with no visible evidence of fiber structure.

The oil sludge that was obtained from the recycling process of spent lubricant exhibits similar characteristics as the grease. The texture and the viscosity are similar to that of grease (Athanasios K. K. and Evangelos, 2001). The low content of heavy metals in the sludge allows it to be used as grease. This is said because it can be handled without the implication of heavy metal poisoning. The chemical content of sludge that resembles wax also gives it an edge to be used as grease. The sludge consists of olefin and paraffin that are left over after the recycling process. Petroleum based grease is usually made up of these chemical compounds. Therefore it is feasible to use the oil sludge form the recycling process of spent lubricant as grease.

CHAPTER 5

CONCLUSION AND RECCOMENDATION

The production of oil sludge from the recycling process of spent lubricant in a lab scale was performed and the sludge was obtained. The process involved was solvent preparation and spent lubricant recuperation. The end product of the process yields base oil and sludge.

There are several ways to separate the sludge from the resulting mixture of base oil and sludge after the recuperation process. The methods used are filtration with thin filter paper and centrifugation.

The filtration method involves the changing of filter papers when it is clogged with sludge thus making it less efficient. The filtration method also takes time as more and more sludge get deposited on the filter paper. There will also be a messy work to scrape out the sludge from the surface of the filter paper to put it in containers.

Centrifugation uses centrifugal force to separate the sludge from base oil. The separation efficiency increases with increasing centrifugal speed when the time is set to be constant. The same result is obtained when the time of centrifugation is increased with a constant centrifugal speed. The separation of sludge from base oil is more effective in the centrifugation process as the sludge gets deposited at the bottom of the centrifugal tube that is used. The removal of sludge is much easier and cleaner if compared to the filtration method. By using a centrifuge, the time taken to separate sludge from base oil is also shorter if compared to that of filtration using thin filter paper. On the whole, it can be said that centrifugation of the mixture obtained from the recycling of spent lubricant is a better method of separation if compared to filtration with thin filter paper. The centrifugation can also be done in a bigger centrifugal separator in the future. The centrifuge that was used for this experiment was a small scale one with the capacity of 50 ml. The bigger centrifuge machine was out of order and its maintenance hasn't been done. In future analysis a bigger capacity centrifuge should be used.

The sludge obtained from the recycling process of spent lubricant is put through a series of test to determine its physical and chemical characteristics. The parameter of oil sludge characteristics that were determined was pH, viscosity, density, heavy metal content and also the chemical composition of the sludge. The pH of sludge on average is 6.59 with an average density of 0.9041 g/ml or 904.13 kg/m^3. The average viscosity is 1419 cP. There is a fair amount of heavy metals present in the sludge and its chemical composition consists of paraffin and olefins. The characteristic of the sludge is used to determine its potential products. By studying the characteristics, the potential products for sludge can be identified. The product identification is done to lessen the dumping rate of sludge. This gives the sludge a more economical value thus generating the production of possible products for sludge.

There are several possible products that have been identified for the sludge. The oil sludge can be used as a raw material for pyrolysis which gives a diesel like product at the end of the process. The pyrolysis is a process of heating a substance in the absence of oxygen. Nitrogen gas is usually used as carrier gas in this process. Similar process is already done in China using the oil sludge for oil refineries. The sludge for recycling of spent lubricant is suitable for pyrolysis because it is similar to the properties that the oil sludge from a refinery. The sludge obtained from the recycling process contains high molecular weight hydrocarbons. This is the main criteria for the pyrolysis process as the hydrocarbons are the substances used to produce diesel oil like liquid. The charred remaining of the pyrolysis can be further used as fertilizer. This optimizes the usage of the sludge.

The oil sludge for the recycling of spent lubricant is also used as filler in cement or concrete mixtures. Sludge is specifically used because of its high viscosity property that allows it to seep into the mixture of cement or concrete while mixing occurs. This allows the sludge to fill up the empty void spaces that naturally occur in concrete that reduces its strength. The concrete produced will be very strong and resistant to impacts and blows form the surrounding thus making it more wanted in the construction industry. There is a setback anyway for this process. The time taken for drying to take place after the sludge is added is increased significantly. This is because the nature of the sludge to dry and bind the aggregates properly is slow. Therefore the concrete that is to be used is supposed to be produced earlier and appropriate drying time is allowed for the concrete to be fully functional without any defects.

The oil sludge produced at the end of the recycling process has the appearance of grease. The texture, color and smell are very similar to that of grease. Therefore further testing was done to confirm its feasibility as grease. The high viscous sludge can be used as grease. Therefore it is recommended that more study is performed to determine the feasibility of sludge being used as grease. On the whole the sludge looks viable to be used as grease.

The research was carried out and finished successfully. All the objectives were met and the possible products for sludge from the recycling process of spent lubricant have been identified.

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APPENDICES

Density Calculation for Sludge

Sample Name: Sludge from spent oil recuperation
Date: 10th January 2009

a) Weight of graduated cylinder: 44.6g

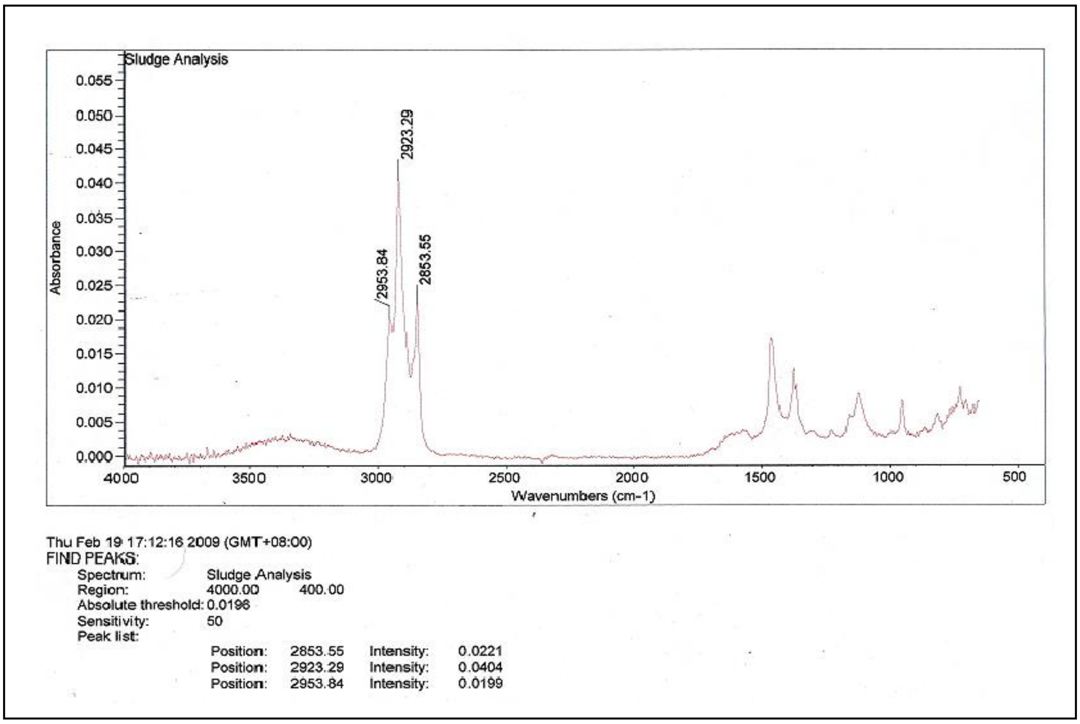
	Sample 1	Sample 2	Sample 3
Wt.of cyl + sludge	64.2 g	65.4 g	65.2 g
Wt. of sludge	19.6 g	20.8 g	20.6 g
Volume of sludge	22 ml	23.5 ml	22 ml
Density (g/ml)	0.8909	0.8851	0.9364

Average density : 0.9041 g/ml

By using equation below, the density is determined in kg/m^3

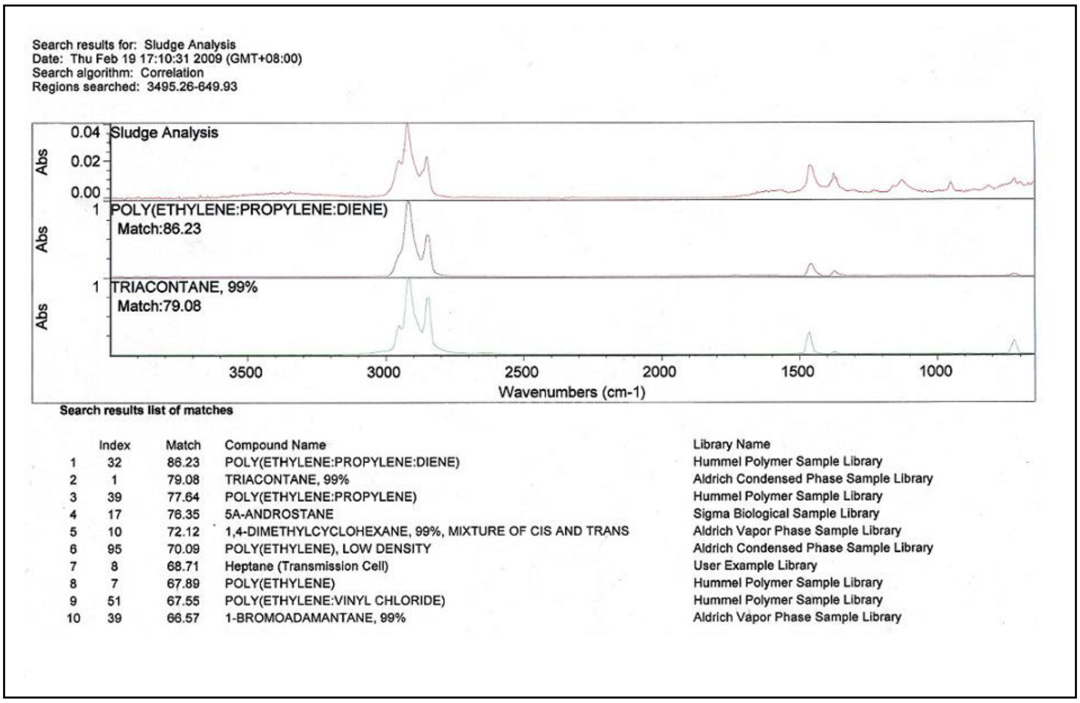
$$\frac{g}{ml} X \frac{1\ kg}{1000 g} X \frac{1000\ ml}{1\ l} X \frac{1000\ l}{1\ m^3}$$

Average density : 904.13 kg/m^3



```
Thu Feb 19 17:12:16 2009 (GMT+08:00)
FIND PEAKS:
    Spectrum:           Sludge Analysis
    Region:             4000.00        400.00
    Absolute threshold: 0.0196
    Sensitivity:        50
    Peak list:
                        Position:   2853.55   Intensity:   0.0221
                        Position:   2923.29   Intensity:   0.0404
                        Position:   2953.84   Intensity:   0.0199
```

Figure 1: Peaks obtained from FTIR



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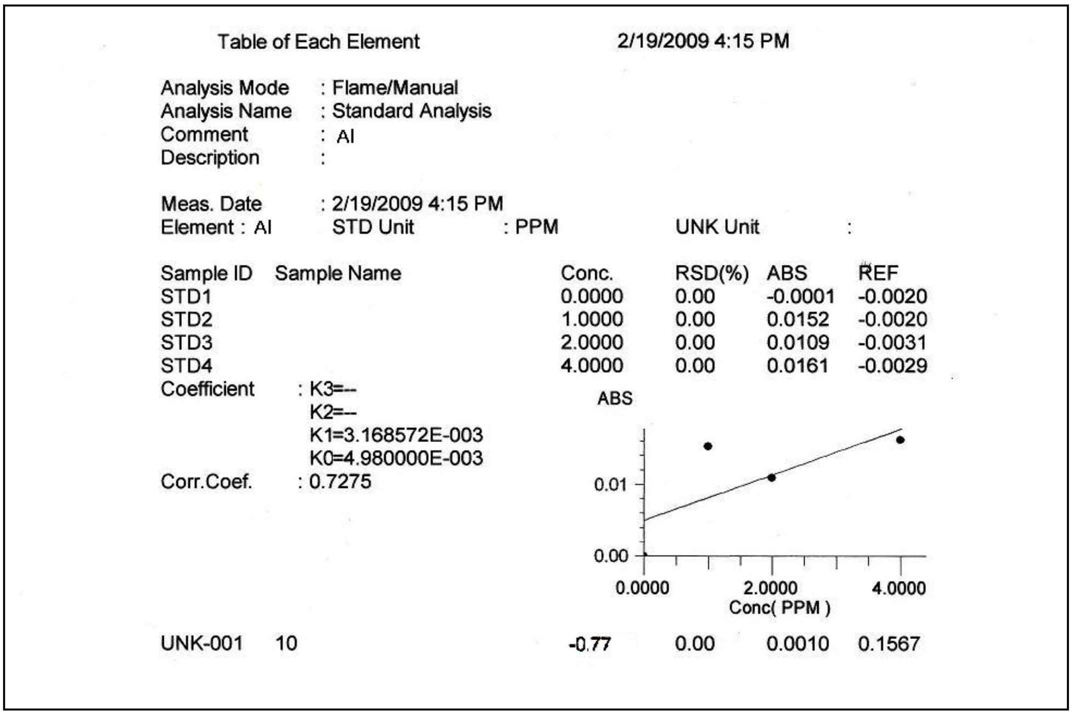
Search results for:  Sludge Analysis
Date:  Thu Feb 19 17:10:31 2009 (GMT+08:00)
Search algorithm:  Correlation
Regions searched:  3495.26-649.93
```

Search results list of matches

	Index	Match	Compound Name	Library Name
1	32	86.23	POLY(ETHYLENE:PROPYLENE:DIENE)	Hummel Polymer Sample Library
2	1	79.08	TRIACONTANE, 99%	Aldrich Condensed Phase Sample Library
3	39	77.64	POLY(ETHYLENE:PROPYLENE)	Hummel Polymer Sample Library
4	17	76.35	5A-ANDROSTANE	Sigma Biological Sample Library
5	10	72.12	1,4-DIMETHYLCYCLOHEXANE, 99%, MIXTURE OF CIS AND TRANS	Aldrich Vapor Phase Sample Library
6	95	70.09	POLY(ETHYLENE), LOW DENSITY	Aldrich Condensed Phase Sample Library
7	8	68.71	Heptane (Transmission Cell)	User Example Library
8	7	67.89	POLY(ETHYLENE)	Hummel Polymer Sample Library
9	51	67.55	POLY(ETHYLENE:VINYL CHLORIDE)	Hummel Polymer Sample Library
10	39	66.57	1-BROMOADAMANTANE, 99%	Aldrich Vapor Phase Sample Library

Figure 2: Peaks matched from the library



```
Table of Each Element                         2/19/2009 4:15 PM

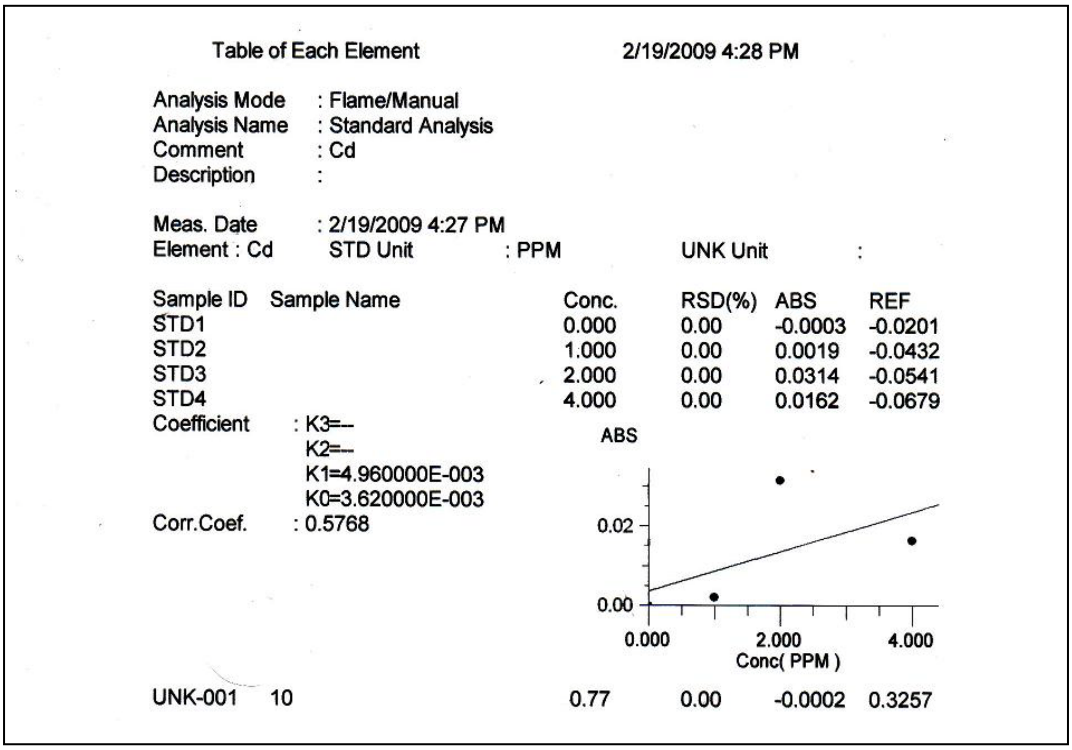
Analysis Mode    : Flame/Manual
Analysis Name    : Standard Analysis
Comment          :  Al
Description      :

Meas. Date       : 2/19/2009 4:15 PM
Element : Al      STD Unit          : PPM           UNK Unit          :

Sample ID   Sample Name                      Conc.     RSD(%)   ABS       REF
STD1                                         0.0000    0.00     -0.0001   -0.0020
STD2                                         1.0000    0.00     0.0152    -0.0020
STD3                                         2.0000    0.00     0.0109    -0.0031
STD4                                         4.0000    0.00     0.0161    -0.0029
Coefficient     : K3=--
                  K2=--
                  K1=3.168572E-003
                  K0=4.980000E-003
Corr.Coef.      : 0.7275

UNK-001     10                               -0.77     0.00     0.0010    0.1567
```

Figure 3: Aluminium analysis with AAS



```
Table of Each Element                         2/19/2009 4:28 PM

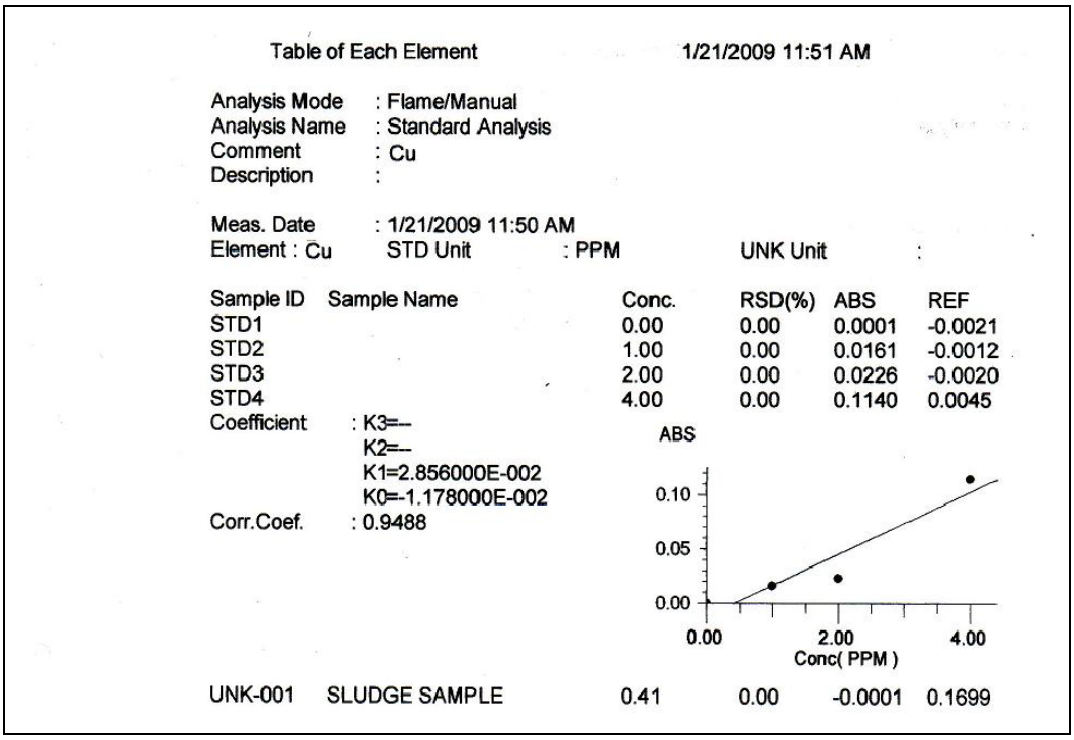
Analysis Mode    : Flame/Manual
Analysis Name    : Standard Analysis
Comment          : Cd
Description      :

Meas. Date       : 2/19/2009 4:27 PM
Element : Cd      STD Unit          : PPM           UNK Unit          :

Sample ID   Sample Name                      Conc.     RSD(%)   ABS       REF
STD1                                         0.000     0.00     -0.0003   -0.0201
STD2                                         1.000     0.00     0.0019    -0.0432
STD3                                         2.000     0.00     0.0314    -0.0541
STD4                                         4.000     0.00     0.0162    -0.0679
Coefficient     : K3=--
                  K2=--
                  K1=4.960000E-003
                  K0=3.620000E-003
Corr.Coef.      : 0.5768

UNK-001     10                               0.77      0.00     -0.0002   0.3257
```

Figure 4: Cadmium analysis with AAS



```
Table of Each Element                              1/21/2009 11:51 AM

Analysis Mode    : Flame/Manual
Analysis Name    : Standard Analysis
Comment          : Cu
Description      :

Meas. Date       : 1/21/2009 11:50 AM
Element : Cu      STD Unit          : PPM          UNK Unit        :

Sample ID   Sample Name                    Conc.     RSD(%)   ABS      REF
STD1                                       0.00      0.00     0.0001   -0.0021
STD2                                       1.00      0.00     0.0161   -0.0012
STD3                                       2.00      0.00     0.0226   -0.0020
STD4                                       4.00      0.00     0.1140   0.0045
Coefficient      : K3=--
                   K2=--
                   K1=2.856000E-002
                   K0=-1.178000E-002
Corr.Coef.       : 0.9488

UNK-001     SLUDGE SAMPLE                  0.41      0.00     -0.0001  0.1699
```

Figure 5: Copper analysis with AAS

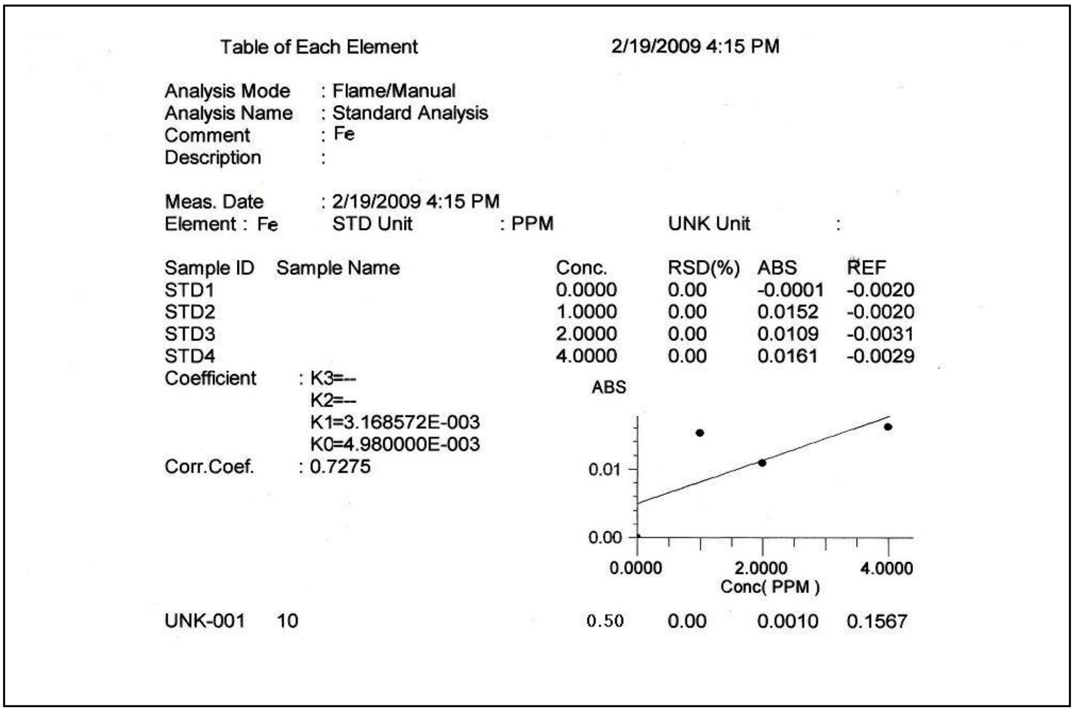


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Table of Each Element                     2/19/2009 4:15 PM

Analysis Mode    : Flame/Manual
Analysis Name    : Standard Analysis
Comment          : Fe
Description      :

Meas. Date       : 2/19/2009 4:15 PM
Element : Fe      STD Unit          : PPM          UNK Unit        :

Sample ID   Sample Name               Conc.     RSD(%)   ABS       REF
STD1                                  0.0000    0.00     -0.0001   -0.0020
STD2                                  1.0000    0.00     0.0152    -0.0020
STD3                                  2.0000    0.00     0.0109    -0.0031
STD4                                  4.0000    0.00     0.0161    -0.0029
Coefficient      : K3=--
                   K2=--
                   K1=3.168572E-003
                   K0=4.980000E-003
Corr.Coef.       : 0.7275

UNK-001     10                        0.50      0.00     0.0010    0.1567
```

Figure 6: Ferum analysis with AAS

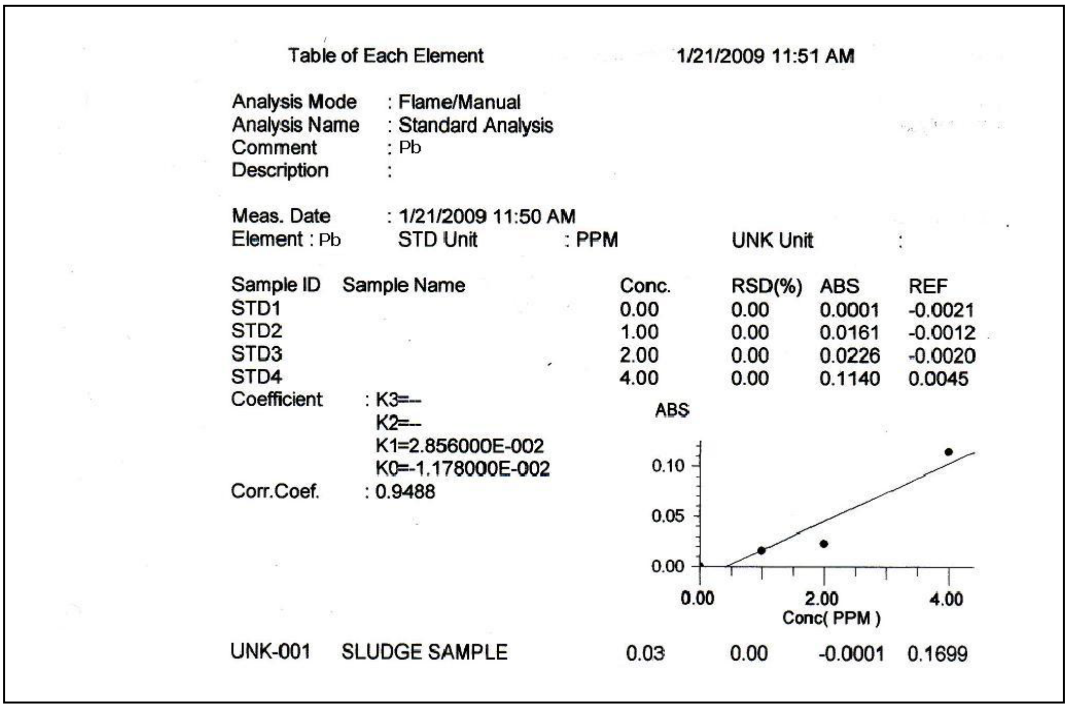

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Table of Each Element                    1/21/2009 11:51 AM

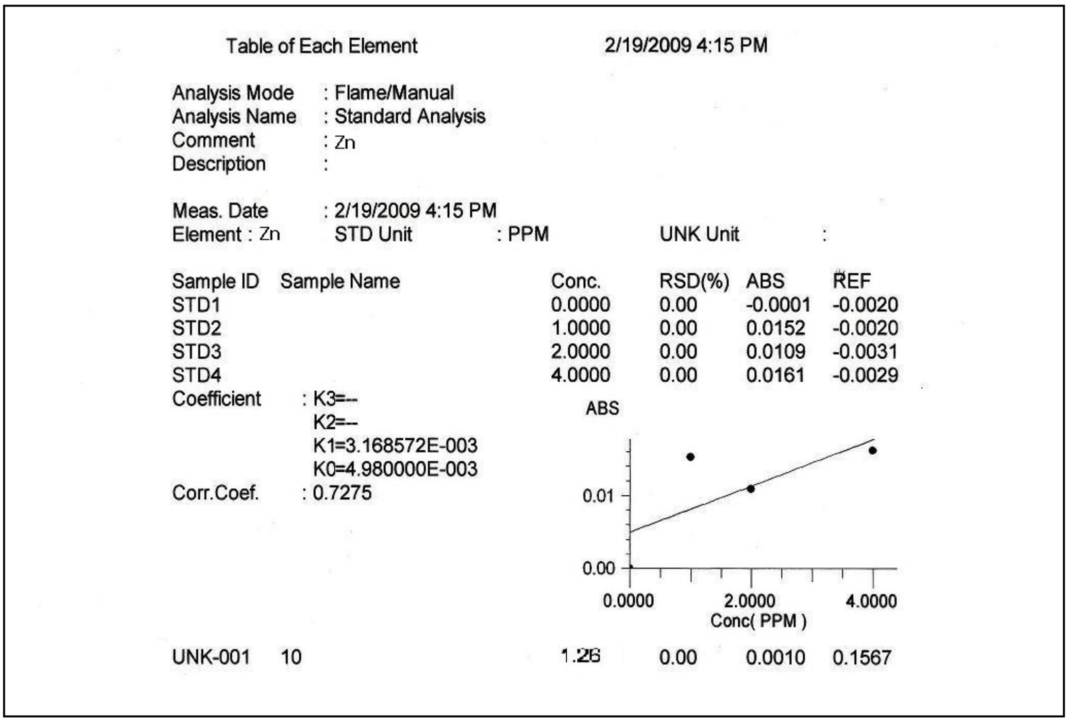
Analysis Mode    : Flame/Manual
Analysis Name    : Standard Analysis
Comment          : Pb
Description      :

Meas. Date       : 1/21/2009 11:50 AM
Element : Pb      STD Unit        : PPM        UNK Unit        :

Sample ID   Sample Name              Conc.     RSD(%)   ABS      REF
STD1                                 0.00      0.00     0.0001   -0.0021
STD2                                 1.00      0.00     0.0161   -0.0012
STD3                                 2.00      0.00     0.0226   -0.0020
STD4                                 4.00      0.00     0.1140   0.0045
Coefficient     : K3=--
                  K2=--
                  K1=2.856000E-002
                  K0=-1.178000E-002
Corr.Coef.      : 0.9488

UNK-001     SLUDGE SAMPLE             0.03      0.00     -0.0001  0.1699
```

Figure 7: Lead analysis with AAS



```
Table of Each Element                  2/19/2009 4:15 PM

Analysis Mode    : Flame/Manual
Analysis Name    : Standard Analysis
Comment          : Zn
Description      :

Meas. Date       : 2/19/2009 4:15 PM
Element : Zn      STD Unit        : PPM        UNK Unit        :

Sample ID   Sample Name              Conc.     RSD(%)   ABS      REF
STD1                                 0.0000    0.00     -0.0001  -0.0020
STD2                                 1.0000    0.00     0.0152   -0.0020
STD3                                 2.0000    0.00     0.0109   -0.0031
STD4                                 4.0000    0.00     0.0161   -0.0029
Coefficient     : K3=--
                  K2=--
                  K1=3.168572E-003
                  K0=4.980000E-003
Corr.Coef.      : 0.7275

UNK-001     10                       1.26      0.00     0.0010   0.1567
```

Figure 8: Zinc analysis with AAS