Regeneration of Used Lube Oil by using different acids (Sulphuric Acid & Nitric Acid)

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ABSRACT

The effect of three different acids (Sulphuric acid and nitric acid) on recycle used lubricating oil to achieve followed by filtration and adsorption of the used oil which carried out first to remove impurities like sand, metal chip and other micro impurities etc. Sulphuric acid yields 80 % while Nitric acid yield 75%. From the experimental analysis of different acids, the Sulphuric acids have high yield compares with Nitric acid and hydrochloric acid. Nitric acid can be used in place of Sulphuric acid and has proved to yield about 75% since cost less to obtain used oil. Recycle of oil is economical process and reduce the environmental pollution produce from the used lube oil. From the experimental analysis of different acids, the Sulphuric acids have high yield compares with Nitric acid and hydrochloric acid. Regenerated oil with Sulphuric acid and Nitric acid has densities 850 Kg/m^3 and 800 Kg/m^3 respectively which is nearer with the fresh oil has density 950 Kg/m^3. Viscosity is the important characteristic of lube oil. Regenerated oil with Sulphuric acid and Nitric acid has viscosity 52 cP and 42 cP respectively which is nearer with the fresh oil has viscosity 58 cP calculated at 40 o C. The use of this method has increased in developed countries reaching up to 50% of the country's need for lubricating oil. Recycling and re-refining of waste into virgin lubricating oil may be suitable option for protecting environment from hazardous waste.

Keywords –*Used Lube Oil Regeneration, Different washing Agents, Sulphuric Acid and Nitric Acid, Adsorption and Filtration*.

1. INTRODUCTION

The lubricating oil acts as a lubricating medium for various automobile parts such as engines and gearboxes. The primary function of lubricating oil is to reduce friction and to provide a heat transfer medium. It also inhibits corrosion and carries away the metal wear parts. The lubricating oil itself doesn't undergo any changes after use but it gets dirty due the addition of combustion products, degraded additives, water and other dust particles during its time inside the engine. The dirt added to the lubricating oil can be obviated and the oil can be restored to its original state.

The basic principle remains the same and utilizes many of the following basic steps

1. Removal of water and solid particles by settling.
2. Sulphuric acid treatment remove gums, greases.
3. Alkaline treatment to neutralize acid
4. Water washing to remove "soap".
5. Clay contact with bleach oil and adsorb impurities
6. Striping to drive off moisture and volatile oils
7. Filtering to remove clay and other solids
8. Blending to specification.

1.1 Main families of additives

- Antioxidants
- Anti-wear
- Metal deactivators
- Corrosion inhibitors
- Friction modifiers
- Anti-foaming agents
- Viscosity index improvers
- Demulsifying
- Emulsifying
- Stickiness improver
- Complexing agent
- Rust inhibitors

Some of these additives include

1. Antioxidants

Antioxidants reduce the rate of oxidation of lubricating oils during its use, thereby reducing the formation of corrosive oxidized products.

2. Detergents - Detergents are added to the motor oil in order to improve engine performance and to prevent material from depositing engine pistons.
3. Antifoam Agents - Foaming of lubricants is an undesirable effect that can cause enhanced oxidation by the intensive mixture of oil with air.

4. Viscosity Modifiers - Viscosity improvers are a long chain, high molecular weight polymers that cause the relative viscosity of oil to increase at high temperatures than at low temperatures. The commonly used additives (alkyl polymethacrylates or copolymers).

5. Pour-Point Depressant- These additives hinder the process of growth of the crystals of paraffin wax, which form in the oil at low temperatures. Polymethacrylates with low molecular masses.

1.2. Sources of Lube oil Contamination

1. The breakdown of the additives and their reaction.
2. Soot and lead from engine blowby
3. Dirt and dust metal particles from engine wear
4. Gasoline or diesel fuels incomplete combustion
5. Water from combustion and blowby vapors.
6. Water from rain water/salt water ingress.
6. The mixing of other materials into used oil.

1.3 Lubricating oil divided in three broad groups

1. Paraffinic

Predominantly straight chains tend to be waxy which have high pour point and good viscosity.

2. Naphthenic

Straight chains with a high proportion of five and to a lesser extent six membered ring structures. Tend to have a low pour point. For this reason, they are used as refrigeration oils. They are highly carcinogenic and are little used in engine oil.

3. Aromatic: Straight chains with six membered ring benzene structures.

1.4 Need of Waste Oil Recycling

1. Improper used oil disposal is waste valuable resource.

2. Every gallon of used motor oil not recovered results in the need to drill for more oil.

3. Most of the crude petroleum produced special hydrocarbon chains for motor oil.

4. Lube base oil can be recovered and 'regenerated' to the quality equal to original form.

5. A large range of waste oils can be recycled.

6. Other options include recovering its heating value.

7. Certain types of waste oils and lubricants can be reprocessed for their direct reuse.

8. Use of waste oils after treatment can be as lube base stock comparable to refined oil.

9. For minimization of pollution and protect environment.

10.Protection of human health & animal from pollution.

11.Recycling can complete about 50% need of lube oil.

12.With recycling process can reduce the cost of lube oil.

1.5 Environmental Pollution

1. The contaminants in waste oil have adverse environmental and health impacts.

2. The presence of degraded additives and contaminants more toxic and harmful to health.

3. By-products of degradation render waste oils more toxic and harmful to health.

4. If put into storm water drains or sewers, they can affect waterways and coastal waters.

5. When dumped in soil or sent to landfill they can migrate into ground and surface waters.

6.Uncontrolled used oils threat to plant and animal life which result in economic losses.

7.Used oil from internal combustion engines accumulates a variety of contaminants.

2. Literature Reviews

In June 2013, by the experimental analysis Udonne J.D and Bakare O.A, it proves that the three acids use effectively activated and remove the slug from the used Lubricating oil and return the oil to it quality form. The clay samples possess high adsorption properties which enabled the removal of impurities and the black color from the lubricating oil. From the results obtained Sulphuric acid yields 90 % while Nitric acid yield 70 % to 80%. Nitric acid can be used in place of Sulphuric acid and has proved to yield about 75% and the cost less to obtain used oil. [1] **In 2015, Studied by Maria Yash P Re-fining** is the use of distilling or refining processes on used lubrication oil to produce high quality base stock for lubricants or other petroleum products. The use of this method has increased tremendously in developed countries reaching up to 50% of the country's need for lubricating oil. Recycling and re-refining of waste into virgin lubricating oil.[3] **As per experimental study solvent extraction process can** be used to recover quality base oil with 94% yield from used lubricating oil. The yield is higher than thin film distillation/clay treatment (70-80%) Solvent extraction of used oil can be carried out at ambient temperature with cheap and low boiling point solvent MEK. If the sludge is allowed to sediment out the solvent to oil ratio is 3.8.[4] **The maximum PSR at butanol as solvent at extraction temperature 45 oC and** 50 oC the PSR

were 11.4 and 11.9 respectively at SOR 6:1 and contact time of 30 minutes. The POL continued to drop between SOR 2:1 and 2.8:1. The optimum PSR and POL was obtained at solvent to oil (SOR) ratio 3:1. extraction temperature 45 oC with contact time 30 Min. The increase in amount of PSR was gradual at solvent to oil ratio (SOR).[8] **The performance was investigated on the PSR (Percentage Sludge Removal) and POL (Percent Oil Loss).** The best results were obtained using composite solvent 25% 2-propanol, 37% 1-butanol and 38% butanone by a solvent to oil ratio of 6:1 at vacuum pressure 600 mmHg and distillation temp. 250 ºC.[10]

3. MATERIAL AND METHODOLOGY

3.1 Methods of Recycling

The recycling processes are the various purification that is used for the treatment of spent engine oil such that it can be used again. The choice of any method determines the level of cleanliness for re-use of the treated oil and various methods have been used for the purification of spent engine oil. The cost of recycling is relatively low compared to its production from crude oil as the number of purification stage involved is reduced.

1. Distillation Clay Method - The sample was made moisture free by first carrying out atmospheric distillation. Distillation is a unit operation in which the constituent of liquid mixture is separated using thermal energy. Basically, the difference in vapors pressure of different constituent at the same temperature is responsible for such separation. The pressure was increased slightly to subside the foam encounter. Mild heating was gently applied to remove the dissolved gases. The distillate obtained was weighed leaving a very dark residue. Filtration is a unit operation in which separation of solid from suspension in liquid with the help of porous medium or screen which retains the solid and allows the liquid passed through is termed as filtration but there was vacuum filtration the filtration which operate with less than atmospheric pressure on the upstream side of the filter medium and atmospheric pressure on the downstream side of the filter medium are referred to as vacuum filtration.

2. Activated Charcoal Clay

The waste lube oil was stirred homogeneously with the help of shaker used lube oil was measured into separating funnel containing Ethyl Acetate. The solution was left at room temperature for 24 hr. there is formation of two layers. The Bottom layer consists of sludge which was removed. Upper layer consists of solution which is treated with activated charcoal for 30 min. charcoal is used for adsorption. charcoal is used for adsorption. Adsorption is phenomenon of enrichment of chemical substances at the surface of solid. Then vacuum filtration was carried out by Clay. Filtration is a unit operation in which separation of solid from suspension in liquid with the help of porous medium or screen which retains the solid and allows the liquid passed through is termed as filtration. but there was vacuum filtration the filtration which operate with less than atmospheric pressure on the upstream side of the filter medium. After filtration oil is used for calculating properties.

3. Fuller's Earth Method

Fuller's earth is used for adsorption purpose based on phenomenon of enrichment of chemical substances at the surface of solid. Then vacuum filtration was carried out by Clay. Filtration is a unit operation in which separation of solid from suspension in liquid with the

help of porous medium or screen retains solid and liquid pass.

4. Acid Clay Method

The suspended particles settled in the waste oil at bottom of flask and the liquid portion was decanted off. The decanted liquid was thermally pre-treated to degrade some of the additives and reduce the workload of the acid. Pre-treated oil was measured in separating funnel and treated with 98% conc. H_2SO_4 in separating funnel with mixture strongly agitated. Agitation refers to the induced motion of the material in a circulatory pattern inside tank or vessel then allowed to settle for 48 hr. After which two layer/phases were formed the sludge was removed from the bottom of separating funnel. After which 100 ml solution of 10% NaOH was added to neutralize the acid it was then allowed to settle for. About 30 min. without agitation. The alkaline phase which is formed at the bottom, was removed and the lube oil washed with hot water. The oil was heated with an elemental burner while connected with vacuum pump.

5. Extraction and Distillation

After extraction process distillation are used to separating solvent from oil and lube oil will separated. There are different solvents are used for the extraction of lube oil. The used lubricating engine oil (W) was collected from motor vehicle service stations. Collected oils were mixed together to represent a complete spectrum of used lubricating engine oil. Pre-treatment of oil involved removal of solid particles by gravity settling. The oil was heated at 140°C and atmospheric pressure for 1 hour to remove residual free and emulsified water. Mixtures were prepared in different ratios from 1:1 to 6:1 by weight of solvent (1-butanol, 2-propanol and mixture of 1-butanol and ethanol) and used lubricating oil. Each system was homogenized at 300 rpm for 30 minutes and placed in water bath for 20 minutes to maintain a constant temperature at 35 oC It was allowed to stand for 24 hours. The procedure was repeated for 20 minutes at 45 oC, 30 minutes at 45 oC, 20 minutes at 50 oC, and 30 minutes at 50 oC.

3.2 Recent Methods for Used oil Recycling

1. Acid Treatment

Recycling of waste engine oils treated using by acetic acid or formic acid. A recycling process was developed which eventually led to comparable results with some of the conventional methods. The recycled oil the potential to be reused in cars' engines after adding the required additives. The recycling process takes place at room temperature. It has been shown that base oils and oils' additives affected by acetic acid. By adding acetic acid to the used oil two layers were separated, a transparent dark red colored oil and a black dark sludge at the bottom of the container. The base oils resulting from other recycling methods were compared to the results of the fresh oil. The comparison showed that the recycled oil produced by acetic acid and formic acid treatment showed excellent results in the properties of the oil comparable to the fresh oil. Using volumetric ratio of 10:1 oil to acetic acid.

Stages of Acid Treatment

1. Addition and Mixing

Take known quantity of used engine oil was measured by measuring cylinder and transferred into a 500 ml beaker. Add acids (acetic acid and formic acid) was measured in a separate 50 ml beaker. The regulator hot plate was switched on and the measured base oil was

placed on top. The temperature of the base oil was maintained at 40–45 oC at this temperature the (acetic acid, formic acid) were introduced into the used engine oil simultaneously with stirring of the mixture for 10 minutes.

2. Sedimentation /Decantation

After acid treatment acidic oil was allowed to settle 24 hours to form sediment at the bottom of the beaker. After this the acidic-oil was properly sediment and decanted into another beaker using piece of cloth while the acidic sludge at the bottom of beaker.

3. Bleaching

The acidic oil in the beaker was subjected to bleaching. The oil was placed on a regulator hot plate and the temperature was maintained at a temperature of 110°C. 6 wt.% of activated bleaching earth was introduced into the oil and the mixture was continuously stirred for 15 minutes. At the end of the bleaching step the bleached oil was neutralized.

4. Neutralization

The bleached oil was neutralized to adjust the pH of the oil to neutrality. At this step, 4 wt.% of the oil of sodium hydroxide was introduced into the bleached oil by taken into consideration the pH of the bleached oil at a given point in time. The bleached oil was neutralized with a continuous manual stirring for 10 minutes. At the end of the bleaching and neutralization steps, the oil was allowed to sediment in the beaker for 24 hours and was decanted into the beaker while the residue at the bottom of beaker was discarded.

5. Sedimentation /Decantation

The oil was allowed to settled in the beaker for 24 hours and was decanted into another beaker, while the residue at the bottom of the beaker was discarded.

6. Filtration

The sediment oil was finally filtered using a filter cloth and the filtrate was collected in a filtration flask and was observed to be clear the residue (filter cake).

2. Solvent Extraction Using Sulphuric Acid and Activated Carbon

The sample of spent engine oil is mixed with 70% concentration of Sulphuric acid in the ratio 10ml to 1ml and then heated to 60 ^{0}C for one hour and was allowed to settle for six hours so as to enable the insoluble mater and water remain to settle at the bottom of the beaker. The treated oil is decanted and sludge formed removed from the beaker. The treated oil is then contracted with activated carbon, and treated at 200 ^{0}C – 250 ^{0}C for 2 -3 hours. The treated oil is filtered and allowed to cool. After that the treated oil is analyzed for various properties. This is unique method by which the spent oil is adequately recycled for reuse and the cost of recycling is relatively low compared from its production from crude oil as the numbers of purification stages are reduced. As per study carried out it was discovered that when 25 liters of spent oil was recycled and 10 liters of lubricating oil was obtained from the process whereas 220 liters of crude oil would be required to produce the same 10 liters of oil.

4. EXPERIMENTAL ANALYSIS

4.1 Selected Process

Treatment with different Acids (Sulphuric Acid, Nitric Acid and Hydrochloric Acid) and Activated Charcoal and Alkali (lime) to be used for the neutralization process of acid which added in the treatments process.

1. Acid Treatment

Known quantity of used engine oil was measured in a beaker and Sulphuric acid was measured in a separate

beaker. The temperature of the base oil (used engine oil) was maintained at 40 - 45 oC at this temperature the Sulphuric acid was introduced into the used engine oil simultaneously with stirring of the mixture for 10 minutes.

2. Sedimentation

After acid treatment acidic oil allowed to settle 4 hours to form sediment at the bottom of the beaker. The acidic-oil was properly sedimented and decanted into another 500ml beaker while the residue (acidic sludge) at the bottom of the beaker was discarded.

3. Neutralization

In the neutralization steps the oil was allowed to sediment in the beaker for 4 hours and was decanted into the beaker while the residue at the bottom of beaker.

4. Bleaching using activated Carbon

At the end of the acid treatment step if the acidic oil is to be bleached using activated carbon. The temperature was increased and maintained at a range of 130 oC–140 oC the activated carbon and hydrated lime was used.

5. Sedimentation /Decantation

In this stage the oil was allowed to sediment in the beaker for 4 hours and was decanted into another beaker while the residue at the bottom of the beaker was discarded.

6. Filtration

The sedimented oil was finally filtered using a filter cloth and the filtrate was collected in a filtration flask and was observed to be clear while the residue (filter cake).

4.2 Materials

1. 98% Sulphuric Acid (H_2SO_4)
2. Nitric Acid (HNO_3)
3. NaOH
4. Activated Charcoal
5. Used Lube Oil

4.3 Experiment Procedure

1. Purification of the used lubricating Oil Filtration of the used oil carried out to remove impurities such as sand, metal chips, micro impurities that contaminated lube oil.
2. Three liters of the used oil was filtered for sample.
3. The lube oil was allowed to settle for twelve hours.
4. The suspended particles in the used oil allowed to settle at bottom of flask and the liquid portion decanted.
5. The decanted liquid was preheated to degrade some of the additives and reduce the work load of the acid.
6. 100 ml of the pre- treated oil was measured into three different beakers for the first sample A at 10 ml each.
7. The first beaker was treated with 98% Sulphuric acid (H_2SO_4), the second beaker with hydrochloric acid and third beaker with nitric acid.
8. Each conical flask was shaken thoroughly to ensure homogenous mixing of the acid and the samples allowed to stand for thirty minutes.
9. The oil samples were measured into a separating funnel and acids sludge was discharged at the bottom of the separating funnel. This was removed gradually and repeated for the two other samples (B & C).
10. 100 ml solution of 10% NaOH (Caustic soda) was added to neutralize the acid of the three oil samples.
11. After that mixture is treated with activated Charcoal to removal of impurities.
12. The oil was allowed to sediment in the beaker for 4 hours and was decanted into another beaker while the residue at the bottom of the beaker was discarded.

13. The sedimented oil was finally filtered using a filter cloth and the filtrate was collected in a filtration flask and was observed to be clear while the residue (filter cake).

14.The method was then repeated for samples of nitric acid and hydrochloric acid.

15. Finally the refined lube oil obtained for analysis.

16. Calculate the % yield of treated oil with different acids.

17. Compares the % yield and select the best acid for the treatment of used lube oil.

4.4 Production of Activated Charcoal

1.Crush the rice husk/sugarcane bagasse/coconut shell and make powder.

2.Adsorbent material washed carefully first with distilled water.

3. Then deionized water to remove particulate material from their surface.

3. Dried it at room temperature to avoid release of color into the aqueous solution.

4. The activation of adsorbent is carried out by treating it with concentrated Sulphuric acid (0.1N) and is kept in an oven maintained at a temperature range of 150 ºC 24 hr.

5.The pH measurements with a pH meter.

6. Then neutralize them with help of 0.1N NaOH or 0.1N H_2SO_4.

7. Again it is washed with distilled water to remove the free acid and put in to oven for removal of moisture and then adsorbent is passed from 500-micron mesh size and collected for experiment use.

5. RESULTS AND DISCUSSION

5.1 Analysis of Various Properties

1. Density of Oil

Density of a substance is equal to the mass of a substance divided by the volume of the substance. The temperature at which the density is been measured must be known for density changes as temperature changes.

Density = Mass of oil / Volume of oil

3. Specific Gravity

Specific gravity is the ratio of the density of the material to density of the equal volume of water. Specific gravity can be calculated by using specific gravity bottle.

2. Viscosity

A decrease in the viscosity of engine oil indicates that the oil is contaminated. Lubrication oils are identified by the Society of Automotive Engineers (SAE) number. The greater or higher the SAE viscosity number and the heavier or more viscous the lubricating oil. Viscosity is defined as the force acting on a unit area where the velocity gradient is equal at a given density of the fluid. Viscosity is strongly depending on the temperature. As the temperature increase viscosity decrease. Viscosity can be calculated by using viscometer.

5.2 % Yield of Regenerated Used Oil

Yield of oil that produce from the used oil is depends on the value or amount of used oil feed. Yield of oil can be calculated by using amount of oil produce from the used lube oil and total amount of oil and sludge produce after dehydration of the both sludge and oil. With dehydration water removal from the oil and sludge.

1. % Yield of oil

% Yield of oil = [Amount or Weight of oil produce / Amount of Total Product] * 100

(Weight of Total Product = Oil + Sludge after dehydration)

1. % Yield by using H_2SO_4 = 80
2. % Yield by Using HNO_3 = 75

5.3 Properties of Fresh and Used Oil

Sr. No.	Properties	Fresh Oil	Used Lube Oil
01	Density (Kg/m^3)	950	860
02	Viscosity @ 40 °C(cp)	58	32
03	Specific Gravity	0.95	0.860

Table Properties of Fresh and Used Oil

5.4 Properties of Regenerated Oil

Sr. No.	Properties	By using H_2SO_4	By using HNO_3
01	Density (Kg/m^3)	850	800
02	Viscosity @ 40 °C(cp)	52	42
03	% Yield	85	75

Table Properties of Regenerated Oil

CONCLUSION

Sulphuric acid yields 80 % while Nitric acid yield 75%. Nitric acid can be used in place of Sulphuric acid and has proved to yield about 75% since cost less to obtain used oil. Recycle of oil is economical process and reduce the environmental pollution produce from the used lube oil. Different acids followed by adsorption with activated charcoal method requires Low overall cost, low energy and less retention time. From the experimental analysis of different acids, the Sulphuric acids have high yield compares with Nitric acid and hydrochloric acid. That will be reduce the degree and nature of contamination and environmental/health risks with disposal. With help of Activated charcoal, we can remove the various impurities presents in the recycled used oil by using different acids. The major drawback to the acid/clay method is the difficulty of removal of the clay sludge. Hence the activated charcoal beneficial than the clay for removal of impurities. Regenerated oil with Sulphuric acid and Nitric acid has densities 850 Kg/m^3 and 800 Kg/m^3 respectively which is nearer with the fresh oil has density 950 Kg/m^3. Viscosity is the important characteristic of lube oil. Regenerated oil with Sulphuric acid and Nitric acid has viscosity 52 cP and 42 cP respectively which is nearer with fresh oil has viscosity 58 cP at 40 oC

FUTURE SCOPE AND BENEFITS

1. With Recycle of oil can reduce the cost of oil.
2. This process reduce environmental pollution produce due to used oil that directly mixed with soil and water.
3. Recycle can produce the oil which id equal or better than the virgin oil.
4. Recycle is cost saving and beneficial process which produce the low sludge.
5. Recycle helps to complete demand of lube oil supply.
6. with recycling or reuse of the used lube oil helps to reduce pollution, cost of oil and loss of oil.

7. As per the feature need this process are economical and ecofriendly.
8. The use of this method has increased in developed countries reaching up to 50% of the country's need for lubricating oil.
9. Recycling and re-refining of waste into virgin lubricating oil may be a suitable option for protecting the environment from hazardous waste.

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Nomenclature

AAS - Atomic Absorption Spectrometry

AC – Activated Carbon/ Charcoal

cP – Centi Poise

CCA – Chambers & Collection Tanks Analysis

MEK - Methyl Ethyl Ketone

PSR - Percent Sludge Removal

PDA - Propane De-asphalting Process

POL - Percent Oil Loss

SAE - Society of Automotive Engineers

TAN - The Total Acid Number

TBN - Total Base Numb