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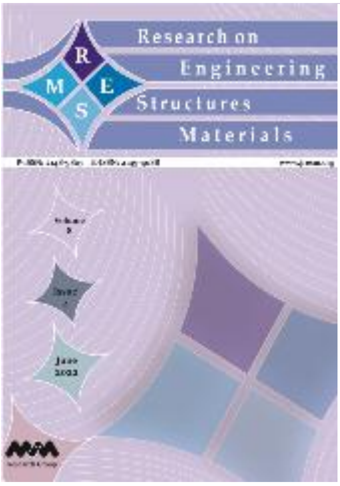

Recycling of waste lubricating oil using ultra filtration membrane and modeling and prediction of its rheological behavior using Gauss-Newton algorithm

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Research Article

Recycling of waste lubricating oil using ultra filtration membrane and modeling and prediction of its rheological behavior using Gauss-Newton algorithm

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Abstract

In this research, ultrafiltration membrane made of Polyacrylonitrile (PAN) was employed to recover the lubricating oil from the waste oil in tandem with other treatment methods. FESEM was also used to analyze the structure of the membrane. The resultant recycled oil was monitored with Rheometer, FTIR and UV Spectrometer and it was found that the method successfully removed >90% of contaminants and heavy metals from the waste oil and improved the properties of the oil. The proposed method proved to be very economical and gave very promising results to reclaim the waste engine oil that can be re-used for various purposes. Also, the rheological behavior of engine oil is studied by a proposed mathematical model. Given that the suggested model was non-linear, the Gauss-Newton (GN) algorithm was employed to determine the ideal values for the model parameters. The experimental results obtained for virgin, waste and recycled lubricating oils from rheometer were used for validation of the model. The proposed model was compared with Reynold's exponential model based on statistical methods (co-efficient of determination (R2) and mean square error (MSE)). It was found that that the proposed model can predict the change in viscosity with temperature with very high degree of accuracy (>99%).

1. Introduction

Lubricating oils work by acting as a separating film between two surfaces that rub against each other and thus reduce friction and wear. Lubricating oils are made up of two materials, first one being the base oil obtained from distillation of crude oil and the second component is made up of numerous additives that improve the base oil's properties and performance. These additives include friction modifiers, pour-point depressants, metallic detergents, viscosity improvers, zinc dialkyl-dithiophosphates, etc. [1]. The engine lubrication system is designed to overcome the mechanical losses of the engine and eliminate the wear products of the machine by preventing increased wear, overheating, and seizing of friction surfaces and reducing the consumption of the specified power [2]. Lubricants are highly specialized products carefully designed to perform many important functions such as starting the engine, reducing friction, protecting machinery from rust and corrosion, lubricating engine parts, and more [3]. After use, the oil deteriorates and loses its properties, so it cannot be used for its intended purpose. Lubricant properties such as TAN, ash content, metal content, moisture content, carbon residue and color scale increase while TBN decreases [4]. Waste oil has a higher viscosity than fresh oil due to

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excessive evaporation of soot and sediments, oxidation products, water, or light fractions [5]. Lubricant deterioration occurs when various additives or foreign substances (metal powder, sulphur, water, carbon, ash, etc.) contaminate the oil, change its chemical composition, and affect its physical properties [6]. Because it is insoluble, stable, and high in heavy metals and other contaminants, used oil must be collected and recycled to avoid polluting the environment [7].

Disposing of used lubricating oil is a huge business that has destructive effects on both environment as well as health of any living organisms. The reason being that almost 20000-25000 barrels of used lubricating oil are collected by each state in India per month and rest of it is dumped into soil or water bodies where it affects both plants and animals. This collected oil is then sold off to industries where they burn it as low-grade oil without removing its harmful contaminants such as metal particles that are carcinogenic in nature. As the oil burns, these contaminants go up into the air and can cause major respiratory problems. A single engine oil change can contaminate huge water bodies when it is disposed of into sewer or drain. These oils are direct substitutes for heating oils and LDOs in foundries, bitumen mills, lead smelters and the battery industry.

Used lubricating oil is a high pollutant material and its impact on the environment is hazardous. The effect of the pollutants on the human health is extremely toxic as the used oil contains some of the deadly carcinogenic contaminants. Oil once used, can be cleaned of its contaminants, and recycled and reused for various purposes. Engine oil is generated from non-renewable petroleum fuels, therefore finding new sources will be more challenging in the future. Oil prices rise as supplies become scarcer. As demand has increased over the past few years, dwindling oil reserves have forced explorers to look elsewhere.

Waste oil can be converted into lubricating base oil, fuel oil, or utilized as a feedstock to create other products with economic value after being adequately cleaned and treated to eliminate pollutants [8]. Various recovery techniques have been created and employed over time based on the oil basis and contaminants. These recycling techniques include acid/clay treatment method [9,10], vacuum distillation and hydrogenation/clay treatment [11,12], solvent treatment method [13], membrane filtration technology [14], Pyrolysis using microwave heating [15], recycling using a washing agent [16], bentonite treatment method [17,18].

Studying the rheological characteristics of used engine oil is crucial for effective recycling. [19]. The study of material flow and deformation is known as rheology. Viscosity is the property of rheology that defines the measure of the resistance to deformation. Rheology especially deals with the study of liquid and soft matter [20]. Because the viscosity of the oil is essential to the engine oil's primary functions, including wear control, friction reduction, and load carrying capacity, it must constantly be at an appropriate level. The viscosity of the oil directly affects the engine oil's strength. Oil can be blended with various additives to increase its viscosity and keep it there for longer. Addition of physical contaminants, polymerization, anti-freeze, wear particles, etc. to the engine oil during its use compromises the viscosity of the oil and the oil ceases to function properly. This leads to decline in the quality of the oil and also causes further damage to the vehicle engine parts. Thus, it is important that the engine oil should be changed after regular intervals so that deterioration of engine performance is averted.

A significant reduction of oil viscosity can cause excessive wear as the oil film becomes thin, increase in friction which in turn causes excessive energy consumption and heat generation, increased particle contamination sensitivity due to reduction in oil film thickness, and failure of oil film at high temperature and high loads, or during start-up of vehicle engine. Similarly, high viscosity can also cause extreme heat generation which causes oxidation of oil, sludge collection, cavitation and lubrication starvation as there is insufficient oil flow, excess energy consumption and poor pumpability during cold-start [21]. Engine oil displays non-Newtonian shear thinning behavior.

Viscosity is temperature dependent characteristic. To achieve better tribological performance, proper maintenance of rheological behavior with temperature is essential [22]. Engine oil must have the ideal viscosity for proper operation; it should not be too thick to pour at low temperatures, nor should it become too thin as the temperature rises so that it won't properly form an oil film between two parts as the engine temperature varies from room temperature to 150°C while in operation. If the oil is too thick, the engine must work harder, producing more heat; if the oil is too thin, there is insufficient friction, resulting in both situations in excessive wear and tear. Beyond certain temperatures, rheological properties remain unchanged showing good flow behavior.

This paper proposes the use of ultrafiltration membranes after waste oil pre-treatment as a promising method for treating lubricating oil. In addition to removing metal particles and dust from used lubricating oil, the ultrafiltration membrane process can improve the oil's flash point and fluidity. The main aim of the proposal is to find a low-cost method to recycle the oil and achieve superlative results by producing higher quality oil. The methods that have been previously used have become outdated and are very expensive in terms of capital investment and operating cost. Also, the purity level achieved is not of a good standard thus the recycled oil is used as a low-grade fuel. Distillation, clay process, and acid treatment method are commonly used processes in India. Distillation process is a high-cost technology and needs huge financial input for plant set-up, thus discouraging budding entrepreneurs to take up the challenge as this will not only prove to be a very successful business if set-up is low-cost but also will contribute a lot towards environmental pollution and health of all living organisms. Clay and acid treatment methods are old methods and do not yield high quality results. Membrane filtration is a promising technology to dispose of used lubricants as it has a higher rejection rate than other methods.

Numerous benefits of membrane filtering include great separation efficiency, simple scaling, a less environmental impact, and good product quality. Additionally, processes can be integrated with one another, such as biological waste disposal processes. [23,24]. Due to its continuous operation requirements, low power consumption, ease of adaptability, versatility, and relatively mild operating conditions when compared to traditional separation processes like distillation, evaporation, and crystallization, membrane separation technology is a rapidly developing field of science. [25]. This process, carried out at a pressure of 0.1 MPa and a temperature of 40 °C [26], enhances the treated oil's lubricating qualities by removing metal flakes, soot, and debris from used engine oil.

In this paper a mathematical model is also developed so that change in viscosity during operation of engine vehicle as an explicit function of temperature can be predicted. Throughout the operation, it is anticipated that other system parameters like shear stress, speed, torque, etc. will remain constant. Given that the suggested model was non-linear, the Gauss-Newton (GN) algorithm was employed to determine the ideal values for the model parameters. To assess the models' efficacy, the proposed and Reynold's models were contrasted with the findings of the experiment.

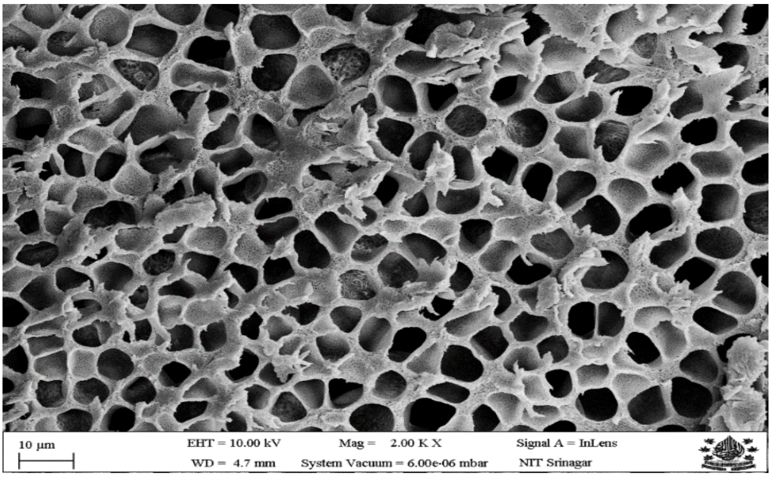
2. Materials

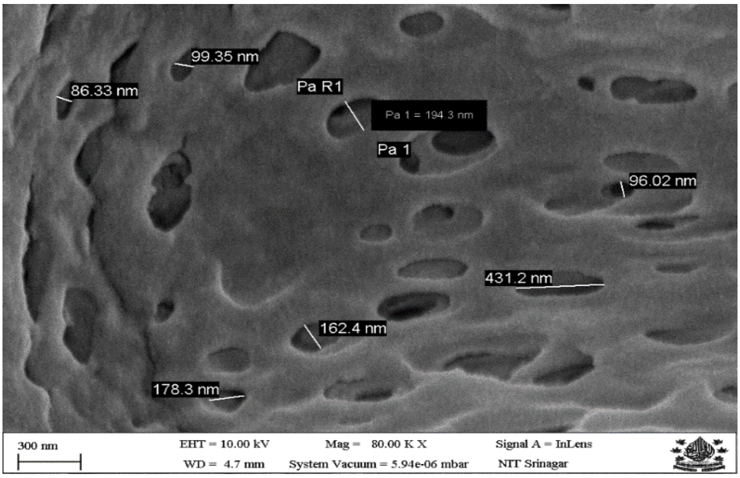
In this study used lubricating oils SAE 5W30 were obtained from different sources. The waste engine oil to be recycled was used for almost 3000 miles. The recycling process comprised of two steps; Pre-treatment and Filtration. The pre-treatment of oil was carried out by activated charcoal, activated bentonite clay, acetic acid, sodium hydroxide for neutralization and Whatman filter paper grade 1 (circular sheet, nominal thickness 180 µm, particle retention 11 µm). Ultra-filtration membrane shown in Fig. 1 was used for the separation process.

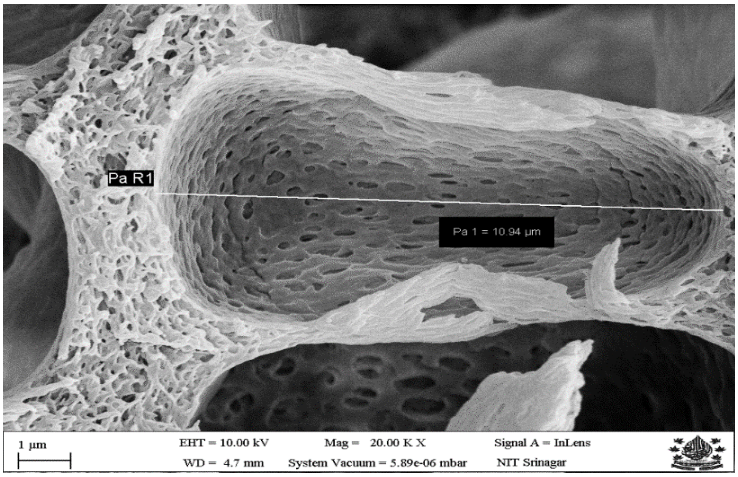
Fig. 1. Ultra-filtration membrane sheet (PAN)

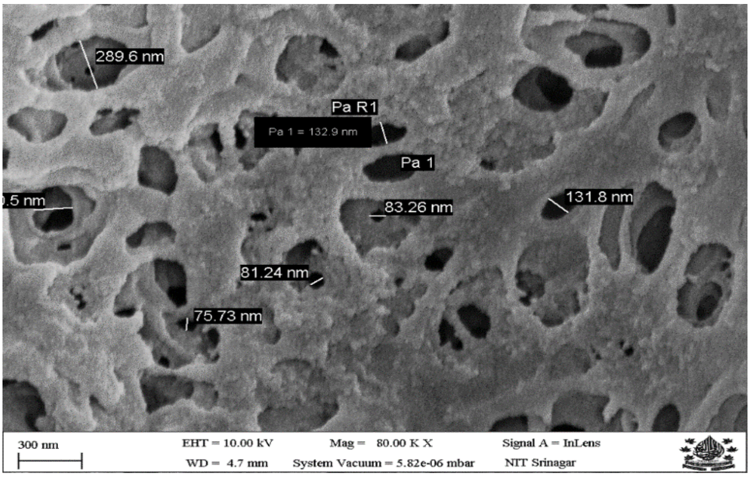
2.1. Ultra-Filtration Membrane (Structural Analysis)

A flat sheet ultra-filtration membrane made of Polyacrylonitrile (PAN) with MWCO of 50 kDa was used for the separation process. Polyacrylonitrile (PAN) is a synthetic polymer with the linear formula (C3H3N)$_n$. Polyacrylonitrile (PAN) membranes combine superior selectivity, high flow rates and low pressures while maintaining quality and process efficiency. The unique nanofiber mesh structure combines ultra-fine pores with ample open space to facilitate fluid flow and filter out particles as small as 0.2 microns. The membrane is made by extruding ultra-thin PAN nanofibers onto a polyester substrate. The nanofibers form a dense mesh that traps particles, colloids, and bacteria larger than zero.




99.35 nm
86.33 nm
Pa R1
Pa 1 = 194.3 nm
Pa 1
96.02 nm
431.2 nm
162.4 nm
178.3 nm
300 nm
EHT = 10.00 kV
WD = 4.7 mm
Mag = 80.00 K X
System Vacuum = 5.94e-06 mbar
Signal A = InLens
NIT Srinagar


Pa R1
Pa 1 = 10.94 µm
1 µm
EHT = 10.00 kV
WD = 4.7 mm
Mag = 20.00 K X
System Vacuum = 5.89e-06 mbar
Signal A = InLens
NIT Srinagar


289.6 nm
Pa R1
Pa 1 = 132.9 nm
Pa 1
.5 nm
83.26 nm
131.8 nm
81.24 nm
75.73 nm
300 nm
EHT = 10.00 kV
WD = 4.7 mm
Mag = 80.00 K X
System Vacuum = 5.82e-06 mbar
Signal A = InLens
NIT Srinagar

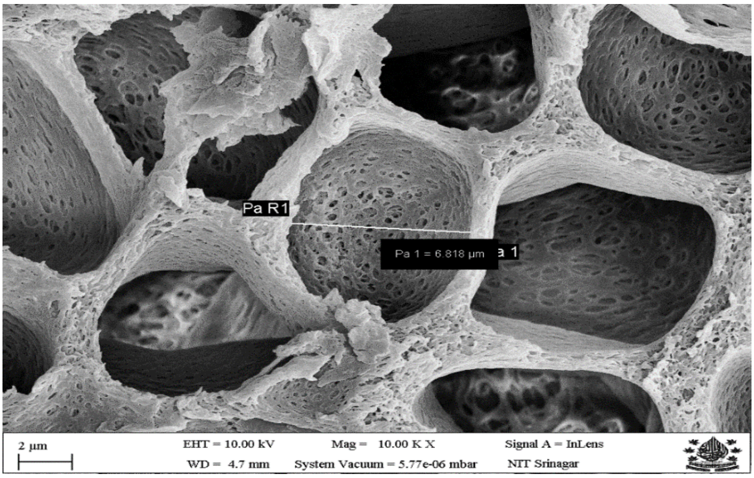


Fig. 2. FESEM images of Polyacrylonitrile UF membrane

The 2-micrometer mesh also has a relatively open structure, allowing water or aqueous solutions to pass through quickly with very little pressure. The main benefits of PAN membranes are:

- Very high flow rate
- Low pressure requirements
- Hydrophilic; no pre-wetting necessary

To analyze the structure of the membrane, FESEM (Field Emission Scanning Electron Microscopy) pictures from the surface and cross-section of the membrane are taken at various magnifications and are displayed in Fig. 2. The membrane has an asymmetrical structure made up of a porous bulk that has separate finger-like chambers contained in a porous solid matrix and a thin, thick surface layer. The surface layer is in charge of pollutant penetration or retention, whereas the pores serve as a mechanical support and let filtered oil move through. Since the heavy metal particles are removed at the pre-treatment stage, thus, the contaminants having smaller size are retained on this membrane.

3. Methodology

3.1. Pre-Treatment

The used engine oil is highly viscous due to the presence of heavy metal particles, dust, dirt, oxidation, polymerization, soot, other contaminants such as anti-freeze, etc. The particle size of these contaminants varies largely in size (20 microns and smaller). Thus, it was not possible to filter the viscous and thick oil using ultrafiltration membrane as the set-upmade of aluminum was not able to withstand the high pressure. So, pre-treatment of used oil was carried out before the ultra-filtration process. It served following purposes:

1. To decrease oil viscosity to improve the flux of membrane filtration [27].

2. To reduce the number of large particles in advance which otherwise could damage the ultrafiltration membrane.

3. To reduce the viscosity of oil sufficiently such that ultrafiltration could be carried out at mild conditions of pressure and temperature.

The stages of pre-treatment included:

1. Acid Treatment: The used lubricating oil was placed in a beaker and a magnetic stirrer was used to heat the oil to 40-50°C. During heating process acetic acid was added slowly to the oil and the mixing was done continuously for about 10 minutes.

2. Sedimentation: The oil mixed with acid was left in the container for about 24 hours and allowed to cool down. A sediment layer was formed at the bottom of the container. This oil was then filtered using Whatman filter paper shown in Fig. 4, to separate it from the sludge at the bottom of the container.

3. Bleaching: Activated charcoal and activated bentonite clay shown in Fig. 3, were added while heating the oil again to 100°C and the solution was mixed for 15 mins.

4. Neutralization: After bleaching, sodium hydroxide was added to the solution to neutralize the effects of acetic acid by reducing the pH of the oil.

5. Filtration: The solution was again allowed to settle down for 24 hours and then again filtered using Whatman filter paper.

Fig. 3. Activated charcoal and activated bentonite clay

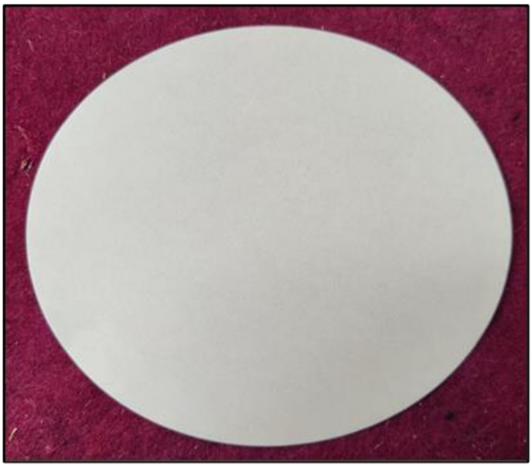


Fig. 4. Whatman Filter Paper (Single sheet)

3.2. Membrane based filtration process

After pre-treatment process, the resultant oil obtained was less viscous and major contaminants were removed during the pre-treatment process. The oil was then allowed to undergo ultrafiltration using an experimental set-up shown in Fig. 5 designed for this purpose.

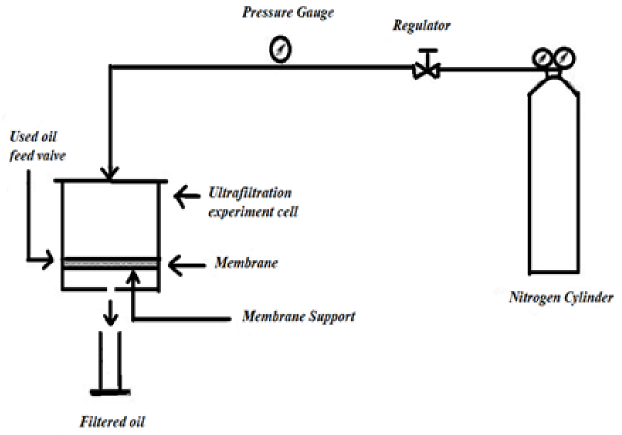


Fig. 5. Experimental set-up for ultrafiltration process

The experimental cell was fabricated from aluminum, since it is cheap and easily available. It had two compartments separated by a support for membrane. Used oil was introduced on the upper side of membrane by feed valve which was then closed. The oil was introduced at the high-pressure side where the pressure was maintained by nitrogen gas. The permeate was collected on low pressure side in a container for collection of recycled oil.

Fig. 6. Virgin, used and recycled engine oil

4. Result Analysis and Discussion

4.1. Fourier Transform Infrared Spectroscopy

Perkin-Elmer, Model Spectrum-Two (ATR Diamond) FTIR (Fourier Transform Infrared) Spectrometer was employed for characterization. The FTIR analysis method scans test materials and examines chemical characteristics using infrared light. FTIR was used to identify contaminants and functional groups in recycled, virgin and waste oil samples.

The ability of FTIR to provide a composite molecular fingerprint of engine oil makes it an extremely valuable analytical tool for condition monitoring of vehicle engines and engine oil as each constituent of oil has its own fingerprint. These constituents present a unique spectrum as they absorb radiation at that spectral location. The remaining radiation is transmitted and is shown as %Transmittance which depends on the amount of constituents present. This allows detection of all contaminants ranging from soot, nitration, oxidation to ZDDP, etc. which determines condition of the oil.

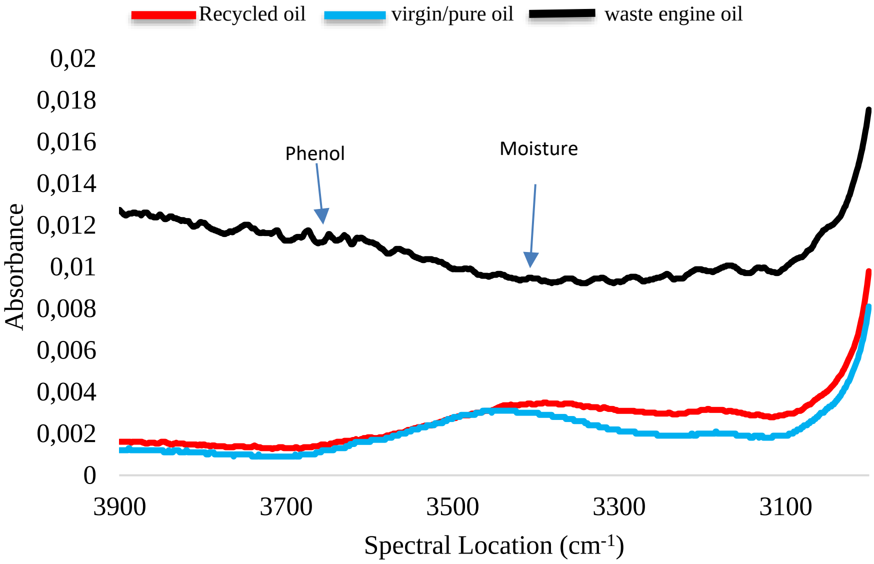


Fig. 7 FTIR of oil sample from spectral locations 4000 to 3000 cm^{-1}

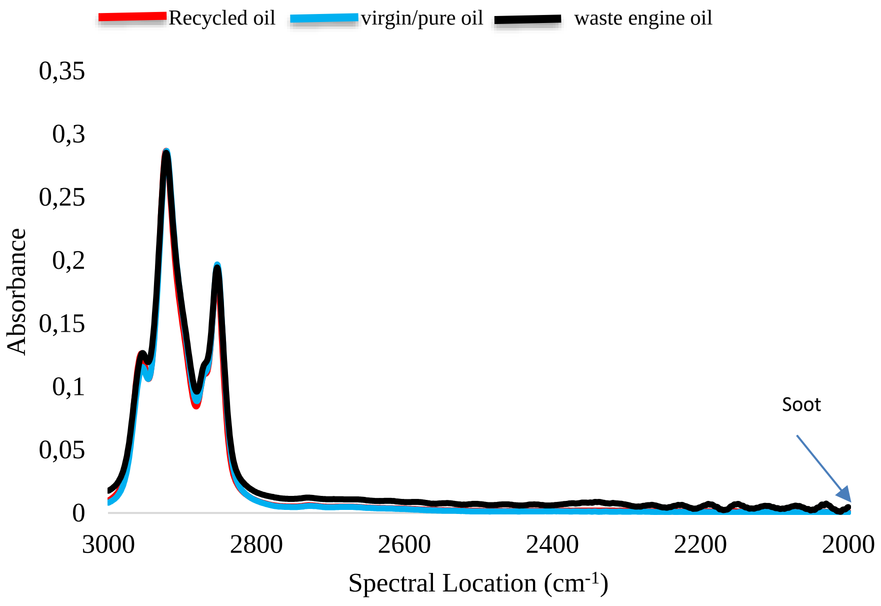


Fig. 8. FTIR of oil sample from spectral locations 3000 to 2000 cm^{-1}

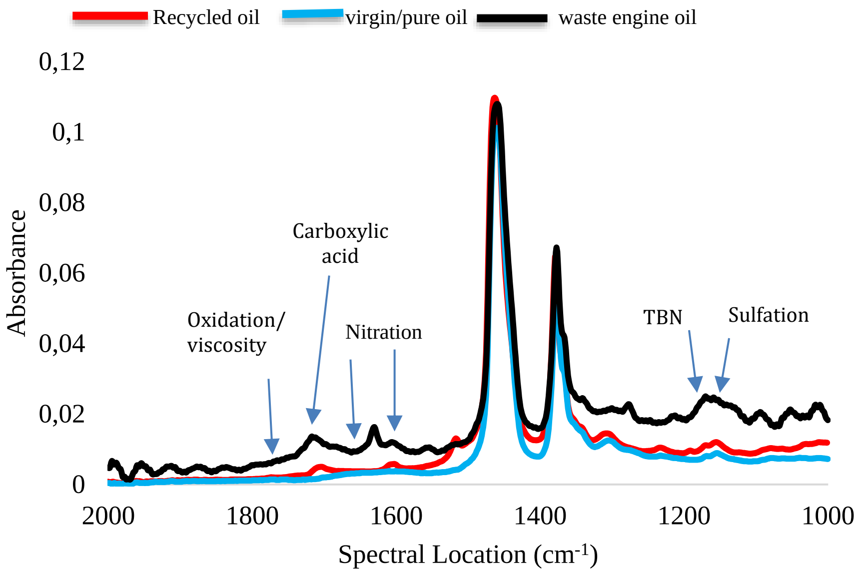


Fig. 9. FTIR of oil sample from spectral locations 2000 to 1000 cm^{-1}

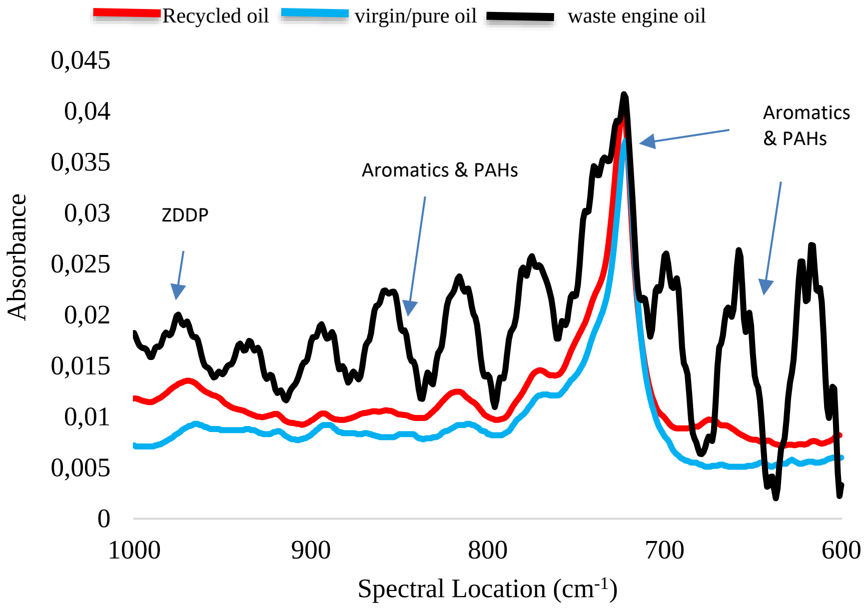


Fig. 10. FTIR of oil sample from spectral locations 1000 to 600 cm^{-1}

Table 1. Values of all oil samples' absorbances as determined by FTIR Spectroscopy

Contaminants/Functional groups	Spectral Location (cm^{-1})	Virgin oil	Used Engine Oil	Recycled oil
Phenol	3650	0.0012	0.011486	0.00143
Moisture	3400	0.003	0.00943	0.0034
Soot	2000	0.0004	0.004716	0.000782
Oxidation/Viscosity	1750	0.0013	0.007535	0.002177
Carboxylic Acid	1725	0.0014	0.011352	0.002701
Carboxylic Acid	1700	0.002	0.011396	0.00476
Nitration	1650	0.0032	0.009706	0.003707
Nitration	1600	0.0038	0.011486	0.005639
TBN	1170	0.008	0.025028	0.011129
Sulfation	1150	0.0086	0.023329	0.01153
ZDDP	980	0.0078	0.018	0.012602
Aromatics & PAHs	850	0.0083	0.019588	0.010239
Aromatics & PAHs	720	0.0365	0.0413	0.03929
Aromatics & PAHs	650	0.0052	0.016238	0.007756

The results of FTIR shown in Figs. 7-10, are depicted in form of absorbance values which are directly proportional to particular constituent quantity at certain spectral location. From the absorbance values in Table 1 it is observed that the contaminant level of recycled oil has decreased considerably when compared to that of used engine oil and the values are closer to that of virgin/pure oil thus depicting the successful removal of contaminants/ functional groups that had caused impurification of oil.

It is clear from the Table 1 that there is significant reduction in the level of contaminant after recycling. The percentage reduction in the level of the contaminants is calculated using Eq. (1).

$$\emptyset = \left[\frac{R.O - V.O}{U.O - V.O}\right] \times 100\% \tag{1}$$

Where ∅ is the Percentage reduction in the level of contaminants after recycling

R.O is the absorbance value of Recycled oil obtained from FTIR Spectroscopy

V.O is the absorbance value of Virgin/pure oil obtained from FTIR Spectroscopy

U.O is the absorbance value of Used oil obtained from FTIR Spectroscopy

The results are given in the Table 2.

Table 2. Percentage reduction in the level of contaminants after recycling

S.No.	Functional group/ Contaminant	Percentage reduction after recycling
1	Phenol	> 97%
2	Moisture	> 93%
3	Soot	> 91%
4	Oxidation/Viscosity	> 85%
5	Carboxylic Acid	> 86%
6	Nitration	> 92%
7	TBN	> 81%
8	Sulfation	> 80%
9	ZDDP	> 52%
10	Aromatics & PAHs	> 82%

The results presented in the Table 2 show that the contaminants are successfully removed from the waste engine oil and there is decrease in viscosity. There is marked improvement in the characteristics of recycled oil as the impurities are successfully removed. Also, there is a significant improvement in the color, as the color changes from pitch black of used oil to darker shade of yellow resembling the color of virgin/pure oil.

4.2. Rheological Study

An AntonPaar MCR 102 rheometer with a Peltier device for temperature control has been utilized to predict the rheological behavior of recycled, virgin and waste lubricating oils. Several experiments were conducted, including determining the fluid behavior and the relationship between viscosity and temperature at various shear rates in continuous rotation mode. Tests were conducted utilizing 50 mm parallel plate geometry. This measuring method is best suited for measuring viscosity of oils and offers quick temperature stability. The distance between two plates was kept at 0.1 mm. The rheometer has a special TruGapTM feature. Independent of temperature and thermal expansion, TruGapTM monitors the gap and accurately adjusts the desired position. Each measurement was performed three times, and the mean was computed, in order to obtain precise data and evaluate the reliability of experimental results.

Changes in rheological parameters (viscosity, shear stress and torque) with temperature were investigated. The change in viscosity with temperature is shown in the graph in Fig. 11. We can see that the viscosity decreases with increasing temperature as the oil becomes thinner with higher temperature and the dependence is not linear. Lubricants exhibit non-Newtonian shear thinning. Lubricating oil is thick at low temperatures and becomes thinner as the temperature increases, thus decreasing the viscosity. It is pertinent to mention that the lubricating oil should not be too thick at low temperatures that makes it difficult to pump or pour, nor should it become too thin at high temperatures that its load bearing capacity is reduced. Beyond certain temperatures, rheological properties remain unchanged showing good flow behavior. Different oils show different variation in viscosity with temperature.

It has been reported that better rheological behaviour as a function of temperature leads to better tribological performance [28]. The presence of contaminants in the oil increases the viscosity and thickness of the oil. From the Fig. 11, it is observed that the viscosity of recycled oil is reduced considerably after removal of contaminants and is closer to that of virgin/pure oil and obeys Reynolds's equation given by Eq. (2):

$$\mu_o = be^{-aT_A} \tag{2}$$

where μo is the dynamic viscosity at atmospheric pressure and TA is absolute temperature. The fluidity was found to be maximum for virgin oil and least for used engine oil due to presence of contaminants. The fluidity of recycled oil is shown in Fig. 12.

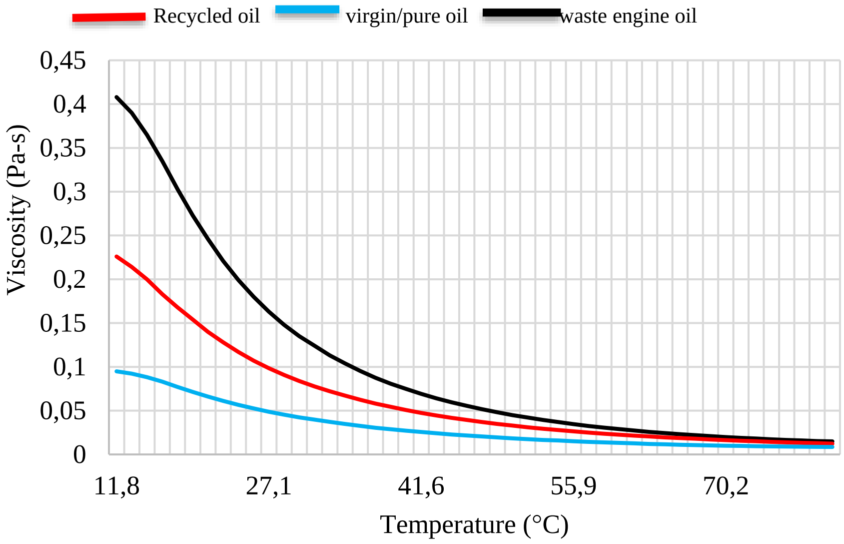


Fig. 11. Effect of temperature on viscosity of oil samples

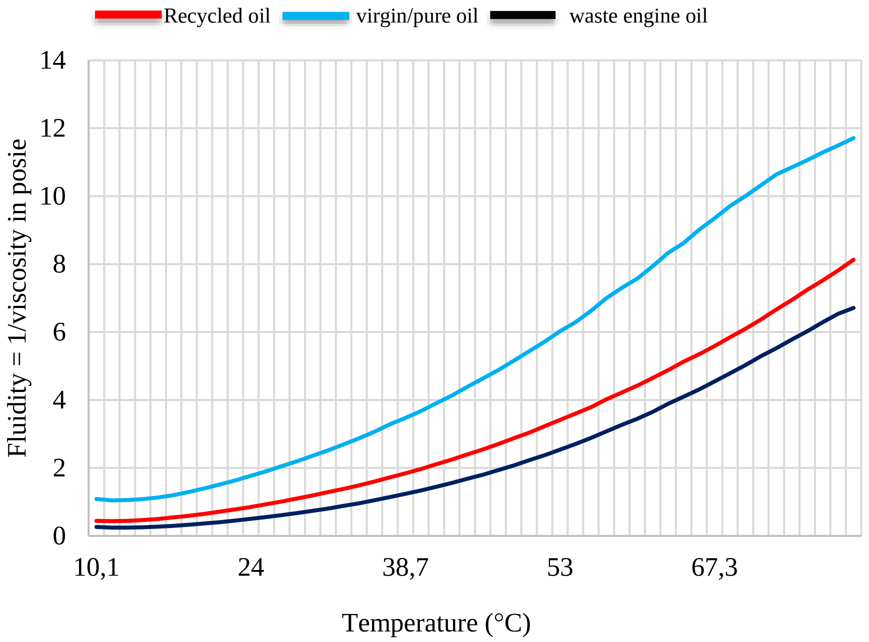


Fig. 12. Change in fluidity of oil samples with increase in temperature

4.3. UV Spectroscopy:

UV Spectroscopy is a low-cost, easy, and fast technique which is useful for identifying contaminants such as heavy metals. Many contaminants are chromophores and they absorb ultra-violet or visible light at specific wavelength. According to the Beer-Lambert law, the absorption spectrum produced by these samples at a given wavelength can be directly related to the concentration of the sample [29]. UV spectroscopy is a type of absorption spectroscopy in which light in the UV range (200-400 nm) is absorbed by molecules. Electromagnetic radiation is used in absorption spectroscopy between 190 and 800 nm and is divided into two regions. UV (190-400nm) and visible (400-800nm) region. The Beer-Lambert law, which is used in UV spectroscopy, asserts that the rate at which the intensity of a monochromatic ray decreases as a function of the thickness of the absorbing solution is proportional to the incident radiation. Moreover, the solution's concentration. Eq. (3) provides the Beer-Lambert Law's expression.

$$A = log\left(\frac{I_o}{I}\right) = ECL \tag{3}$$

where A is the absorbance, Io is the light intensity that strikes the sample cell, I is the light intensity that leaves the sample cell, C is the solute's molar concentration, L, the sample cell's length in centimeters, and E, its molar absorptivity.

The UV Spectroscopic readings of used lubricating oil and recycled oil are presented in Fig. 13. The used lubricating oil shows very small absorbance peak at around 400 nm which indicates presence of hydrocarbon chromophores with polycyclic aromatic hydrocarbons. A small peak at around 620-650 nm depicts presence of N=O in the used engine oil and its absence is seen in recycled oil after its removal. Presence of various heavy metals in used engine oil as wear particles and their absence in recycled oil is seen at specific spectral locations.

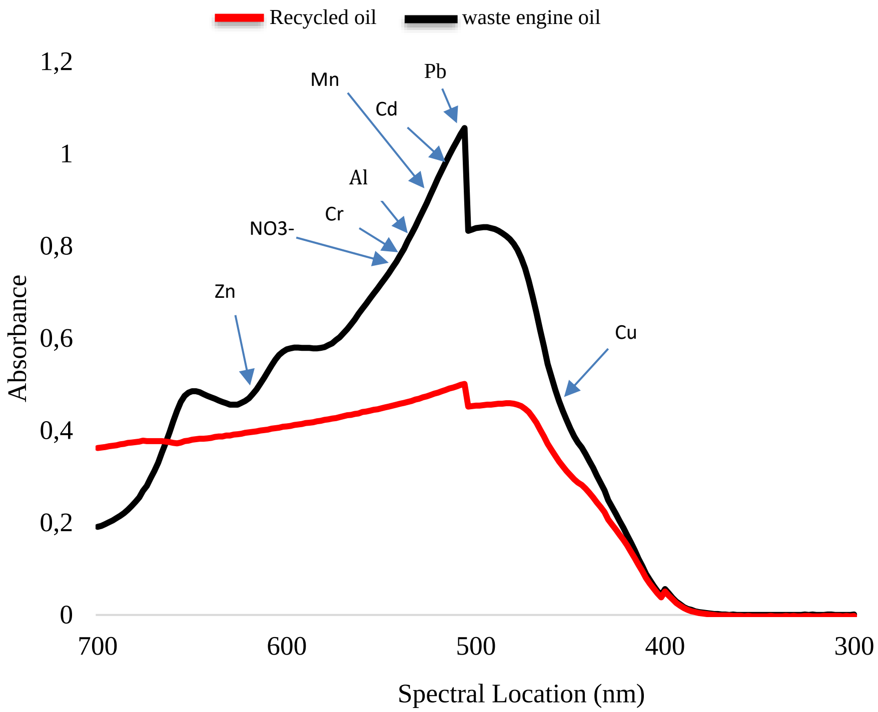


Fig. 13. Level of heavy metals in waste and recycled oil by UV Spectroscopy

4.4. Particle Size Analyzer

The AntonPaar Litesizer 500 was used to analyze the particle size distribution. This analysis is aimed to characterize nano and micro-particles in the waste and recycled oil samples.

Particle size analysis is a proven test method for oil analysis. The principle is simple: by measuring the number of particles in waste oil, the contamination ratio of the oil is known. That way, it helps to determine whether the oil is clean enough for reliable operation. Particle size analyzer test graphs in Figs. 14 and 15 clearly show decrease in the level of contaminants in the recycled oil when compared to that of used oil. It also shows the intensity of the contaminant particle size occurring in each sample.

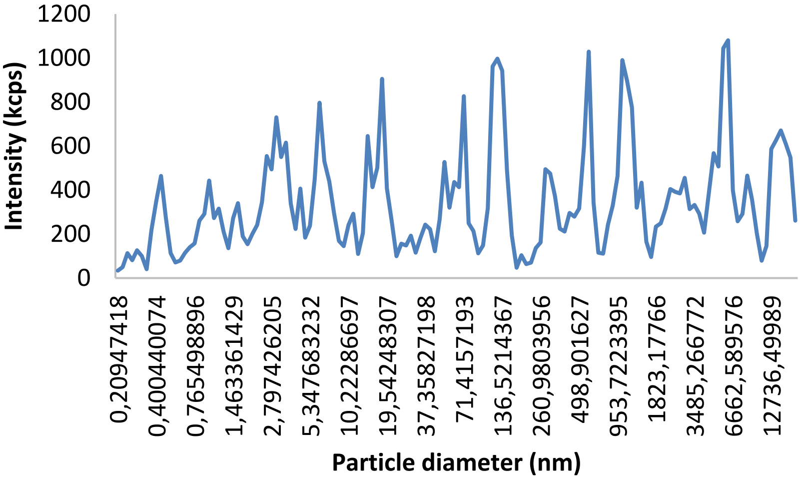


Fig. 14. Particle size distribution of Waste oil

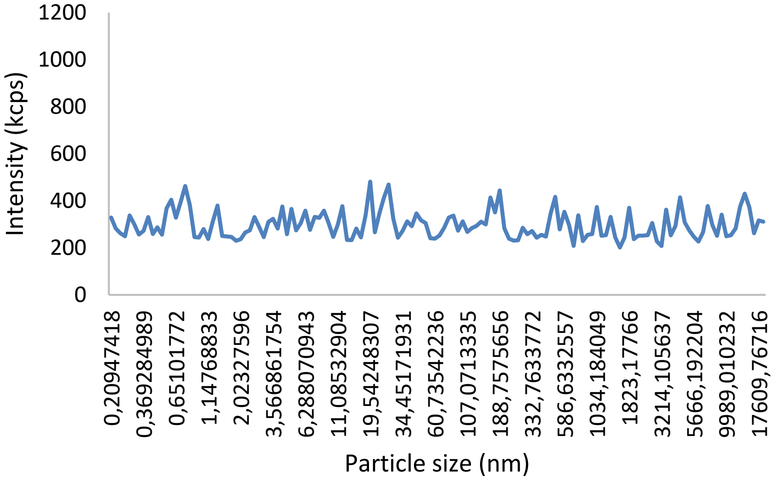


Fig. 15. Particle size distribution of Recycled oil

Modeling and Optimization Using GN Algorithm

The viscosity of liquids decreases exponentially with increase in temperature. It is observed that variation of viscosity with temperature in lubricating oils obeys Reynold’s equation given by equation (2):

$$\mu o = be^{-aT_A} \qquad (4)$$

where μo is the dynamic viscosity at atmospheric pressure and T_A is absolute temperature.

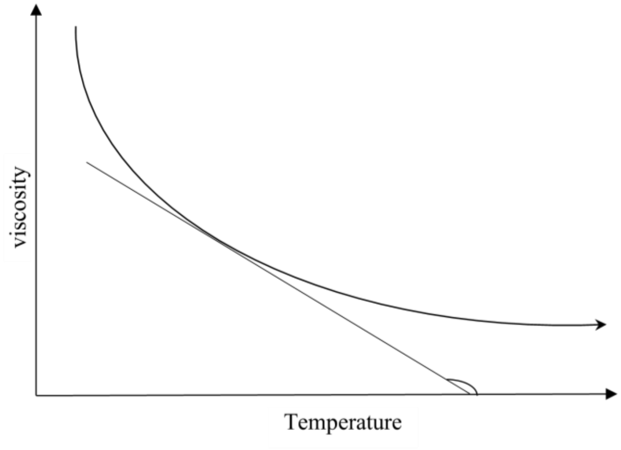


Fig. 16. Schematic representation of change of viscosity with temperature

To take non-linearity into consideration we assume,

$$-\frac{d\mu}{dT}\ \alpha\ T^{n} \tag{5}$$

$$-\frac{d\mu}{dT} = kT^{n} \tag{6}$$

Where k is the constant of proportionality

$$d\mu = -kT^{n}\, dT \tag{7}$$

On integration we get;

$$\mu = -\ \frac{kT^{n+1}}{n+1} + C \tag{8}$$

$$\mu = AT^{m} + C \tag{9}$$

where;

$$A = \frac{-\ k}{n+1} \tag{10}$$

$$m\ =\ n\ +\ 1 \tag{11}$$

Since the Eq. (9) is nonlinear and the constants cannot be determined by least square using logarithmic transformation. Eq. (9) describes the non-linear relationship between viscosity and temperature, which cannot be transformed into a linear form using a logarithmic transformation. Thus, the model parameters A, m, and C cannot be estimated using the least square technique. The Gauss-Newton technique was used to iteratively estimate the ideal values of A, m, and C. The three steps of the Gauss-Newton algorithm to estimate the model parameters are:

identifying Jacobian, Z_j calculating Forcing vector, D_j and assessing solution matrix, λ_{j+1},

where;

$$\lambda_{j+1} = \left[Z_j^T Z_j\right]^{-1} \left[Z_j^T\right]\left\{D_j\right\} \tag{12}$$

$$Z_j = \left\{\begin{matrix} \left(\frac{\partial\mu}{\partial A}\right)_1 & \left(\frac{\partial\mu}{\partial m}\right)_1 & \left(\frac{\partial\mu}{\partial C}\right)_1 \\ \left(\frac{\partial\mu}{\partial A}\right)_2 & \left(\frac{\partial\mu}{\partial m}\right)_1 & \left(\frac{\partial\mu}{\partial C}\right)_1 \\ \left(\frac{\partial\mu}{\partial A}\right)_3 & \left(\frac{\partial\mu}{\partial m}\right)_1 & \left(\frac{\partial\mu}{\partial C}\right)_1 \\ \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots.. \\ \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots.. \\ \left(\frac{\partial\mu}{\partial A}\right)_N & \left(\frac{\partial\mu}{\partial m}\right)_N & \left(\frac{\partial\mu}{\partial C}\right)_N \end{matrix}\right\}_{N\times 3} \tag{13}$$

$$Z_j = \left\{\begin{matrix} (T^m)_1 & (AT^m logT)_1 & 1 \\ (T^m)_2 & (AT^m logT)_1 & 1 \\ (T^m)_3 & (AT^m logT)_1 & 1 \\ \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots.. \\ \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots.. \\ (T^m)_N & (AT^m logT)_N & 1 \end{matrix}\right\}_{N\times 3} \tag{14}$$

$$D_j = \left\{\begin{matrix} (S)_1 - (\mu)_1 \\ (S)_2 - (\mu)_2 \\ (S)_3 - (\mu)_3 \\ \dots \quad\quad \dots \\ \dots \quad\quad \dots \\ (S)_N - (\mu)_N \end{matrix}\right\}_{N\times 1} \tag{15}$$

where $(S)_i$, $i = 1,2,3 \dots . N$ are the experimental viscosity values at various time intervals, and;

$$\lambda_{j+1} = \left\{\begin{matrix} A \\ m \\ C \end{matrix}\right\}_{3\times 1} \tag{16}$$

where λ_{j+1}, $j = 1,2,3, \dots\dots, m$ is the matrix containing model parameters. A, m and C were initially picked at random to evaluate λ_1. The optimal values of λ_{j+1} were obtained by successive iterations using

$$\lambda_{j+1} = \left[Z_j^T Z_j\right]^{-1} \left[Z_j^T\right]\left\{D_j\right\} \tag{17}$$

The data of waste/used engine oil along with virgin and recycled/filtered oil was collected from Anton paar rheometer used for experiment and the data was used for developing the models. The results obtained from proposed model are compared with experimental data and Reynold's model in this section. MATLAB was used for programming Gauss-Newton algorithm in order to evaluate the optimal values of A, m and C and were initialized by taking arbitrary values.

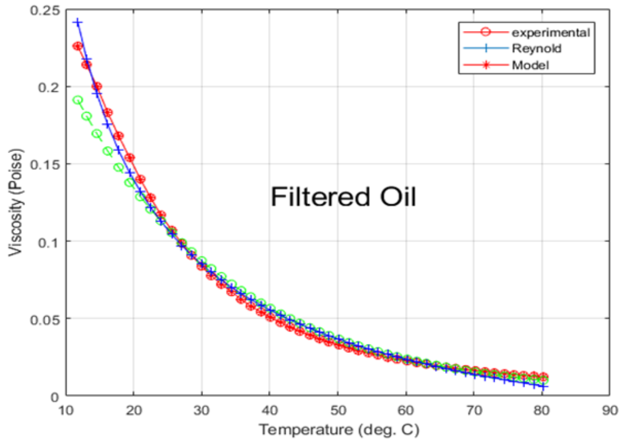


Fig. 17. Change of viscosity with temperature (Filtered/Recycled oil)

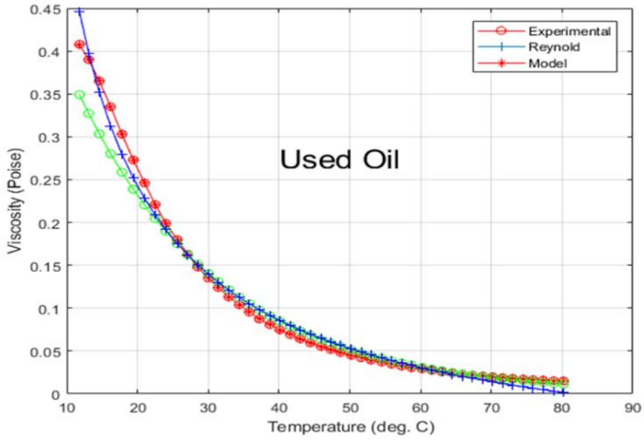


Fig. 18. Change of viscosity with temperature (Used oil)

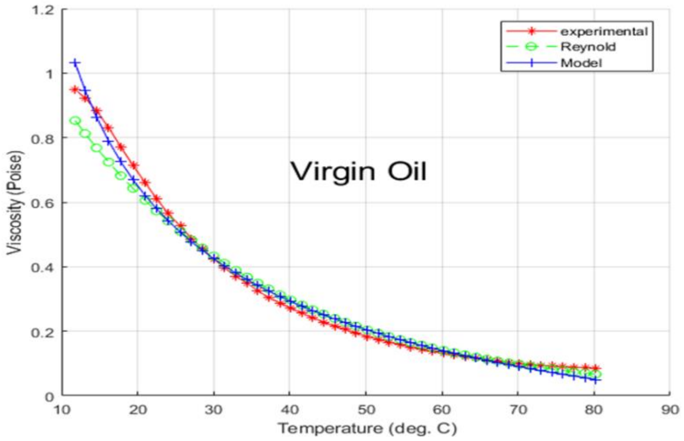


Fig. 19. Change of viscosity with temperature (Virgin oil)

Eqs. (18), (19), (20) are obtained for the results for viscosity of virgin, used and filtered/recycled oil.

$$\mu_{virgin} = -0.4620 + 5.9228T^{-0.5582} \tag{18}$$

$$\mu_{used} = -0.0973 + 4.8750T^{-0.8892} \tag{19}$$

$$\mu_{filtered} = -0.1254 + 4.1105T^{-0.7740} \tag{20}$$

Evaluation of competence of the models was estimated and presented in Table 3. From Table 3 it is obvious that in case of the proposed model there is a good correlation ($R^2 =$ 99.59% for virgin oil), ($R^2 =$ 99.44% for Used oil) and ($R^2 =$ 98.28% for Filtered/recycled oil) between viscosity and temperature. The results of proposed models indicate that ≥99 % of observed variation can be explained by model's inputs.

Table 3. Statistical parameters of Reynold's model and proposed model

	R^2		MSE	
	Reynold's	Proposed	Reynold's	Proposed
Virgin oil	98.92	99.59	1.60×10^{-03}	5.82×10^{-04}
Used oil	97.76	99.44	4.09×10^{-04}	1.24×10^{-04}
Filtered oil	98.28	98.28	1.08×10^{-04}	5.41×10^{-08}

Mean squared error (MSE) of proposed model and Reynold's model in Table 3 indicates that the error between the predicted and experimental values of viscosity is very small. Therefore, the model is in good agreement with the experimental results and can be used to predict the change viscosity of the engine oil with temperature with high accuracy during the operation of the vehicle.

6. Conclusion

Proper lubrication is essential for survival and maintenance of any machinery. It not only ensures smooth operation but also increases the life of the machine by reducing friction and wear. Thus, use of lubricating oil and changing it at right time is mandatory. Also, improper disposal of waste oil causes irreversible harm to the environment and living organisms, hence finding an alternate is a necessity.

The proposed process is a very promising method to recycle the waste oil and reclaim it for further use. In this research, pre-treatment process involving acid treatment, bleaching and neutralization is followed by ultrafiltration process by Polyacrylonitrile (PAN) membrane. Following conclusions are drawn from the investigation, and the results are very promising.

- The reduction in contaminant level was found to be >93% in moisture, >91% in soot, >97% in phenol, >92% in nitration, >85% in oxidation and viscosity, >86% in carboxylic acid, >81% in Total Base Number, >80% in sulfation, >82% in Aromatics and Polycyclic Aromatic hydrocarbons and >52% in ZDDP.
- There is evident change in color and odor due to removal of contaminants.
- There is marked decrease in viscosity (from 0.0408 Pa-s of waste oil to 0.226 Pa-s of recycled oil at 11.8°C) and considerable improvement in fluidity.

- Significant reduction in levels of heavy harmful metals such as aluminum, cadmium, chromium, copper, lead, manganese, zinc, nitrate as is observed by UV Spectroscopy. In addition to above, the study can also help to investigate the condition monitoring of engine parts. The methodology adopted is environment friendly with minimal impact on ecosystem and low-cost. The resultant product is of higher quality as the method exhibits high separation efficiency. The method can find in application for other types of waste oils obtained from industries and factories which include transmission fluid, refrigeration fluid, compressor oils, metal working oils, hydraulic fluid, etc. which can provide benefit on a larger scale globally.
- Particle size analyzer test graphs clearly show decrease in the contaminant level in recycled oil.

This research also develops a mathematical model to describe the rheological behavior of recycled motor oil. It is observed that the viscosity decreases with increase in temperature as it is known that oil thins-out at higher temperature, and the relation is non-linear. The lubricants display non-Newtonian shear thinning behavior. Lubricating oil is thick at low temperatures and becomes thinner as the temperature increases, thus decreasing the viscosity. It is pertinent to mention that the lubricating oil should not be too thick at low temperatures that makes it difficult to pump or pour, nor should it become too thin at high temperatures that its load bearing capacity is reduced as the temperature in the vehicle engine varies from ambient temperature to 150°C. Beyond certain temperatures, rheological properties remain unchanged showing good flow behavior. Different oils show different variation in viscosity with temperature. Presence of contaminants in oil increase the viscosity and thickness of the oil and it is observed that the viscosity of recycled oil is reduced considerably after removal of contaminants. Gauss-Newton method use is appropriate because the suggested model is non-linear. The model equations show that under specific circumstances (Shear stress, speed, torque, etc.), the viscosity of engine oil decreases exponentially with temperature. The following results have been extracted from the study:

- Coefficient of determination, R^2 is approx. 99% for the Proposed model and approx. 98% for Reynold’s model.
- The suggested model can predict the change in viscosity with temperature with an extremely high degree of accuracy, as demonstrated by Mean Squared Error.
- Additionally, there is relatively little difference in the anticipated and experimental viscosity values.

Abbreviations

$\emptyset$ is the Percentage reduction in the level of contaminants after recycling

R.O is the absorbance value of Recycled oil obtained from FTIR Spectroscopy

V.O is the absorbance value of Virgin/pure oil obtained from FTIR Spectroscopy

U.O is the absorbance value of Used oil obtained from FTIR Spectroscopy

μ_o is the dynamic viscosity at atmospheric pressure

T_A is absolute temperature

A is the absorbance

I_o the intensity of light incident upon the sample cell

I the intensity of light leaving the sample cell

C the molar concentration of the solute

L the length of the sample cell (cm)

E the molar absorptivity

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