Detailed Sensitive Detection of Impurities in Waste Engine Oils Using Laser Induced Breakdown Spectroscopy, Rotating Disk Electrode Optical Emission Spectroscopy and Surface Plasmon Resonance

Cherry Dhiman, Ayushi Paliwal, Mohd. Shahid Khan, M. N. Reddy, Vinay Gupta, Monika Tomar

Abstract—The laser based high resolution spectroscopic experimental techniques such as Laser Induced Breakdown Spectroscopy (LIBS), Rotating Disk Electrode Optical Emission spectroscopy (RDE-OES) and Surface Plasmon Resonance (SPR) have been used for the study of composition and degradation analysis of used engine oils. Engine oils are mainly composed of aliphatic and aromatics compounds and its soot contains hazardous components in the form of fine, coarse and ultrafine particles consisting of wear metal elements. Such coarse particulates matter (PM) and toxic elements are extremely dangerous for human health that can cause respiratory and genetic disorder in humans. The combustible soot from thermal power plants, industry, aircrafts, ships and vehicles can lead to the environmental and climate destabilization. It contributes towards global pollution for land, water, air and global warming for environment. The detection of such toxicants in the form of elemental analysis is a very serious issue for the waste material management of various organic, inorganic hydrocarbons and radioactive waste elements. In view of such important points, the current study on used engine oils was performed. The fundamental characterization of engine oils was conducted by measuring water content and kinematic viscosity test that proves the crude analysis of the degradation of used engine oils samples. The microscopic quantitative and qualitative analysis was presented by RDE-OES technique which confirms the presence of elemental impurities of Pb, Al, Cu, Si, Fe, Cr, Na and Ba lines for used waste engine oil samples in few ppm. The presence of such elemental impurities was confirmed by LIBS spectral analysis at various transition levels of atomic line. The recorded transition line of Pb confirms the maximum degradation which was found in used engine oil sample no. 3 and 4. Apart from the basic tests, the calculations for dielectric constants and refractive index of the engine oils were performed via SPR analysis.
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Keywords—Laser induced breakdown spectroscopy, rotating disk electrode optical emission spectroscopy, surface plasmon

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resonance, ICCD spectrometer, Nd:YAG laser, engine oil.

I. INTRODUCTION

THE sensitive detection of composition of waste engine oil as a renewable fuel is a subject of great concern due to issues related to environmental security and human health. Every year almost 24 million metric tons of waste engine oil is disposed all around the world [1]. Before disposal, the detection of elemental contamination is a potential tool to avoid the risks related to clean air environment protocol. The continuous monitoring stages of lubricants at many intervals are extremely essential in order to keep check on the physical, chemical, mechanical, and optical properties which provide the information about the operation of machinery parts at different levels of their utility and functioning. Waste engine oils consist of aliphatic and aromatic hydrocarbons which is a source of fuel energy. The most basic technique of pyrolysis has proven to be one of the best ways to clean the waste engine oil and made it as a source of fuel after recycling. The other fundamental techniques for the detection of impurities in engine oils are total acid number (TAN), total base number (TBN), blotter test and water content measurement [2].

Recent developments in the laser based experimental studies by various research groups and the experimental methods such as RDE-OES, SPR and LIBS have contributed tremendously for the critical studies for the detection of toxicants in few concentrations ranging from ppm to ppt traces [3]-[5]. Harith et al. presented the percentage decay of engine oils using LIBS technique by measuring the CN and C2 molecular stages collected from various cars with different mileage [6]. The basic engine oils properties were studied and mentioned by Sagi et al [7]. The elemental studies on wear metals in lubricants oils and fuel have also been reported by other authors [8]-[9].The unique features of RDE-OES and LIBS techniques comprise the detection of multi-elemental traces in concentration of few parts per million levels. For last several decades, LIBS has proven to be one of the best spectroscopic techniques for the standoff analysis of the samples for their elemental composition in trace concentrations levels. In LIBS, high power pulsed laser was used to excite the sample on which plasma was generated.

From LIBS plasma, the information about the spectral features of atomic transition lines can be studied and investigated. However, due to complexity in handling the LIBS spectral data, RDE-OES technique is more adaptable. Also, the comparatively faster detection accuracy in sub ppm levels using this technique makes it more reliable than other established methods for the degradation of waste engine oils. Contamination of engine oils contains the impurity elements due to wear and tear which changes its optical parameters such as refractive index and extinction coefficient. In such a direction, SPR technique is another more accurate and sensitive optical method in comparison to the various other detection methods such as Fourier transform infrared spectroscopy and electrochemical methods [10]-[12]. SPR technique relies on the excitation of surface plasmon wave at the metal-dielectric interface by the evanescent wave using various coupling arrangements. The resonance condition can be achieved when the propagation constant of SPW matches the propagation constant of the evanescent wave which is termed as surface plasmon resonance. SPR can be studied in angular interrogation and wavelength interrogation mode. In angular interrogation method, SPR plots consist of variation of the reflectance (i.e. normalized reflected intensity) versus incident angle which is further used to determine the dielectric constant (or refractive index) [13].

In the present research work, the contamination analysis was validated by other optical techniques such as RDE-OES, LIBS and the comparison of dielectric constants of different engine oil samples have been studied using SPR technique.

II. EXPERIMENTAL

A. Water Content Measurement

Presence of water content in the engine oils is one of the major problems that can result the damage of the machinery operations by forming small sized fragments of by-products and accelerate the process of oxidation. Due to this reason, the heavy machinery based engines for their longer working hours of operations require the monitoring for water check such as machinery in industrial sector, aircraft and diesel engine in railways. Hence, engine oil samples were collected to examine their water content status. The hot plate was used to check the water content and the presence of no crackling sound ensures the presence of no water content in any percentage in all four samples.

TABLE I
WATER CONTENT MEASUREMENTS IN ENGINE OIL BY CRACKLING SOUND ON HOT PLATE

S. No.	SAMPLE	Water Content
1.	Oil No.1	No
2.	Oil No. 2	No
3.	Oil No. 3	No
4.	Oil No. 4	No

B. Kinematic viscometric Studies

Another important method is kinematic viscometric which has been adopted by the engine oil monitoring laboratories as a routine work. In the viscometer studies, viscosity measurements were performed using kinematic viscometric bath. In this technique, engine oil was filled in a capillary tube up to the mark of the viscosity action of capillary tube and then it was kept in the viscometer bath and the time was measured so that the engine oil would perform the capillary action. 'T' time was taken by the capillary action multiplied by the tube constant gives the viscosity of the engine oil. The temperature of viscometer bath chamber was maintained at 40 °C and tube was placed for 40-45 minutes in the chamber. After removing the tube from the chamber, the time of capillary action (T) was recorded and it was multiplied with the capillary tube constant.

TABLE II
RESULTS OF KINEMATIC VISCOMETER BATH (JAYANTI SCIENTIFIC INSTRUMENTS)

S. No.	SAMPLE	Tube const.	T/factor (sec)	Viscosity
1.	Oil No.1	0.87	53	46.11
2.	Oil No. 2	1.23	112	137.76
3.	Oil No. 3	0.92	117	107.64
4.	Oil No. 4	0.87	17	14.79

C. RDE-OES

In RDE-OES technique, the engine oil was placed under the effects of high potential applied to the electrodes for plasma discharge. The discharge produces the collision energy interactions among the atoms and molecules which excite them to their higher excited states or transition levels. Thus, the atomic lines can be emitted as the photons from different energy level having different wavelength and detected by detectors with charged coupled array detector. The spectra were recorded by the instrument (Spectroil model: Oil-L, No. 6253/10 manufacturer for COFMOW).

D. LIBS

The LIBS experiment was set-up to investigate the elemental composition of the engine oil samples. The collected engine oil samples were placed for LIBS studies and the sample plate was firmly attached. The Nd:YAG Laser from Quantel Brilliant was used for designing the LIBS experiment on engine oils with Laser pulse width of 6ns. The Nd:YAG laser can be used on different laser harmonics such as 1064nm, 532nm and 355nm. For the LIBS experiment on engine oils wavelength 1064nm with pulse energy of 50mJ and repetition rate 1Hz was used. The higher repetition rate can ignite the fire; so to avoid the combustion of fuel, lower repetition rate of laser should be chosen. The intense laser pulses were focused on to the surface of engine oils kept in a patridish. The laser beam was focused by the optic arrangement called beam expander with the optics of 2 inch diameter fused silica convex lens of focal length 30 cm. After folding the laser beam by prism type optics at 90 degree, the intense laser beam pulses were focused by 1 inch diameter fused silica plano convex length of focal length 10 mm that makes the focal spot of size 72 μm and power density 2×10^{11} W/cm^2. The LIBS plasma can thus be collected from the surface of engine oils using lens integrated with a fiber optics

coupled to the ICCD spectrometer. The optical fiber with core diameter of 400 μm was used to collected signal with the spectrograph ANDOR coupled with the Integrated ICCD detector used by cherry et al on LIBS on grease [5].

The ICCD was synchronized with the laser control and the Q-switch pulses, i.e. the triggered output from laser, were fed to ICCD spectrograph. The spectrograph was used by employing the delay of 1 μs and gate width of 20 μs to record the plasma continuum emission consisting of atomic, elemental transitions from emission of single shot of Nd:YAG pulsed laser at 1 Hz. The average LIBS spectra signal of plasma emission from engine oils was used using grating of 1800 line/mm between 200-700 nm. The analysis of LIBS spectra was performed offline by using Plasus-specline software by matching the spectral results from the NIST database provided in software and other published data on the elemental analysis.

E. SPR

A schematic used for studying the optical properties of engine oils using SPR technique is shown in Fig. 2. The thermal evaporation technique was used to deposit thin gold film of thickness 40 nm on the surface of the right angled BK7 glass prism which was used for SPR studies for used engine oils. A specially designed liquid sample cell made of glass was prepared and attached to the prism by index matching liquid. The liquid sample cell was attached to the prism such that the Au thin film would remain in direct contact with liquid media for studying the degradation analysis of engine oils. SPR reflectance data were measured for prism/Au/oil sample systems for analyzing the contamination in the engine oil samples [14].
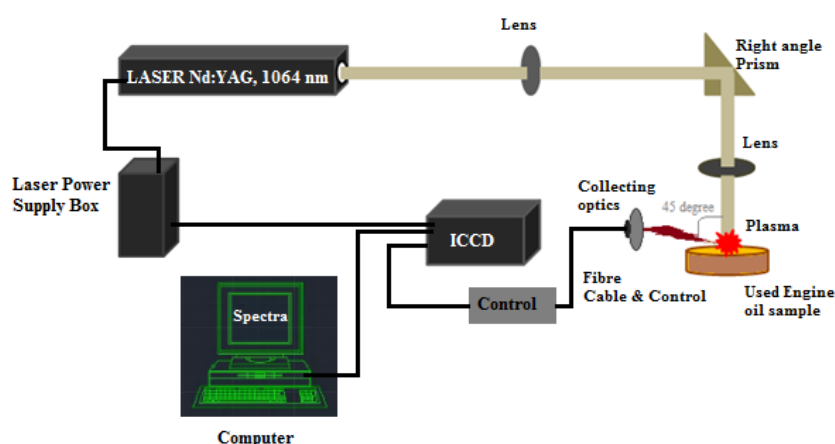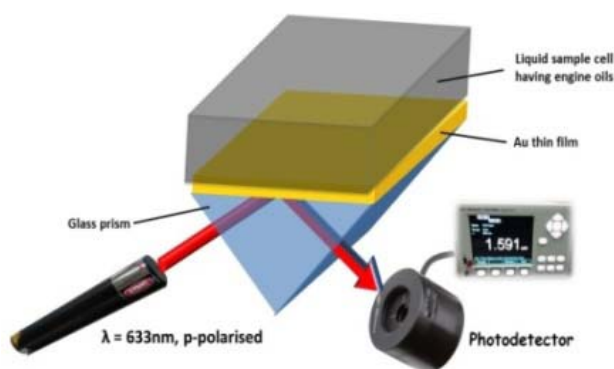

Fig. 1 Schematic of LIBS

Fig. 2 Schematic of SPR studies

TABLE III
ELEMENTAL COMPOSITION OF ENGINE OILS USING RDE-OES IN PPM

SAMPLE	Pb	Al	Cu	Si	Fe	Cr	Na	B
Oil No. 1	0	0	0	4	1	0	5	0
Oil No. 2	0	0	0	5	2	0	6	0
Oil No. 3	0	1	0	1	2	0	5	0
Oil No. 4	22	28	44	78	327	5	26	1

III. RESULTS AND DISCUSSION

According to water content analysis, it was found that there was no water content present in all four samples and the results are presented in Table I. Through water content measurements, the presence of no water ensures that the degradation in the waste engine oils would be because of the wear elements from the engine parts. The detailed kinematic viscometric bath calculations and the observed studies are presented in Table II which indicates that the viscosity of different waste engine oils with respect to the engine oil from different company type. The estimated quantification of impurity elements in the engine oils were identified using RDE-OES technique. On investigating the collected four samples, the presence of impurities elemental lines in parts per million (ppm) were obtained such as lead (Pb), Aluminum (Al), Copper (Cu), Silicon (Si), Iron (Fe), Chromium (Cr), Sodium (Na), and Barium (Ba). Firstly, elemental quantitative analysis of engine oil samples were analyzed by the RDE-OES and the presence of impurity elements are presented in Table III. RDE-OES results show that the engine oil sample no. 1 sample no. 2 and sample no.3 comprises the impurity elements such as Si, Fe and Na. Some traces of aluminum were found in sample no. 3 and sample no.4. Sample no. 4 was the most

degraded which consisted of impurity elements such as Pb, Cu, Cr and B. The presence of lead indicates the high degradation in engine oil sample no. 4. which can cause the chronic damage to the machinery parts and the results are presented in Table III.
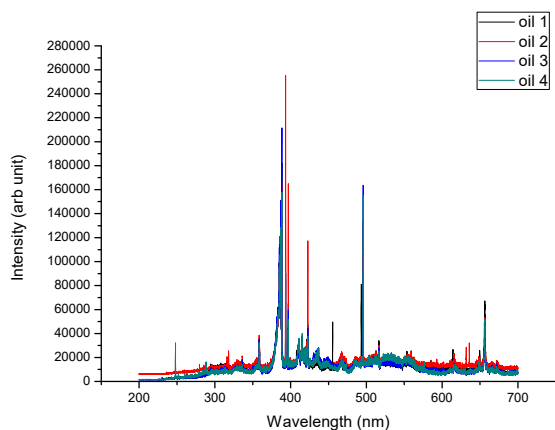

Fig. 3 LIBS spectrum of four engine oils

Similar elemental studies were cross examined by LIBS plasma based studies in which opaque plasma following the local thermal equilibrium condition consisting the atoms, ions and neutrals of all the impurity elements and the emission signals were captured by the spectrometer for the elemental detection. The LIBS spectrum shown in Fig. 3 was obtained by the plasma formation on the samples by the triggered Q-switched laser pulses and the emission signals measured by the ICCD spectrometer. The elemental impurity lines were further analyzed by plasus-specline software and the maximum intensity lines corresponding to various impurity elements showed that impurity elements. The impurities of Fe, Si, Al and Cu were found in the LIBS spectra of engine oil sample no. 1. The impurity lines such as Fe, Cr, Si, Al and Cu were found in engine oil samples such as engine oil sample no. 2. Some impurities of lead were found in engine oil sample no. 3 and 4 other than Fe, Si, Al and Cu. The estimated results from LIBS studies on engine oil samples are tabulated in Table IV.

For the reflectance plot measurement of SPR technique, wavelength $\lambda = 633$ nm was used for the excitation on samples in angular mode interrogation mode for prism/Au/air system. SPR measurements confirm the sensitive analysis of the degradation with the change in resonance position and the changes occur in the wave vector of resonance angle due to the change in the refractive index. The used engine oils samples consists various complex constituents such as contaminants in the form of additives and wear particles which are responsible of changing the optical properties of the Lubricants like color, transparency, refractive index and absorbance.

The comparative and detailed SPR based studies on engine oils was demonstrated by Aghayan et al. which was more sensitive and accurate as compared to other fundamental

techniques such as TAN, TBN, blotter test and water content measurement [15]. The absorption of surface plasmons due to the introduction of contaminants in engine oils leads to a noticeable change in resonance angle (θ_{SPR}) and reflectivity minimum (R_{min}) which is presented in Fig. 4. For the prism/Au/air system, reflectance (R) was found to decrease significantly and reach a minimum value of about $R_{min} \sim 0.66$ at an incident resonance angle, $\theta_{SPR} \sim 43.0^o$. Introduction of impurities in the engine oils causes change in color, transparency, and the absorption of light which leads to the change in the relative angular shift in the position of the minimum SPR. The SPR reflectance curve showed a shift towards higher angle on replacing the air media with the oil samples (i.e. prism/Au/oil sample system) and the minimum reflectance drastically increased to $R_{min} \sim 0.89$ at $\theta_{SPR} \sim 47.3^o$ for engine oil sample no. 1. On replacing the oil sample no. 1 with other samples, the reflectance minimum SPR dip was examined to be continuously shifting towards higher angles and the graphs are presented in Fig. 4. The maximum shift in R_{min} and θ_{SPR} was observed for engine oil sample no. 4 which shows the maximum contamination. The best fitted theoretical reflectance curves of SPR phenomenon are shown by solid lines in Fig. 4 using Fresnel's equations. The SPR reflectance data were fitted for prism/Au/air system and the value of dielectric constant for Au thin film at $\lambda = 633$ nm was estimated by fitting the SPR reflectance data which were found to be about $\varepsilon_1 = -12.2+i1.51$. The obtained value of complex dielectric constant of Au was close to the corresponding reported value (12.8+i2.7) [16]. The dielectric constant of Au thin film was kept constant and the SPR reflectance graphs for prism/Au/Oil sample system were fitted for the determination of dielectric constant and refractive index of engine oil samples.
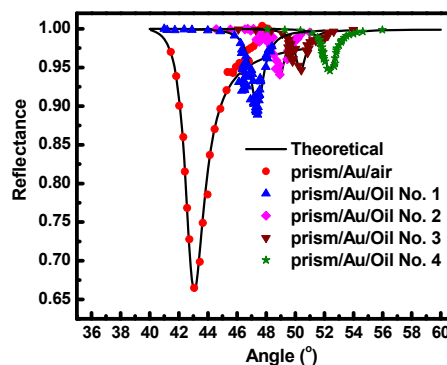

Fig. 4 SPR reflectance curves obtained for four engine oils in Kretschmann configuration

The dielectric constant and refractive index of engine oils were calculated from the recorded graphs of SPR on used engine oils samples. Further, Fresnel equations were used to fit the SPR data and the tabulated results have been presented in Table V. Table V shows that the SPR resonance angle (θ_{SPR}) is directly related to the real part of dielectric constant (ε') and hence refractive index (n) shows that FWHM of SPR

curve clearly affects the variation of imaginary part of dielectric constant (ε'') and hence extinction coefficient (k). In the present studies on engine oils, four used engine oils samples were studied and the maximum dielectric constant and refractive index were obtained for engine oil sample no. 4 as 1.929+0.031i.

TABLE IV (A)
ELEMENTAL IMPURITIES IN ENGINE OIL 1 FROM LIBS

Elements	Wavelength
Fe	248.007, 305.052, 311.077, 315.982, 323.513, 326.564, 336.564, 336.142, 337.88, 338.962, 358.505, 359.084, 376.966, 377.391, 380.48, 380.944, 382.18, 384.343, 385.192, 385.192, 385.579, 386.235, 387.201, 388.398, 391.449, 393.496, 394.809, 396.972, 400.023, 400.796, 407.902, 410.065, 411.108, 415.356, 416.824, 418.137, 419.798, 421.69, 422.81, 422.81, 434.358, 436.907, 439.727, 443.666, 444.361, 445.983, 446.64, 449.073, 455.523, 471.512, 473.637, 490.09, 493.527, 495.844, 507.006, 511.718, 516.584, 518.4, 518.4, 525.236, 528.248, 539.603, 553.661, 563.626, 566.677, 568.677, 568.029, 576.95, 585.486, 589.116, 601.437, 614.298, 622.563, 623.876, 649.791, 652.572, 656.434, 658.443, 664.043
Si	305.0072, 323.4517, 385.3665, 385.6018,386.2595, 418.335, 419.813, 444.4116, 518.525, 566.6677, 568.144, 614.2487, 623.9614, 652.6609,658.3707,664.0618
Al	358.6557, 384.2016, 388.4353, 394.8724, 400.9583, 416.8462, 448.987,489.891, 528.3733, 576.9137,585.376,622.619,358.6557,
Cu	315.8673, 323.5706, 338.0712, 339.0668, 359.2352, 377.2526, 380.5227, 382.0875, 386.046, 387.2768, 388.4131, 391.2491, 393.3268, 399.8369, 400.6165, 408.0509, 411.1296, 415.3623, 417.9512, 419.8656, 421.6912, 444.4831, 490.1427, 493.7221, 518.3367, 525.0524,539.7295, 553.505, 563.7155,614.2956, 622.3715,656.4501, 658.3458,664.1396,315.789,318.2172, 382.6921,384.9582, 386.046

TABLE IV (B)
ELEMENTAL IMPURITIES IN ENGINE OIL 2

Elements	Wavelength
Fe	248.007, 315.943, 318.067, 336.142, 358.505, 382.836, 385.115, 385.501, 386.158, 387.201, 388.359, 391.256, 393.496, 394.886, 396.972, 411.069, 414.429, 415.317, 416.824, 418.06, 419.72, 421.69, 422.81, 430.81, 430.38, 43.826, 436.946, 469.813, 473.65, 495.844, 512.838, 512.838, 516.584, 552.155, 559.223, 616.345, 631.909, 636.003, 643.998, 656.357
Cr	318.0286, 334.334, 344.144,347.297, 387.7963, 396.9743, 399.5788, 407.486, 435.1755, 463.1606, 480.5254, 567.5257,
Si	385.6018, 386.2595,
Al	387.0049,391.2362,394.8724,
Cu	387.2768,388.4131,391.2491, 393.3268, 394.6945,

TABLE IV (C)
ELEMENTAL IMPURITIES IN ENGINE OIL 3

Elements	Wavelength
Fe	247.968, 261.216, 297.829, 307.524, 336.104, 358.505, 374.61, 380.326, 382.218, 384.42, 385.154, 385.154, 385.501, 386.235, 387.201, 388.359, 391.449, 393.496, 394.886, 397.011, 410.953, 414.661, 415.317, 416.785, 417.326, 418.098, 419.798, 421.729, 422.81, 434.011, 436.444, 437.1, 438.104, 444.207, 449.343, 467.187, 469.852, 471.474, 473.753, 493.488, 495.844, 512.954, 516.507, 536.745, 541.302, 563.626, 610.397, 648.865, 656.319, 680.38
Si	385.6018, 386.2595, 419.813, 467.3256, 541.3099
Al	358.6557, 384.2328, 387.0049, 388.4353, 394.8724, 416.8462, 422.6816, 422.6816,610.3764
Pb	415.282,416.8033
Cu	297.8287, 374.539, 380.1556, 382.0875, 384.9582, 386.046,387.2768,388.4131, 391.2491, 393..268, 394.6945, 411.1296, 415.3623, 417.1851, 417.9512,419.8656, 421.6912,436.537, 444.0883, 467.1702,469.747,512.7702, 516.3251, 536.8383,563.7155,610.5746,656.4501

TABLE IV (D)
ELEMENTAL IMPURITIES IN ENGINE OIL 4

Elements	Wavelength
Fe	247.968, 251.753, 269.056, 272.107, 288.29, 295.358, 321.118, 358.505, 359.045, 364.839, 367.31, 378.356, 378.858, 382.682, 384.188, 385.154, 385.463, 386.235, 387.162, 388.359, 393.496, 394.886, 397.011, 400.1, 403.499, 408.25, 413.386, 414.622, 415.279, 416.862, 418.098, 419.682, 421.651, 422.81, 423.737, 425.591, 434.319, 434.667, 435.594, 435.98, 437.023, 442.739, 445.443, 446.061, 449.961, 452.24, 457.879, 467.264, 469.736, 471.474, 473.675, 483.562, 493.45, 495.844, 501.483, 503.337, 506.774, 510.945, 511.37, 512.761, 514.151, 516.584, 522.378, 539.217, 542.886, 554.597, 556.056, 565.055, 565.527, 568.531, 570.655, 576.371, 587.34, 602.711, 628.897, 656.396, 672.115, 680.457, 681.153, 691.156
Si	251.6112, 288.1577, 385.6018, 386.046, 386.2595, 403.5278, 419.813, 467.3256, 539.3146, 542.892, 691.1748
Al	272.0916, 384.2328, 387.0049, 388.4353, 388.4131, 394.8724, 403.4882, 408.4003, 416.8462, 422.6816, 423.7541, 434.7785, 435.5031, 467.1702, 570.4874
Cu	268.93, 272.1677, 288.4196, 288.1577, 384.9582, 393.3268, 394.6945, 408.0509, 415.3623, 417.9512, 419.8656, 421.6912, 425.5635, 435.4626, 469.747, 483.6799,501.6629, 503.4254, 506.7094, 510.8334, 511.1915, 512.7702,539.1656, 556.0573, 565.6629, 628.8696, 656.4501, 680.6216
Pb	415.282,416.8033,

TABLE V
COMPLEX DIELECTRIC CONSTANT AND REFRACTIVE INDEX VALUE OF ENGINE OILS

S. No.	Sample	Complex dielectric constant	Complex refractive index
1.	Oil No.1	1.805+0.01i	1.343+0.001i
2.	Oil No. 2	1.834+0.02i	1.354+0.002i
3.	Oil No. 3	1.889+0.03i	1.374+0.003i
4.	Oil No. 4	1.929+0.03i	1.388+0.003i

IV. CONCLUSION

The engine oil samples were tested and examined for the impurities to evaluate the degradation of the tested samples. The optical methods like RDE-OES and LIBS have been studied to characterize the engine oil samples in terms of the impurities composition and degradation feature. The results estimated from RDE-OES and LIBS methods were compared and it was found that out of the four used engine oils samples, engine oils sample number 4 was the highly degraded and composed of impurity elements. Using optical analysis of SPR measurements, engine oil sample no. 4 was found to have highest dielectric constant and refractive index resulting in maximum contamination. Thus the applications of three optical methods have been validated for the degradation analysis of engine oil samples.

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