Recent Advances in the Use of Sensors and Markers for Fuel Adulteration Detection: A Review

Gubihama Joel* and Linus N. Okoro

Department of Petroleum Chemistry, School of Arts and Sciences, American University of Nigeria, Yola, Adamawa State, Nigeria
**Corresponding Author*

***Abstract*:-Crude oil distillates (gasoline, kerosene, and diesel) and biofuels are vital energy sources that drive the manufacturing and automobile industry. Unfortunately, price disparity among these fuels induces marketers to adulterate for profit making. Fuel adulteration is a common practice in developing nations due to poor infrastructure and absent of strict laws. Fuel adulteration reduces engine performance, causes harmful greenhouse gases to be emitted through exhaust tailpipe of automobiles and contributes to global warming. These makes fuel adulteration an environmental threat hence the need to find innovative methods and techniques that will help in detection for easy prosecution of offenders. The use of sensors in modern chemistry has generated great interest due to their enhanced precision and accuracy. New policies on the use of markers for distinguishing different organic solvents has also aided in their identification when used as adulterants in fuels. Some organic compounds are also employed as markers to aid in the detection of impurities in fuels. There view will focus on recent research works that have utilized the use of sensors and makers for fuel adulteration detection.**

***Key words:* Refractive index, fuel adulteration, fiber optic grating, refractometer, wavelength, organic solvents**

I. INTRODUCTION

1.1 Fuel Adulteration

Fuel adulteration is defined as the act of blending petroleum/bio fuels or organic/inorganic solvents in little proportion with a large amount of desired commercial petroleum/bio fuel in order to increase the total volume of the desired commercial petroleum/bio for profit. Fuel adulteration is a criminal offence and strict embargoes set by law enforcement agencies have helped in the mitigation and control of this malpractice in most developed countries. However, fuel adulteration is on the rise in many developing nations like India, Brazil and Nigeria. In most developing nations where the government subsidizes kerosene due to high consumption and demand of the product by poor citizens, some marketers have taken advantage of this kind gesture by mixing the subsidized kerosene with high price diesel and petrol in order to boost their profits. Fuel Adulteration leads to knocks in automobile engines caused by wear and tear of pistons due to reduced lubrication and late ignition. Greenhouse gases are also released via tailpipe emissions from incomplete combustions of the adulterated fuel. These gases pollute the air and contribute to global warming. Various methods have been explored by law enforcement agencies, laboratories and researchers to check and identify adulterated fuel. This paper is going to review some of the sensors and markers that have been utilized to identify adulterant in fuels.

1.2 Sensors

A sensor is a device that measures a physical quantity and transmits it as signal to a device which observes and converts the signal to data for interpretation to meaningful information. The use of sensors has proven to be effective, time efficient and cost friendly in many industries. Several sensors have been utilized for detection of adulteration. This work will focus on sensors that have been explored in this decade (2011-2019). They include; optical sensor, photo detector, photonic crystal fiber, thermal imagning, sound/ultrasound, electrical metamatrerial sensors, high sensitivity float and microfluidic sensing device.

1.3 Markers

Some countries have adopted the use of some chemical markers in solvents. These markers identify solvents but do not impact on the chemical composition of the solvents. Even though identifying these solvents based on markers is sometimes difficult, it has evolved over time to become one of the favorite methods for identifying solvents that are used as adulterants in commercial fuels. This paper will review the use of extrinsic and quanizarin markers for fuel adulteration detection.

II. LITERATURE REVIEW

2.1 Sensor Based Approach

2.1.1. Optical Sensors

Optical sensors are small, multi-plexable lightweight, and immune to electromagnetic interference (EMI). They can function without both Electromagnetic Susceptibility (EMS) and electric current (at sensing point). These qualities give optical sensors a competitive advantage over other traditional methods of fuel adulteration detection.

Patil and Shaligram (2011) proposed a novel and enhanced sensor to detect adulteration of diesel with kerosene known as refractometric fiber optic sensor. Multimode plastic optical fiber is bent into S shape and uncladded to specific shape. It is then analyzed and simulated using a beam propagation RSOFT software. According to their study, the diesel covers the S shaped ration of the fiber when immerserd in diesel. The output intensity of the sensor is affected due to the change in refractive index causes by kerosene adulterant in diesel. They noticed an increase in sensitivity almost four times as the bend curvature of the fiber increses by two fold. Hence, they concluded that the method is indeed an efficient and highly effective method for adulteration detection in liquid fuels (Patil & Shaligram, 2011).

Etched Fiber Bragg Grating was used for petrol analysis to determine kerosene adulteration by Kumar *et al*(2012). According to their research, they observed that as the percentage of kerosene in petrol changes, a blue shift by the Bragg resonance wavelength occurs; such that the central FBG wavelength of 1550.30µm shifts by 0.05µm to 1550.35µm as the percentage of petrol increases by 20% as shown in figure 1. Kumar *et al* proved that the method is capable of detecting adulteration as low as 10% compared to other traditional ASTM testing parameters. They also confirmed its safety with inflammable fuels, sensitivity and portability for on-road measurements. This reiterates the suitability of the method in petrochemical and automotive industries(Kumar, et al., 2012).

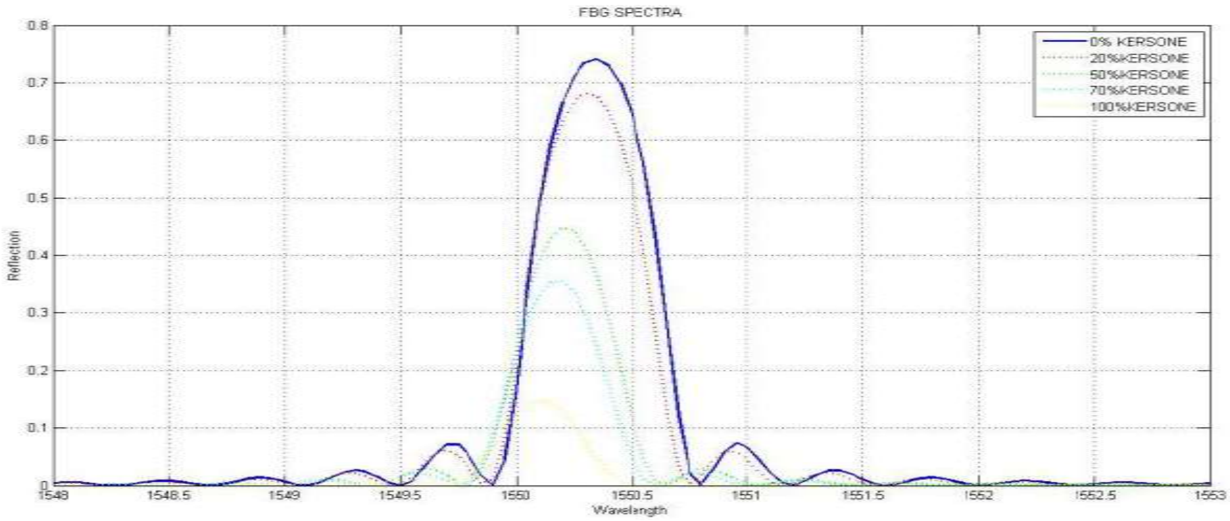

Figure 1: Reflectivity change of Fiber Bragg Grating at diverse adulteration percentages of kerosene in petrol. Sourced from(Kumar, et al., 2012)

Sadat (2014) employed an electronic technique to determine diesel adulteration by kerosene in which an LED light is guided through an optical fiber whose covering is removed over a tiny range so that the evanescent wave interacts with the measured sample (diesel). Figure 2 shows the variation of bulk absorbance of diesel-kerosene adulteration and the percentage adulteration with kerosene. According to their findings, the Conventional UV-VIS spectrophometer gave similar results which suggest that optical method is not only reliable and efficient but simple, compact and portable and could displace other traditional methods in the automobile and petrochemical industry (Sadat, 2014).

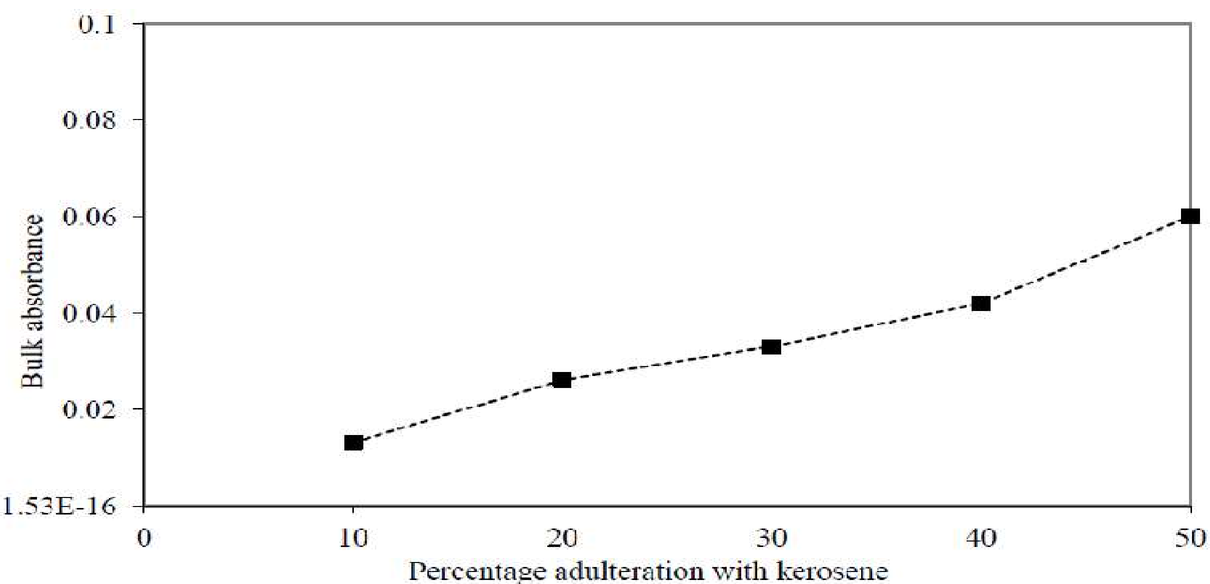

Figure 2: Variation of bulk absorbance of diesel with kerosene adulteration (Sadat, 2014)

Borne out of the desire for a fast and accurate fuel adulteration mechanism, Kude and Patil (2017) proposed a new method of detecting adulteration using optical fiber sensor (evanescent wave) bordered with peripheral interface controller (PIC) by means of refractive index. They formulated and tested the prototype which employed evanescent wave absorption technique to successfully identify percentage adulteration in diesel and petrol by kerosene. Their findings revealed a detectable capacity of 5% pollutant in diesel and petrol compared to traditional techniques with more than 10% detection capability (Kude & Patil, 2017).

Pathak *et al*(2017) experimented with acentrally controlled No-Core Fiber Sensor (NCFS) whose working principle depends on transitory wave's absorption occurrence for fast identification of kerosene in petrol. According to their experiment, The NCFS's sensing head is formed by hitting the NCFC between two multi-fibers which results in 390nW/% and 110nW/% sensitivity for high and low level adulteration. They exhibited the theoretic verification of confinement loss and intensity variation by finite element method. The result found indicated good repeatability and quick response time for the device while the experimental results correlated with the theoretical outcome. Pathak *et al* recommended this sensor for use in the automobile industry due to its size and its stress-free fabrication (Pathak, et al., 2017).

Verma*et al*(2018) explored a new approach for detecting adulteration of petrol and diesel by kerosene using a new fiber optic sensor based on Surface Plasmon Resonance SPR. The basis of their experiment rested on the principle that the refractive index of gasoline and diesel will vary linearly on addition of kerosene. Furthermore, a systematic detection of adulteration level was achieved based on the SPR in Krestchmann configuration. They found this method portable, highly sensitive, easy to construct and suitable for remote sensing. The device demonstrated immunity to electromagnetic interaction, a unique quality for the development of SPR based optical fiber sensors important for petrochemical studies (Verma, et al., 2018).

Kulkarni and Sujata(2019) proposed a simple, cheap, miniature and sensitive device based on fiber optic combined with optoelectric detection system. They utilized the method for identifying levels of kerosene adulteration in petrol as low as 5%. The results showed thatthe method is capable of detecting 1% variation in adulteration of kerosen in petrol (Kulkarni & Sujata, 2019).

2.1.1.1 Optical Refractometers

Patil and Shaligram (2011)presented a simple modulated fiber optic sensor that works on the guiding principle of refractive index variation. They utilized the sensor for determining kerosene concentration in diesel. The experiment set up comprises of two parallel fiber sensor probe (transmiting and receiving fiber) attached to a reflector sensor. The medium between the sensor probe and reflector is the adulterated diesel probe as shown in figure 3. Furthermore, The results shown in figure 4 revealed well defined seperated peaks in sequential order with adulteration interval of 10%. The tested fabricated prototype on different adulterated fuels was found to be simple, safe, compact and portable for use in-situ measurements. For easy understanding and comprehension of the results, a microcontroller was incorporated so as to automatically synchronize the final display (Patil & Shaligram, 2011).

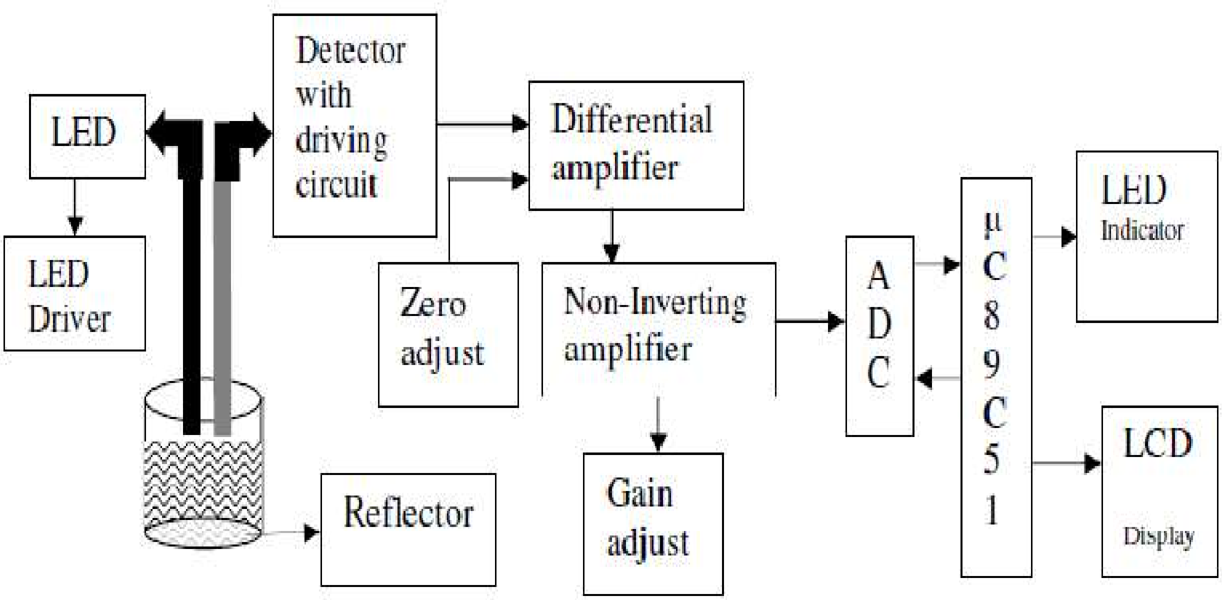

Figure 3: Block Diagram of Adulteration Detector Instrument (Patil & Shaligram, 2011)

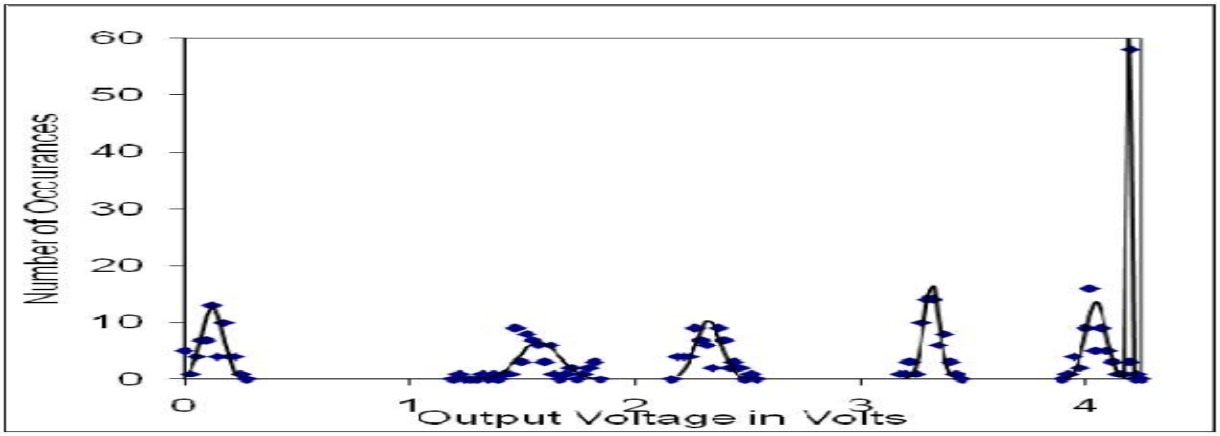

Figure 4: Repeatability measurement of adulteration fuel at 10% interval (Patil & Shaligram, 2011)

Kishor *et al* (2011) reported the design and development of optical time-domain reflectometer OTDR for adulteration detection in petrol. They discorvered that the optical sensor is an effective techniquefor determining the quantity of adulterants in petrol thoughthe method is fondly used indetectingfaultin optical sensors. The operation of the sensor was found to be easy and cost effective. These all round qualities makes this method very effective for adulterant determination in petrol (Kishor, et al., 2011).

For a long time, the concept of determining the purity of liquid substance using refractive index has been utilized in the petroleum, pharmaceutical and chemical industries. The refractive index has proven its significance in the identification of liquid impurities over the years. The temperature, wavelength of probe and pressure of liquid is used as a determining tool for refractive index of liquids. Kanyathare and Peiponen (2018) introduced a simple, reliable and inexpensive hand held refractometer to screen adulterated diesel oil. The hand held refractometer measures the refractive index and subsequent excess permittivity with the help ofa temperature correction table which enables estimation of refractive index at varying temperature. The refractive index is then used to determine the true and ideal permittivity of diesel. According to Kanyathare and Peiponen, it is possible to screen permittivity values based on the value of excess permittivity(difference between the values of true and ideal permittivity).In order to validate the results, the values found in Tanzania using the hand held refractometer was compared with the values found in Finland from laboratory tests conducted using the precise Abbe refractometer and hand held refractometer. Since the method could successfully detect adulteration of diesel by kerosene as low as 5%,it could be said that the technique is an effective screening mechanism for adulterated diesel (Kanyathare & Peiponen, 2018).

Kanyanthare*et al*(2018) in a research, worked with an optical sensor with the aim of screening adulterated diesel oil. Figure 5 shows a schematic diagram of the working principle for the device. According to the study, the device differentiates the mismatch between the refractive index of the fuel sample and the glass. The device also studied the interaction of film from fuel with the surface. The experimental results showed that the highest mismatch was between the glass and diesel oil while the lowest was noticed amid the glass and 15% adulterated diesel oil. At inception, they used an Abbe table refractometer to measure the refractive index of each fuel sample at room temperature for easy comparison. They also conducted a sensitivity test of the prototype using samples of diesel adulteration with kerosene at low concentration (Kayanthare, et al., 2018).

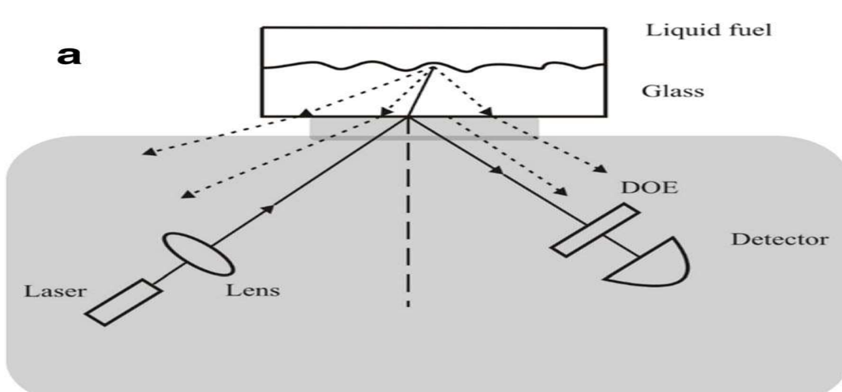

Figure 5: Schematic diagram showing a handheld sensor used for fake diesel oilscreening. Sourced from (Kayanthare, et al., 2018)

Kayanthare*et al*(2018) employed an Anne Refractometer to measure the refractive indices of binary fuel mixtures. Initially, the transmittance spectrum was measured in the visible and near-infrared NIR range. This was necessary for the determination of excess permittivity of binary mixtures. Excess permittivity is paramount in data interpretation of liquid interaction especially in binary liquid mixture. Relatively low adulteration of 5 to 10% was distinguished from 15% adulteration by means of imaginary optical properties. Their study is a solid prove that the device combined with extinction coefficient can screen, discriminate and separate adulterants from kerosene and diesel fuels under field conditions(Kayanthare, et al., 2018).

2.1.1.2 Optical waveguideSensors

Dutta *et al* (2013) fabricated an evanescent optical waveguide sensor to detect adulterant in petrol. The sensor is composed of silica-silicon wafer as core layer, silicon oxynitride and planar waveguide geometry as shown in figure 6. Out of the various technologies used is the Plasma Enhanced Chemical Vapor Technology. According to the results, the proposed sensor possesses 40 times more and 20 times more sensitivity than the existing planar waveguide and asymmetric waveguide structure respectively. Figure 7 shows the adulterant concentration in petrol decided by the measured normalized power against kerosene concentration (petrol-kerosene and diesel-kerosene). According to Dutta *et al* the sensor is now universally accepted due to its easy fabrication, independent polarization and high sensitivity (Dutta, et al., 2013).

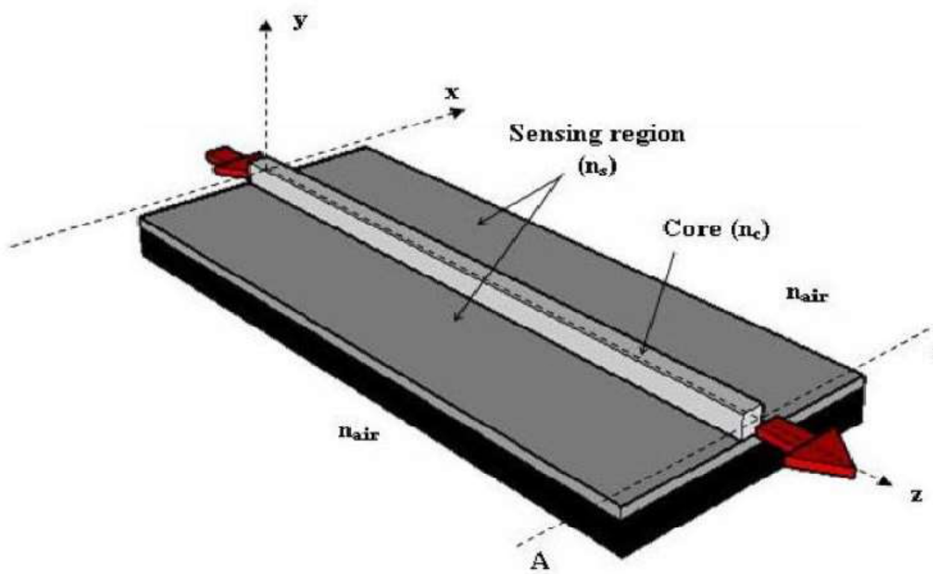

Figure 6: The sensing region of the planar waveguide placed on the top of the silicon substrate in 3D(Dutta, et al., 2013)

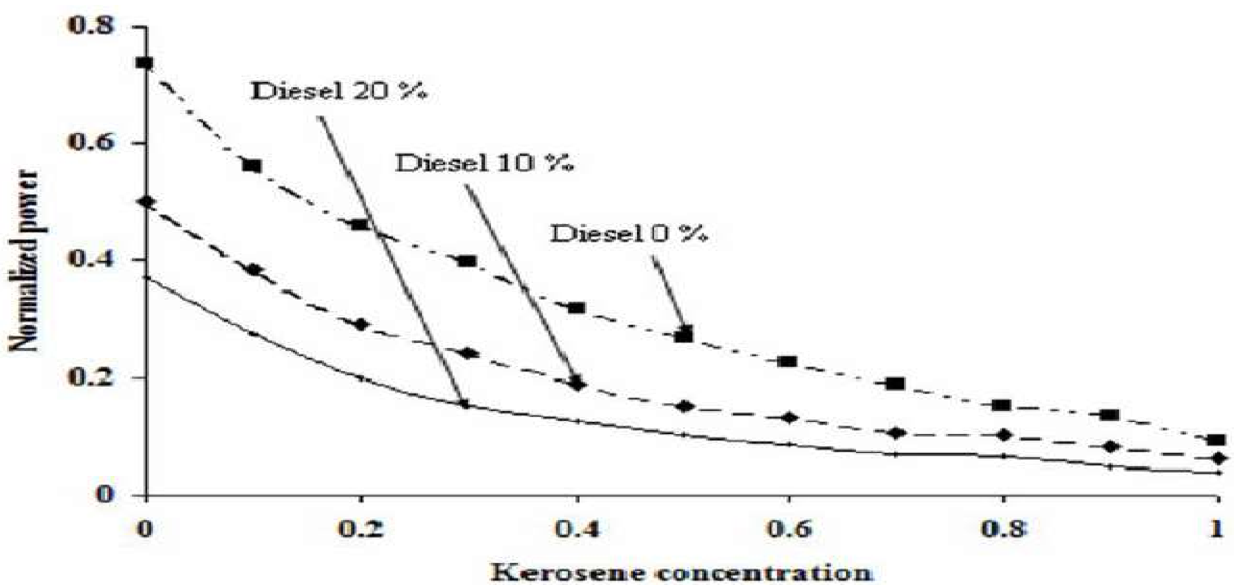

Figure 7: Normalized power against kerosene concentration(Dutta, et al., 2013)

Dutta *et al* (2013), in another research developed a similar evanescent waveguide optical sensor with similar properties as the former to detect adulterants in petroleum products. A waveguide of width 50µm and length 10,000µm developed using SiON technology was embedded on the sensor so as to enhance its sensitivity. A thin cladding layer with refractive index that is smaller than that of the core was used to support the wave guiding film. According to Dutta *et al*, spot determination of adulterant concentration and independent polarization without chemical analysis was used in order to achieve speedy and time saving technique. (Dutta, et al., 2013).

Dutta *et al* (2016) used a waveguide sensor which functions on the principle of Frustrated Total Internal Reflection (FTIR) to detect adulterated products and to confirm their versatility. They found great similarity between the values produced by developed sensor and the resulting predictions from the measured sensitivity. They found this method to be better in performance due to its high sensitivity at very short time requirement.(Dutta & Sahu, 2016).

In a study conducted by Yadav *et al* (2019), a fabricated metal clad planar waveguide with large electromagnetic radiation film confinement and a hollow prism was used to analyze petrol and diesel for adulteration. A linear refractive index variation and percentage change in concentration of kerosene was observed. The estimated theoretical maximum sensitivity at 105.88 degree/RIU (petrol) and 190.00degree/RIU (diesel)agreed with respective maximum sensitivity 105.24degree/RIU and 182.68 degree/RIU estimated with the proposed sensor. According to their study, the stability of the approach was also high (Yadav, et al., 2019).

2.1.2 Photo Detector Sensor

Felix *et al* (2015) measured the amount of product formed after heating the adulterated fuels using a photo detector at the end of an infra-red light source. The process worked on the principle of appearance and disappearance of liquid between the IR receiver and the IR transmitter. The microcontroller detects the percentage adulteration by comparing the values of output voltage to the level of liquid remaining. Level detection using image processing technique was employed where a camera lens captured images (320*240 pixel resolutions) for Raspberry pi to process. Captured Images of liquid level before and after heat was applied were compared. The amount of adulterated liquid found was converted to percentage and displayed. They concluded that the image processing technique was an accurate method (Felix, et al., 2015).

2.1.3 Photonic Crystal Fiber Sensor

Moutusi *et al* (2019) proposed a dual core Photonic Crystal Fiber for petrol adulteration analysis. Finite element method was used to numerically study the sensing probe. The sensing probe which has a single analyte channel adopted finite element method for the numeric study. Two solid light guiding cores are situated at the center of the fiber where the gasoline sample is injected. They investigated the sensitivity as well as the mode coupling using varying level of gasoline adulteration. The results for the simulation indicate extreme sensitivity marked at 20,161.2nm/RIU for small probes. The study concludes with the concept that the device can be used to manufacture a portable adulteration detection sensor to study environmental pollution (De, et al., 2019).

2.1.4 Thermal Imagning and GLCM Sensing Device

Ganesan and Somasunsaram (2019) combined thermal image processing with Gray Level Co-occurrence Matrix (GLCM) algorithm to study fuel adulterantion. The thermal camera comprising optical lens and infrared emitter emits infrared light to the object (fuel mixture) for the detector to produce image with temperature details known as thermo gram. According their study, signal processing element process the electrical pulses converted by the thermogram which then translate to image data. Theyutilized the GLCM features to determine the effective region from the image extracted which showed how the adulteration was been spread. Experimental results indicated 98% accuracy for adulteration detection level at 5 and 10% in petrol(Ganesan & Somasunsaram, 2019).

2.1.5 Sound/Ultrasound Sensor

Gupta and Sharma (2010) carried out an investigation to determine how the adulteration of petrol and diesel with kerosene will affect the sound speed. They employed the Pulse-Echo (PE) method which works on the guiding principle of converting acoustic energy emitted and received by the transmitter to electrical energy. According to their findings, the time delay T_D between the received and the transmitted energy pulse is equivalent to the speed of sound in both fuels. The second method which is the continuous wave method shows that the transmitter emits acoustic vibrations which are excited and treated to recuperate the time delay T_D. The results they found indicated how feasible the speed of sound is in detection and study of quantity of adulterants in automobile fuels. They concluded that sound analyzer usedis feasible with little modification in the study of fuel adulteration(Gupta & Sharma, 2010).

In order to evaluate the purity of liquids, Tomar & Tomar (2011) used ultrasonic grating in combination with physical optics to diagnose adulteration in liquid mixtures. Density, concentration or refractive index determines the diffracted dots. The experimental setup consisted of ultrasonic cell comprising thick, cubic shaped optical flat glass walls. The dot seperation of the solution with 100% concentration of kerosene and ethyl glycol were quantified to be 0.0020m and 0.00215m respectively. They found its sensitivity adequate enough to identify adulterants in little amount using the variation in succesive diffracted dots which is determined by a Position Sensitive Detector PSD. The method was confirmed to be quick and economical for fuel adulteration detection(Tomar & Tomar, 2011).

Using a slightly different approach, Daingade et al (2018) were able to distinguish between adulterants in fuel samples using ultrasonic sensor which measures distances by emitting and receiving sound waves at specific frequencies. The ultrasonic sensor detects adulteration at 90-95% accuracy which could be the perfect solution for the automobile industry (Daingade, et al., 2018).

2.1.6 Electrical Metamaterial Sensing Device

Rawat et al (2016) proposed the use of a complementary split ring resonator CSRR [2.47 GHz (ISM band)] that functions on the Babinet's principle of electrical metamaterial to detect kerosene adulteration in petrol. As seen in the figure below, the sensor displayed the S21 response for 10, 20 and 30 percent kerosene in petrol, an indication that it is possible to detect adulteration at 10% minimum (Rawat, et al., 2016). The sensor has great potential in the automobile industry due to its cheap fabrication, good repeatability and rapid recovery. It also provides extreme miniaturization. Thus it can be implemented not only at gas stations, but also directly in automobile engines.

With the goal of developing an alternative approach to adulteration detection methods, Bakir *et al* (2019) proposed aninnovativesensor called metamaterial application with the aim of operating in the X band. A new sample containeris placed just behind the structure for easy operation. By having a wider bandwidth and higher quality factors, it is expected that the proposed sensor will exceed other similar sensors with resolution and sensitivity. further validation ofthe method was undertaken by first measuring the dielectric constant using Agilent 85070E dielectric measurement kit then simulating the studies and finally experimenting test results with sensor layer. The results obtained revealed excellent adaptability of the method for different electrochemical sensing applications on condition of changing dielectric constant from 2 to 6 (Bakir , et al., 2019).

2.1.7 Micro-Fluidic Sensing Device

A micro-fluidic device using a Micro-electromechanical systems (MEMS) technique was developed by Goel *et al* (2013). The device which uses MEMS technique is tested on automobile fuels with known adulterants and various blending ratios of bio-fuels with conventional fuels under suitable viscosity ranges. The geometric design of the sensor is shown in figure 8. Based on its dependence on viscosity and density, the method can also be used to check adulteration in edible consumer goods and to sense hemoglobin in blood. It is efficient in real-time detection of bio-fuel blend ratio and adulterated automobile fuels and has garnered interest particularly in the automobile industry. According to them, development of a prototype that will be integrated with existing microcontroller of automobile for real time testing of fuels is in advance stage(Goel, et al., 2013).

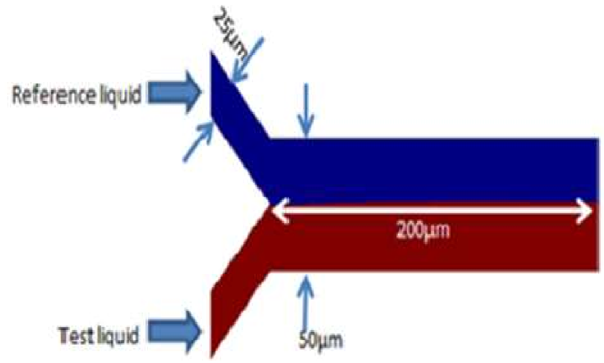

Figure 8: Y shaped microfluidic device geometry design (Goel, et al., 2013)

Microfluidics combined with 3D printing was explored by Patil and Awasthi (2017) in their pursuit to differentiate adulterants in liquid mixtures. The technology comes with a lot of promise as seen by increasing patronage in households and industrial applications. Patil and Awasthi further worked with various forms of 3D optical microfluidic devices to measure adulterants present in biofuels and milk based on the variation in their viscosity. The study has proven the device's capability to checkmate adulteration in foods and machine fuels by corrupt marketers (Patel & Awasthi, 2017)

Viscometers developed in the past lack reproducibility in measurements and has a maximum limit of detection which limits its versatility in the diagnostics and medical field. Borne out of the desire to develop a lab-on-a-chip platform for viscosity test, Sankaran *et al* (2017) validated the effectiveness of a 3D printed micro viscometer in measurement of adulteration of automobile fuels. They further designed the micro viscometer using the stereolitho graphic SLA based Digital Light Processing DLP 3D printer so as to analyze the variation in dynamic viscosity of petrol, kerosene and diesel. The results revealed an accuracy of 0.95 which proved the device's ease of operation, automation capabilities and versatility in applications where viscosity is the monitoring parameter (Sankaran, et al., 2017).

2.2 Marker Based Approach

2.2.1 Extrinsic Marker

Selai *et al* (2014) invented a new precise and cheap method for checking adulteration in aviation fuel, gasoline and diesel (Selai, et al., 2014). The method employs gas chromatography to detect an extrinsic marker and determines its concentration in adulterated gasoline and diesel fuels without the use of mass spectroscopy. The extrinsic markers used primarily have organo sulfur molecule(s) bearing S-S bond, C-S bond or both.

2.2.2 Quanizarin Marker

For the purposes of quality control, Haloulos *et al* (2016) embarked on an investigation to quantify mass concentrations of quanizarin in 97 samples of unleaded petrol. Quanizarin is a marker used for easy identification of octane number. In the study, they obtained samples from retail stations located in different regions of Greece and analyzed them. According to their statistical analysis results, the concentration of quanizarin fell below 3mg L^{-1} specification limit and hence a significant difference was observed between samples of different geographic regions. They attributed this to the way fuel supply chain is handled and coordinated in Greece(Haloulos, et al., 2016).

III. SUMMARY, CONCLUSION AND RECOMMENDATION

This paper has successfully reviewed research works that have employed optical sensors which incorporates fiber optic grating and refractometers to measure the change in refractive indices of adulterated and non-adulterated fuels. The use of sound ultrasound sensors, microfluidic sensors, and electrical metamaterial sensors were also critically reviewed. Furthermore, we studied the importance of extrinsic markers and quanizarin markers using mass spectroscopy and chromatography as separation techniques in the detection of fuel adulteration. Despite the extensive work reported, research is still ongoing on how to improve sensor accuracy and sensitivity. Further research is also ongoing to incorporate sensors into automobile and machines for quick detection of fuel adulteration. Further research should focus on the use of nano-sensors and nano markers for improved optimization and efficiency.

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