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A prototype of an optical sensor for the identification of diesel oil adulterated by kerosene

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Abstract

Background: Liquid fuel adulteration has several far-reaching impacts such as environmental pollution. A widespread practice is typically the adulteration of diesel oil by kerosene. The relatively cheap price of kerosene is probably the most important reason for its usage in illegal adulteration. Herein, we demonstrate the use of a prototype optical sensor for efficient tracking of adulterated diesel oil.

Methods: In this study, a prototype of an optical sensor for screening of fake diesel oil is proposed. The device exploits the phenomenon of laser light reflection from a fuel film over a roughened glass plate. The sensing mechanism of the device is based on the refractive index mismatch between the glass and the fuel sample, and the wetting property of the fuel film over the roughened surface. For the sake of comparison, the refractive index for each of the fuel samples was measured at room temperature with the aid of an automatic temperature controlled Abbe table refractometer. The sensitivity of this prototype optical sensor was tested using training sets of diesel oil samples adulterated with low concentrations of kerosene.

Results: Originally, a commercial handheld glossmeter, with a new innovation of a removable sensor head for liquid inspection is presented as a prototype sensor for the screening of possible adulteration of diesel oils with kerosene. The significant difference in the signal readings obtained from carefully prepared training sets of adulterated diesel oil composed of low percentages (5–15%) of kerosene has proven the high sensitivity of the developed sensor.

Conclusions: The ability to detect low concentrations of kerosene in diesel using the newly developed hand-held prototype sensor proves its high sensitivity compared to a high-accuracy Abbe refractometer. We envisage that this proposed sensor could, in the future, be made accessible to the authorities as a mobile fake fuel measurement unit.

Keywords: Diesel oil adulteration, Refractive index, Light reflection, Immersion liquid, Contact angle

Background

Monitoring the quality of liquid fuels is important not only during the production of fuels in refineries, fuel transportation and storage, but also in issues regarding the control of authenticity of fuels by regulating authorities. Liquid fuel adulteration, for example, mixing of diesel oil by kerosene has several far-reaching impacts such as environmental pollution. Optical spectroscopy is a useful tool for testing the quality of liquid fuels, and typically it is based on spectral fingerprints identification

of organic molecules in the infrared spectral range [1, 2]. Usually, one can measure either transmission or reflection spectrum from liquid samples. The basic idea of the optical spectra measurement is that molecules absorb the incident electromagnetic radiation, and the detected absorption bands are used for the identification and concentration studies of materials, such as liquid fuel. Since the purity of the fuel product is an important issue, the development of biodiesel and bioethanol products has stimulated research on the study of fuel purity (see for example [3–5]), which is also the issue in case of adulterated diesel oils. The trend regarding field measurements of liquids, including fuels, is to develop portable sensors that are based on the exploitation of

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spectroscopic techniques [6, 7]. In principle, this can be realized, in some cases, with the aid of modern technology that is based on mobile telephones [8]. In the past, we have contributed to the development of various types of spectral measurements techniques for detecting the purity of transparent and turbid liquids that are present in process industry [9], and liquid products, such as red wine [10, 11].

The absorption properties of liquid fuels are well-known in the near infrared (NIR) spectral range and are typically measured using a spectrophotometer by scanning the wavelength of the probe radiation. Probably the most popular method is based on recording of the transmission spectrum from the fuel sample with a spectrophotometer. Unfortunately, things become more problematic and challenging in an attempt to identify different grades of diesel and kerosene due to their similar spectral features in the NIR region. Therefore, it is usually quite difficult or even impossible to differentiate these fuels from each other using only the simple transmittance data. The challenges involved in the use of NIR in tracking fuel adulteration is intensified in a typical practice where diesel oil is adulterated with about 10–30% of kerosene. Adulteration of fuels is a serious problem [12], therefore recently developed spectral measurement and analytical methods have been suggested for the screening of adulterated liquid fuels [13–15]. The relatively cheap price of kerosene is probably the most important reason for its usage in illegal adulteration. Diesel adulteration can be realised by mixing small amount of different substances such as, small amounts of distillate fuels, gasoline boiling range hydrocarbons, waste industrial solvents such as industrial used lubricants, small amounts of heavier fuel oils, kerosene etc. to diesel oil. The adulteration of diesel oils is not a problem for only developing countries but a global challenge [16] due to its significant contribution to global warming. However, it is worth mentioning that the practice is relatively rare in developed countries compared to developing countries. Also petrol/gasoline is adulterated, but typically it is much easier to screen adulterated petrol than diesel oil by optical measurement techniques, such as optical spectra.

Perpetrators of fake fuels are so smart that, once a screening method is developed, they change the concept of preparing a fake fuel to avoid screening by, for example, using lower kerosene concentrations during the adulteration processes. Hence, in this study we have used adulterated samples with relatively low percentage of kerosene, namely 5%, 10% and 15%. The ability to detect these low concentrations of kerosene in diesel using our method proves its relatively high sensitivity over the conventional spectroscopic methods.

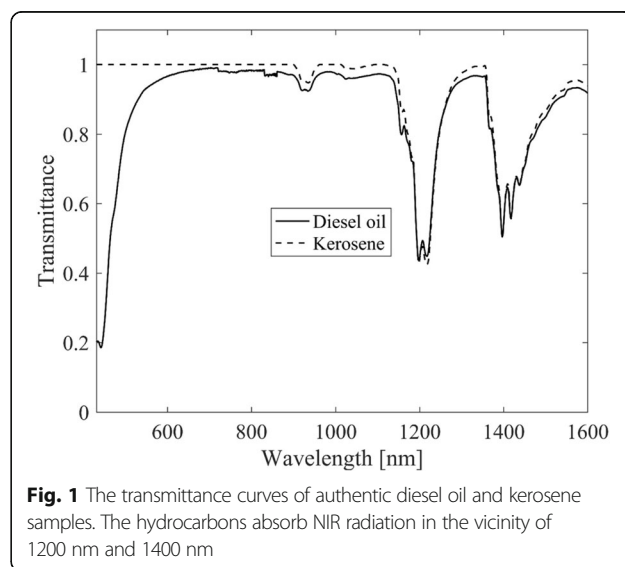
In this paper, we report on the development of a prototype of an optical liquid sensor. The prototype is based on a refined sensor that was originally developed for gloss inspection from rough surfaces in field measurement conditions, such as cold-rolling products in metal industry [17]. We have modified such a glossmeter by implementing a removable sensor head that was used in laboratory conditions for the detection of authentic and adulterated diesel oils.

The idea of the operation principle of the prototype is based on the detection of specular reflection of scattered laser beam from a roughened glass plate. The strength of the measured signal depends on the refractive index mismatch between the glass and the fuel sample as well as the wetting property of the fuel (i.e., the contact angle between a solid-liquid interfaces).

Methods

For the laboratory measurements, the diesel oil was purchased from a gasoline station (the origin of the crude oil of the diesel oil is Russia) and the kerosene sample used in this study is a low odour commercial product (Alfa Aesar). A well-known laboratory quality kerosene, which is readily available for scientists across the globe, was used as a reference. The mixtures composed of diesel oil and kerosene were prepared by carefully measuring and mixing the volumes of the two liquids. The mixtures were shaken well to guarantee optimum mixtures.

Differentiating diesel oils grades and kerosene is problematic because both show rather similar absorption fingerprints at visible and near infrared spectral regions. This is illustrated in Fig. 1, where the transmittance curves of the diesel oil and the laboratory grade kerosene, which were measured using a cuvette and a



spectrophotometer (Perkin Elmer Lambda 9), are shown in the Vis-NIR spectral range. Although, there is a big difference in the transmittance of the diesel oil and kerosene below 600 nm, this difference practically vanishes if we mix even a small amount of kerosene in the diesel oil (i.e. practically, similar transmittance spectra for the pure and fake diesel oils are observed below and above of 600 nm).

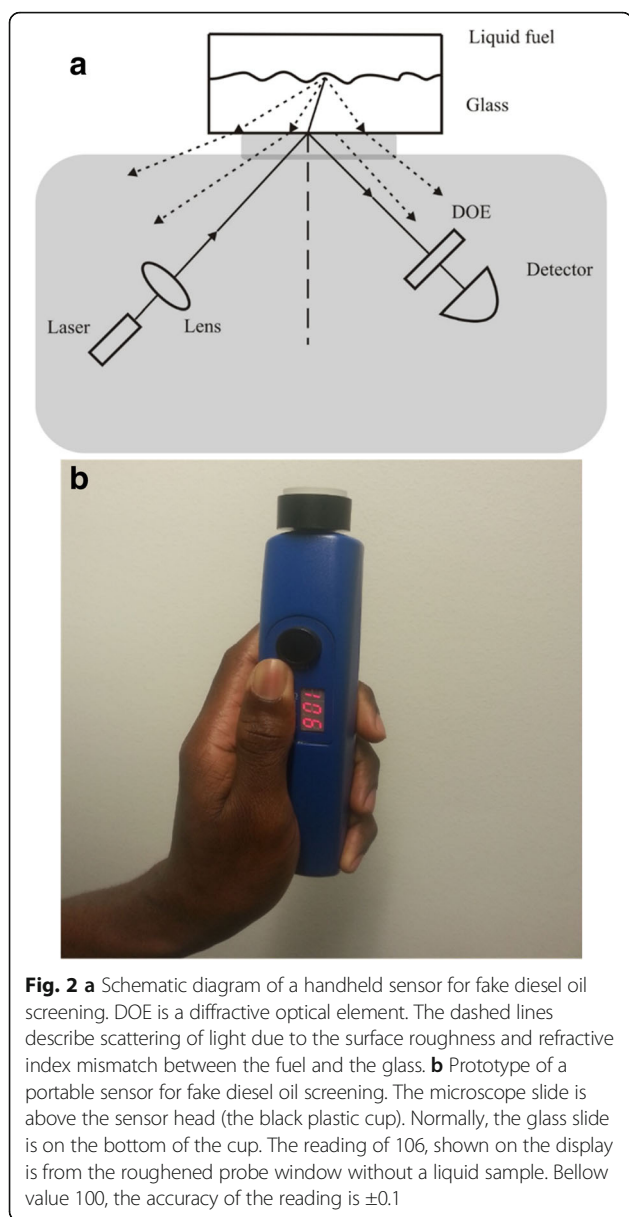
The refractive index for each of the fuel samples was measured at room temperature with the aid of an automatic temperature controlled Abbe table refractometer (Atago RX5000) with a measurement wavelength of 589 nm, and a relatively high accuracy of the refractive index reading ± 0.00004 . This refractometer is designed for laboratory use and requires alternate current for its operation hence it is not suitable for field measurements in the case of lack of AC electric current supply. Therefore, a battery-operated device is preferred. A hand-held Abbe refractometer could be a potential candidate for such a purpose but unfortunately, the accuracy is usually ten times lower than with a table model. To test the applicability of a hand-held refractometer for screening of fake diesel oils, we conducted a field measurement campaign of fake diesel oils in Tanzania. The positive outcome was that this device can be used for screening a diesel oil, but only for pass or fail due its inability to differentiate between samples with 5–15% kerosene. Additionally, the complexity of the problem can be realized based on the fact that the refractive index of diesel oil and kerosene depends on the origin of the crude oil [18], which can be a source of complexity in screening fake diesel oils by refractive index reading.

The inability to differentiate the adulterated samples with high confidence (i.e. regarding the information of the kerosene percentage in the diesel oil) during the field testing of diesel oils in Tanzania by using the hand-held refractometer, stimulated us to develop another and completely different type of a portable optical sensor for fake diesel oil screening. The prototype of a sensor that we suggest in this article is based on the idea that a roughened glass can be used for the identification of liquids, namely using the concept of refractive index matching between the roughened glass and the immersion liquid [19]. For this purpose, we have designed and tested in laboratory conditions a modified sensor head mounted on a commercial hand-held glossmeter [17] (MGM-Devices, Finland). The modification includes implementation of a roughened glass in the sensor head. We wish to mention that in our recent work, we have successfully used this glossmeter for screening fake antimalarial tablets [20].

Before delving into the detailed description of the prototype sensor, we wish to remark that we have developed accurate liquid purity sensor, which is based on the

use of a diffractive optical element (DOE) [21, 22] and laser beam transmission of a sample compartment. A similar DOE forms the key element in this new prototype sensor. The DOE is a computer-generated hologram, which was fabricated by electron beam. Details of the sensing properties of the DOE have been described previously [23]. Given a brief description of DOE, the idea is to use the specular component of simultaneous diffuse and specular reflection of coherent light incident on the DOE, to reconstruct the computer-generated hologram (CGH). This new prototype sensor operates in the reflection mode rather than the transmission configuration utilised in the previous DOE-based liquid purity sensors, which were not hand-held devices. The layout of the current portable prototype sensor that operates on a rechargeable battery is sketched in Fig. 2 (a). The light source is a low power (output power 0.8 mW, wavelength 635 nm) semiconductor laser. It is possible to choose other wavelengths as the source of light for the device, but the DOE of the commercial hand-held glossmeter, our modified sensor, was designed for this wavelength of operation. The choice of such semiconductor laser is due to its good output stability, which has been tested in laboratory conditions under mechanical and thermal external disturbances. The DOE and the laser are inside the handheld device shown in Fig. 2 (b). The diameter of the aperture of the output laser beam is 2 mm. The angle of incidence and detection of the laser beam is 6 degrees with respect to the surface normal of the sample. The laser beam is focused (focal spot diameter ca. 50 μm , given by the MGM Ltd) on the surface of a glass slide (VWR microscope slide ECN 631–1550). The refractive index of this glass at 635 nm is 1.4570. The other face of the glass was roughened, hence it acts like a light diffuser. The glass disk has diameter of 25 mm and thickness of 3 mm. The smooth face of the disk is in contact with the 2 mm aperture of the device. Roughening of the glass was performed by using diamond grinding pads (crisscross surface finish) with different grits, and the measured average surface (measured with a stylus profilometer) roughness was $R_a = 0.48 \mu\text{m}$, which is comparable to the wavelength of the laser of this study. Nussbaumer et al. [24] have described, with the aid of a facet model of dielectric surfaces, different situations of light transmission through a roughened glass plate in a stable immersion liquid.

In Fig. 2 (b), we demonstrate how the prototype portable sensor, in principle, can be used for detecting fake fuels. For the sake of illustration, Fig. 2 (b) shows the roughened probe window placed in front of the aperture of the device. In real measurement, the rough window is sealed by an O-ring inside the measurement head (i.e. a black plastic cup), and it cannot be seen in the side view.



A drop of fuel sample, 10 μL , is introduced by a digital pipette (use of a disposable cheap plastic pipette is possible in field measurement conditions) over the centre of the roughened glass disk.

We measured the contact angle of both the authentic (diesel oil and laboratory grade kerosene) and mixed fuel samples over a glass plate. The measured contact angle of kerosene was 11.2°, and that of diesel oil was 25.9°. The range of values obtained for the contact angles of the mixtures were between the values obtained for the kerosene only and diesel only. The contact angle has an influence on the spreading speed of the fuel drop, i.e., wetting of the rough glass surface. In principle, contact angle measurement can be used for identification of fake diesel oils, but then the measurements must be

performed in controlled laboratory conditions (i.e. not favourable for field conditions) that utilises expensive and non-portable device. We remark here that, rigorous theoretical treatment of the contact angle in the setting of the present prototype requires measurement of transmission or reflection spectra at very wide spectral range, and rather complicated calculation of the Hamaker constant using the theory of Lifshitz as described in the review article [25]. These calculations usually assume smooth surface. As far as we know, rigorous theoretical model for contact angle (based on the Hamaker constant) that incorporates surface roughness and wetting, has not been presented in the literature, so far.

The fuel drop spreads relatively quickly over the rough window of the prototype. The spreading of kerosene is faster than spreading of diesel oil, which can be understood from the point of view of the much smaller contact angle of kerosene. We made long sequence of experiments, and repetitions to optimize the fuel drop volume and measurement time instant. With the present setup and fuel samples, the optimum drop volume was 10 μL . In the case of a smooth glass disk, the thickness of the fuel layer was calculated to be ca. 32 μm . Studies involving the spreading of liquid, contact angle and surface roughness have been investigated elsewhere [26, 27]. These studies describe rather complicated hysteresis phenomenon of the wetting process of a rough surface.

During the measurements, the sensor head was covered by a non-transparent lid in order to prevent leakage of stray light to the detector. The probe window was removed from the measurement head, i.e., plastic cup, and rinsed after each measurement with ethanol to remove the fuel film.

Results and discussion

In Table 1, we show the refractive index data (n) of pure diesel oil, kerosene, and adulterated diesel oils. It is obvious that the table model Abbe refractometer can differentiate all the samples by the refractive index reading, which is linear to the percentage of adulteration. The highest refractive index mismatch is between the diesel oil and the glass, and lowest between the 15% adulterated diesel oil and the glass.

Table 1 Refractive index (n) of authentic diesel oil and kerosene fuels, measured with a table model of Abbe refractometer. The refractive index of the roughened glass is 1.4570

| Sample | n | S (1 min) | S (20 min) |
|-----------------------|---------|-------------|--------------|
| Diesel | 1.46373 | 130 | 109 |
| Kerosene | 1.44230 | 160 | 170 |
| Diesel + 5% Kerosene | 1.46269 | 166 | 133 |
| Diesel + 10% Kerosene | 1.46163 | 134 | 126 |
| Diesel + 15% Kerosene | 1.46060 | 127 | 114 |

Since the data of Table 1 were measured at room temperature, different readings in field measurement conditions are obtained. This is due to the thermo-optical constant of these fuels. In other words, the refractive index of the fuel depends on the temperature of the fuel [28]. In principle, upon the choice of the laboratory grade kerosene as a reference, we can compare the measured refractive index values of suspected fake diesel oils with that of the reference at the same measurement temperature, either out-of-doors field conditions, or inside-the-doors laboratory conditions. Hence, the temperature of the sample is not a big issue.

First, we measured the signal (S) from the smooth glass disk and got the value $S = 184$ air as the ambient medium. Then the same disk was roughened as described above, and repeated the measurement and got $S = 106$ for the rough disk. Table 1 also shows the readings (S) obtained from the fuel samples with the prototype, which are averages of several measurements. The prototype was fixed on a stable stand during the measurements. The first column shows S after 1 minute of the introduction of the fuel drop over the rough glass, and the second column gives data after 20 min had elapsed, and the system was stable. The highest signal was obtained for kerosene and lowest for diesel oil. Both 1 min and 20 min data for the adulterated samples are consistent regarding the relative magnitude of measured S reading. However, by comparing the 1 min and 20 min data, it is evident that dynamic processes occur in the very beginning of the experiment as manifested in the rather high reading for 5% adulterated diesel oil obtained after 1 min. This dynamic processes almost vanishes after 20 mins (thus when the oil has fully settled on the disk) which makes the data measured after 20 min more reliable. The accuracy of the reading is about ± 1 S units. The strength of the detected signal depends on the refractive index mismatch between the rough glass and the fuel interface where scattering of light occurs. Furthermore, the surface smoothness of the back surface of the fuel film-air interface has importance, namely it may imitate a plane/curved mirror or a rough surface depending on the liquid settling over the rough surface, and especially the contact angle. In case of diesel oil, due to its high contact angle, the surface of the fuel layer that settles on the roughened glass tends to assume similar shape as the surface undulations on the rough glass, and hence acts similar to the case of rough glass surface-air only, which yields the low S reading at 20 min. On the other hand, kerosene, due its relatively low contact angle, tends to fill the valleys of the rough surface and forms more or less like a plane mirror yielding the relatively high S reading obtained at 20 min. This phenomenon resembles the case of the smooth glass for which $S = 184$. The signal strength depends simultaneously

on both the surface roughness and the refractive index mismatch. The complexity of the phenomenon is obvious because the data for the adulterated samples shown in Table 1 are not following a simple rule that one might assume, namely increasing the percentage of kerosene should result in higher values of S . However, this is not the case, since wetting of the rough surface is an important factor that should be considered regarding the strength of the detected signal. Unfortunately, the behaviour of the contact angle of a liquid over a rough surface is rather a difficult and complex phenomenon that demands development of appropriate theory. This theory is assumed to be based on van der Waals interactions and the exploitation of the Hamaker constant, which depends on the permittivity of the glass and fuel, respectively. This permittivity is not the well-known conventional permittivity because it depends on purely imaginary wavelength (this can be calculated from a Kramers-Kronig relation [25] where the real-valued angular frequency is replaced by purely imaginary-valued angular frequency). This type of theoretical treatment is beyond the scope of this study. So far, we know that the measured signal is a delicate indicator of small change in the magnitude of the refractive index of the fuel and, in addition to even or uneven settling of the fuel, due to its viscosity, over a rough surface. Anyhow, from Table 1 it is evident that the experimental data for 5–15% adulteration supports the validity of the measurement method with relatively high confidence. Naturally, the stable laboratory conditions favour the operation of the prototype, therefore more design and tests are necessary to further develop and optimise the portable prototype sensor to meet the quite challenging field conditions.

Conclusions

We developed a portable prototype of a hand-held sensor, which is based on the detection of light reflection (scattering) from a rough glass-fuel film interface. The measured signal depends on both the surface roughness of the glass, refractive index mismatch between the glass and the fuel film, and the fuel's contact angle. According to the preliminary tests conducted in the laboratory, it is possible to differentiate authentic and adulterated diesel oils. The hand-held glossmeter provides a platform for multipurpose mobile device for measurements of fake products. On one hand, it can be used for detection of fake pharmaceutical tablets (solids), and on the other hand, for fake diesel oils (liquids). One unique merit of the proposed prototype sensor is its ease of use, which does not demand any chemometrics analytical methods. Even the use of chemometric analytical methods would be very challenging and near to impossible in cases where both the fake and the authentic fuels show similar spectral features.

Finally, we wish to mention that the proposed prototype sensor has a wireless data transmission system, which can be used for studying the dynamic processes of wetting of liquids over a rough glass surface. This will permit the monitoring of a time-dependent signal that carries information on hysteresis of liquid wetting immediately after a drop of liquid is introduced over the surface of a material. We believe that this prototype will have general importance on the detection of dynamics of wetting of any liquid over a transparent roughened surface.

Abbreviations

DOE: Diffractive optical element; N: Refractive index; S: Signal

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Availability of data and materials

Detail about data has been provided in the manuscript.

Authors' contributions

Sample preparation, measurement of spectra, measurement with prototype and writing parts of the manuscript was done by BK (MSc). Measurement of the refractive index of the samples and some contributions in writing was done by KK (PhD), and JR (PhD). Preparation of the rough window together with measurement of surface roughness was done by PS (PhD). Finally, contribution to the development and measurement of samples with prototype, and writing parts of the manuscript was done by PB (PhD) and K-EP (Prof). All authors read and approved the final manuscript.

Ethics approval and consent to participate

Not applicable

Consent for publication

Not applicable

Competing interests

The authors declare that they have no competing interests.

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