

Determining the Composition of 100 Nano-Liter Fuel Mixture in a Silicon Micro-Cuvette Using Ultraviolet Absorption Spectroscopy

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Abstract—Silicon microfabrication technology was employed in the generation of a cavity in the form of a truncated inverted pyramid with very smooth sidewalls and a silicon dioxide UV transparent optical window at the bottom. This can be used as a micro-cuvette for testing of fluids. 100nl fluid volumes can be tested using this platform by ultraviolet absorbance spectroscopy employing a deuterium lamp and an UV spectrometer. Mixtures of commercially sourced diesel with ethanol, and diesel with kerosene were tested in this work. Two zones of interest were identified in the absorption spectrum of diesel. For a 0 to 10% diesel in ethanol mixture, a clear shift of the UV absorption edge from 297nm to 336nm was observed. For the diesel in kerosene mixture a linear shift of the absorption edge from 335nm to 365nm was observed for the entire composition range. Our results indicate that a single wavelength source and detector at 345nm is sufficient to determine the diesel in kerosene composition using this micro-cuvette, which would be very useful in detection of adulteration in fuels. [2020-0344]

Index Terms—Micromachining, spectroscopy, microfabrication, microfluidics.

I. INTRODUCTION

THE testing of liquid mixtures has many important applications, and there is a demand for real-time fast testing of nanoliter-scale volumes. In this work we have designed and fabricated a novel system that allows real-time testing of 100 nanoliter amount of mixture of liquids by ultraviolet spectroscopy. This technique is versatile and is compatible with a wide range of chemistries, such as the formation of nanoparticles through chemical synthesis [1], biomedical processes [2], as well as for the testing of alcohol, spices and edible oils [3]–[6].

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In this work however, we focus exclusively on the mixtures of ethanol and diesel, and kerosene and diesel. The first mixture finds importance in bio-ethanol for biofuel applications, and the second is typically linked to adulteration due to low cost of kerosene. Another important application lies in the area of cold flow properties of kerosene-diesel mixture. Kerosene is typically added at refineries to meet cloud-point guidelines and avoid the formation of wax during pipeline transportation of fuels in cold weather [7]–[11]. In either case, there is a need for a quick, low cost determination of fuel composition, based on equipment that is portable and powered by renewable or rechargeable sources. Our system is targeted towards these applications. However it is not designed to be compatible to combustion processes, where thermal and structural considerations must be made.

Testing of biofuels by a wide range of techniques has been reported in the literature. Electrical impedance sensors [12]–[14] for adulterated biofuels have been demonstrated using various electrode geometries. Simultaneous use of mass and capacitance transducers to measure the amount of ethanol in gasoline has been reported [15]. Electrochemical impedance spectroscopy was employed by Jafari *et al.* [16] to explore the corrosive behaviour of metallic components of gasoline and ethanol fuel mixtures. Optical fiber-based sensors have been used to test adulteration of petrol and diesel using kerosene [17], [18]. Lima *et al.* [19] suggested a technique of photo-thermal analysis for adulterant detection in gasoline based on the time-variation of vapour-phase thermal diffusivity. Fluorescence based techniques has been used in determining the properties of such fuels, [20] along with visible [21], near [22] and mid infra-red [23] as well as Raman spectroscopy [24].

Testing of diesel-alcohol mixtures using visible spectroscopy has been a challenge due to the presence of various additives that are routinely mixed, which have signatures in the visible range. These limits testing to the ultraviolet (UV) region, and there are some reports of UV spectroscopy of diesel mixtures [25], [26]. However, these reports indicate that due to very high optical absorbance of diesel at shorter wavelengths, the transmitted signal is reduced to the equivalent noise level of the detector, even for special cuvettes of 1mm path length [27], and further dilution using a suitable solvent (cyclo-hexane) was necessary. Ethanol on the other hand has little UV absorption.

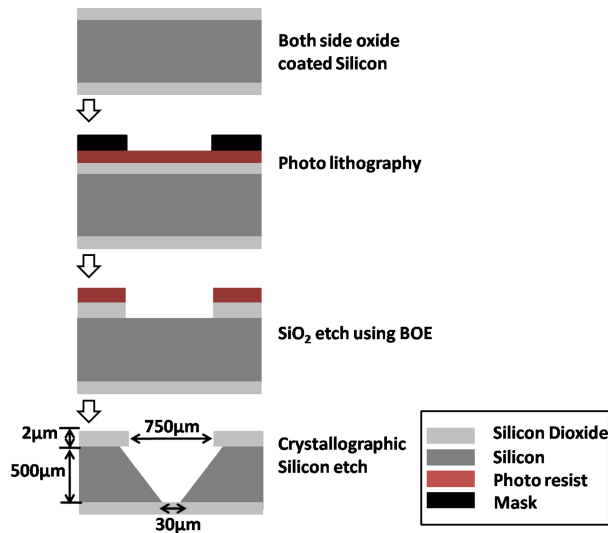


Fig. 1. Schematic of the fabrication of μCuv .

The mixture of kerosene and diesel has also been studied by various groups, and the signature in the Infrared has been employed in transmission spectrum [28] for the detection of mixture composition. In this work however we focus on the UV domain, that is in the range of 200nm-400nm. There are several inherent advantages of the optical testing method over electrical impedance spectroscopy. No specifically designed and fabricated electrodes are necessary, which eliminates the possibility of contamination and corrosion. Unlike IR measurements, UV testing can be carried out in well-lit environment and no optical isolation is necessary.

II. EXPERIMENTAL METHODS

The challenge in optical absorption spectroscopy, as has been mentioned previously, is the very high absorption coefficient of diesel in the UV. In this work, this has been addressed by use of a cavity with optical path length of less than 500 micrometers, which reduces the attenuation of the light as it passes through the liquid mixture. This cavity which can be used as “micro-cuvette” (μCuv) was fabricated using the process described below.

Fig 1 shows the various process steps, starting from a silicon wafer that is coated on both sides by a thermally grown silicon dioxide layer of thickness 2 micrometers. Initially a square window of $750\mu\text{m} \times 750\mu\text{m}$ is photolithographically opened in the top SiO_2 layer using wet chemical etch process in a buffer oxide etch (BOE) bath. Subsequently, the wafer is exposed to a KOH bath under conditions where crystallographic wet chemical etch is favoured, leading to the exposure of the (111) planes of silicon. The details of the bath composition and temperatures used have been already reported elsewhere [29]. The etch process is carried out till the bottom SiO_2 layer is reached, which forms a UV-transparent optical window about $30\mu\text{m}$ square.

The resulting structure is shown in Fig 2(a), which is a Scanning Electron Microscope (SEM) image of the etched μCuv arrays. It can be observed that the inner surfaces are well oriented at an angle of 54.7° , and are very smooth.

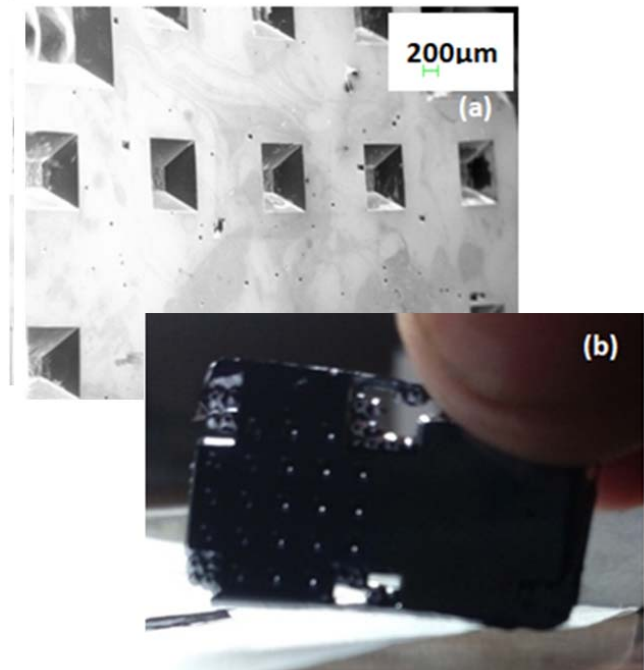


Fig. 2. (a) Scanning Electron Microscopic image of μCuv (b) Image showing the transmission of visible light through SiO_2 transparent window.

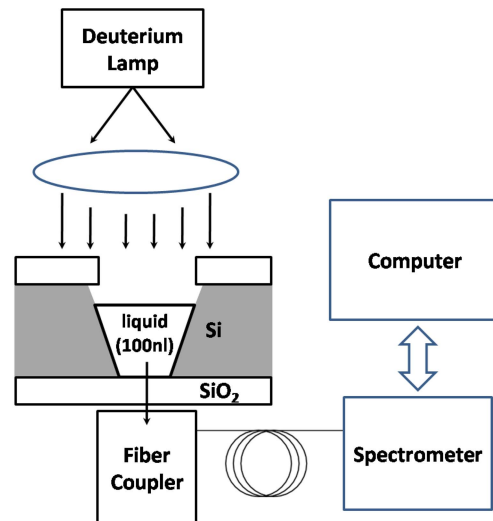


Fig. 3. Experimental set up for optical absorption spectroscopy using μCuv .

Furthermore, the crystallographic etch process has led to the formation of a series of nearly identical structures. The volume of the μCuv is around 100nl.

The transparency of the bottom layer of silicon dioxide can be seen from the image in Fig 2(b). An optical microscope (Leica DM2700) was used to view the liquids inside this μCuv , and their surface wettability, evaporation rate, etc were determined.

The schematic of the measurement setup is shown in Fig 3. The μCuv is shown with a test fluid inside it. A deuterium lamp was used as the excitation source, from which UV light (195nm-350nm) was made incident on the top aperture.

Light is transmitted through the bottom transparent window formed by the silicon dioxide layer, and is collected through

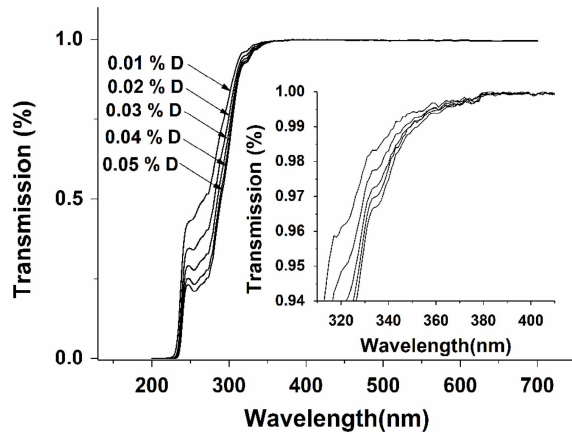


Fig. 4. Transmission spectroscopy of diesel/cyclo-hexane mixture over the UV-VIS range. The inset shows a magnified portion from 330nm to 400nm.

an optical fiber into an UV spectrometer USB 2000 Plus from Ocean Optics.

The measurement setup was calibrated using optically transparent liquid such as cyclo-hexane. All optical components used in this setup are appropriate for operation in the 200nm-400nm range.

The fluids under test were diesel, kerosene and ethanol that were obtained from commercial sources, with the standard additives that are present. No filtration or purification was carried out before testing. Initial optical transmission measurements were carried out using a PerkinElmer Spectrophotometer Lambda 1050 using standard quartz 1cm cuvettes in order to test the optical properties of the fuels and fuel mixtures.

III. RESULTS AND DISCUSSION

A. Spectrophotometry

In order to benchmark our samples and compare them to report in the literature, initial studies were carried out using a commercial spectrophotometer. The mixtures of diesel and cyclo-hexane (D/CH) and diesel and kerosene (D/K) were first investigated, and their optical absorption properties were determined. This was also meant to eliminate the possibility that any local additives which can affect the UV absorption results. The results of the D/CH mixture are presented in Fig 4. It should be noted here that cyclo-hexane is UV transparent, and the optical properties shown here are exclusively from the diesel component. Comparing the D/CH data with that obtained from the literature [27], it can be verified that the nature remains the same, even though the shape of the peaks at ~ 280 nm has some minor variation. This variation may be linked to the fact that, the data in ref [27] is for a diesel-ethanol mixture, which has then further been diluted using cyclo-hexane, and in Fig 4 the ethanol is absent from the mixture.

The absorption spectra exhibit the characteristic features of diesel. The transmission at wavelengths over 390nm is nearly 100%, and falls sharply at shorter wavelengths. The peak at ~ 280 nm has been used earlier as a signature for diesel, but it could be only observed at high dilutions, using a special 1mm path-length cuvette [27]. However, optical testing at 280nm requires special and costly optics, as typical optical glass does not exhibit sufficient transmittivity.

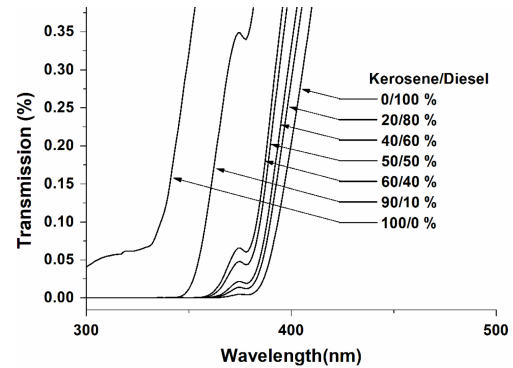


Fig. 5. Transmission spectroscopy of diesel/kerosene mixture of different concentration in cuvette.

We have therefore identified the spectral range 300nm-400nm range for closer investigation. From Fig 4, a sharp absorption edge is observed around 300nm, and for decreasing D/CH ratios a reduction of absorption coefficient can be observed in this spectral range. The inset we present a magnified image of the variation of optical transmission for D/CH mixtures under various compositions in the wavelength range 330nm-400nm. While less expensive optical components can be used for this wavelength range, the variation of absorption is relatively small at these low concentrations of diesel. These initial results indicate that optical measurements can be carried out at either of the two wavelength ranges. For low diesel concentrations, the spectral region around 290nm-300nm can be employed, and for higher diesel concentration, the region around 380nm would be more appropriate.

These results were verified for various mixtures of diesel and kerosene (D/K) where the diesel concentration ranged from 0 to 100%. The results of the spectrophotometer are presented in Fig 5. A clear shift of the absorption edge can be observed from ~ 330 nm (100% kerosene) to nearly 390nm (100% diesel). However, due to the very high absorption in diesel, the shift of the absorption edge for concentration above 40% diesel is quite small, and measurements in this range is susceptible to the presence of noise in the instrument.

IV. MICRO-CUVETTE MEASUREMENTS

Based on the above measurements, we have carried out optical transmission measurements using the μ Cuv based setup as described in the previous section. It has to be emphasized that the volume of fluid mixture within the μ Cuv is only 100nl.

The setup for optical measurements using silicon μ Cuv has already been discussed previously, and a deuterium lamp was used as source of UV light and a fiber-spectrometer was employed for detection. The μ Cuv was filled with fluid mixtures, and observed using a low magnification microscope (10X). Fig 6 shows images of the μ Cuv with a fluid load.

The empty μ Cuv with the $30\mu\text{m} \times 30\mu\text{m}$ optical window at the bottom is shown in Fig 6(a). The smooth sidewalls obtained by crystallographic etching of silicon can be observed in this picture. One further advantage of the process is that the geometry of the cavity and the dimension of the bottom

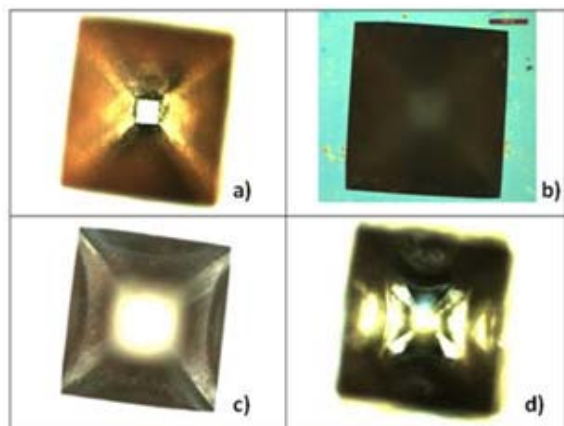


Fig. 6. (a) Empty μ Cuv, (b) after filling with liquid, (c,d) after partial evaporation.

window is determined by the crystal symmetry and is relatively independent of the fabrication process.

Fig 6(b) shows the μ Cuv filled with a liquid mixture. This can be carried out as a part of microfluidic system, with channels populating an array of such reactors. An added flexibility of this process is that the optical path length can be controlled by the volume of the fluid within the reactor which will allow the testing of liquids with very high optical absorption. However, for accurate analysis, the formation of the meniscus due to surface tension, as visible in Fig 6(b) and 6(c) must be taken into account. An added complexity arises for very small liquid volumes as the wetting angle for the liquid in contact with silicon is very different from that formed when in contact with the silicon dioxide optical window. These studies involve detailed optical simulations and will be presented elsewhere. In this work, we limit our studies to measurements obtained using a fully filled μ Cuv. Another point of concern is the volatility of the liquid, especially when the μ Cuvis not capped. Our results indicate that the D/K mixture is not volatile, but the diesel/ethanol (D/E) mixture may evaporate over time. Our optical results have therefore been carried out immediately after filling the μ Cuv for D/E fluid mixtures.

Mixtures of diesel and kerosene, that were initially tested using a spectrophotometer were placed within the μ Cuv, and light from a deuterium lamp was made incident. No attempt was made to focus the light within the $750\mu\text{m} \times 750\mu\text{m}$ area, and it may be assumed that a parallel beam of light was made incident on the liquid surface. Light from the bottom of the μ Cuv was collected through the optical window into a fiber collimating coupler and finally into a spectrometer.

Fig 7 indicates the optical transmission spectra of a series of diesel/kerosene mixture obtained from the μ Cuv setup. It should be noted that each spectral measurement was carried out using an integration time of 100ms, and an average of 3 scans was made, such that the total time required, including data transfer and recording is less than 10 seconds. This is critical for many applications where reaction kinetics can be determined from a time variation of the absorbance. The noise observed in the spectra can be reduced or eliminated by using longer integration times.

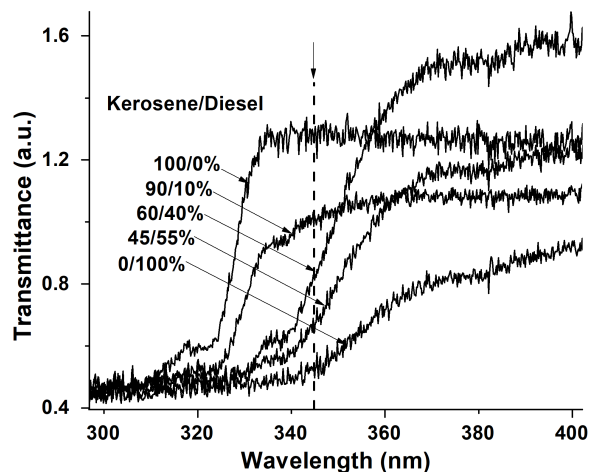


Fig. 7. Transmission spectroscopy of diesel/kerosene mixture using the μ Cuv setup.

From the spectra it is clearly seen that the absorption edge is shifted from 380nm to 330nm with the dilution of the diesel by kerosene. Comparing these results with those that obtained from the commercial spectrophotometer with a quartz cuvette with 1cm path, it can be observed that the data for higher diesel concentrations are more clearly differentiated using the current setup. This is due to the fact that the optical path has been reduced to $\sim 500\mu\text{m}$, which is a 200 times smaller than that for the cuvette. This is very beneficial for measurement of highly absorbing fluids such as diesel.

This shift of the absorption edge has been quantified in Fig 8(a) where the mid-point of the sharply rising slope is plotted for various D/K mixtures. From this calibration curve for the position of the absorption edge, the composition of an unknown liquid can be identified. This technique is versatile and can be expanded for other liquid mixtures, by appropriate choice of the spectrometer grating. The accuracy of the measurement will depend on the resolution of the instrument.

We can also envision a simpler, specific system for testing of fluids. The vertical dashed line in Figure 7 indicates an optical absorption measurement at a 345nm for the various liquid mixtures tested. The wavelength specific measurements are presented in Fig 8(b). The error bar represents noise in the spectra, and can be easily reduced at the cost of a longer integration time. It is clear that the absorption constant at the particular wavelength can be a clear indicator of the composition of the fluid, as long as the optical path length and other instrumentation constants remain the same.

The content of diesel and kerosene fuels depends on the source of crude as well as on the subsequent processing. Vempatapu *et al.* [30] has carried out extensive measurements on the mixture of diesel and kerosene from sources similar to those used in this work employing high resolution mass spectroscopy and other techniques. They conclude that with the increase of kerosene content there is an increase in the n-C₁₀ to n-C₁₂ ranges and a corresponding decrease in the n-C₁₅ to n-C₂₀ compounds. In addition, there is an increase of content of the alkylbenzenes. It is possible that our results, that is the increase in UV absorption in the 300-350nm range, and consequent shift of the absorption edge with increase of

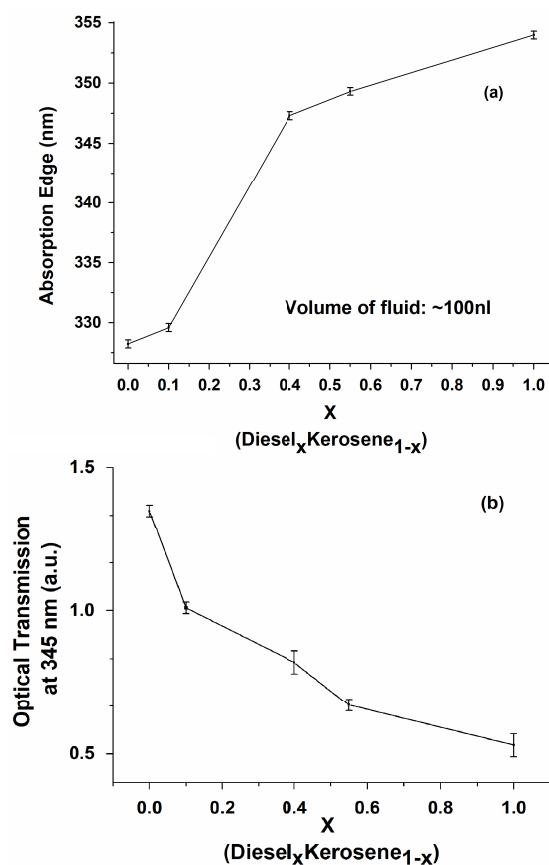


Fig. 8. (a) Shift in the absorption edge with the dilution of diesel with kerosene, (b) The optical transmission at 345nm for various concentrations (here X represents the percentage concentration of diesel in diesel/kerosene mixture).

diesel content is linked to these variations. However further studies are necessary to establish these correlations. While our system is capable of testing fuels in location, studies can be carried out at a subsequent stage correlating the results with lab-based measurements such as Gas Chromatography, High-Performance Liquid Chromatography (HPLC), Mass spectroscopy [30], Nuclear Magnetic Resonance (NMR) [31]–[33] etc for a more detailed analysis [34]–[38].

We extend this study to diesel/ethanol mixtures. As previously indicated the wavelength range for detection of diesel in a mixture may be chosen to be either around 345nm as showed for D/K mixtures, or around 290nm, and in this case the shorter wavelength range is more appropriate. Spectroscopic measurements were performed using the μ Cuv system for diesel/ethanol mixtures and the results are presented in Fig 9.

UV transmission spectra were obtained after placing various solutions of diesel and ethanol in the μ Cuv system. The diesel content was varied from 1% to 10% and no further dilution was carried out. A clear shift of the absorption was observed from 297nm to 336nm for these mixtures, with increasing diesel content. For these measurements, the stronger absorption caused the level of noise to increase. However in a process similar to that described previously, a specific wavelength \sim 300nm can be chosen and a single wavelength source and detector based system can be designed to test for these mixtures.

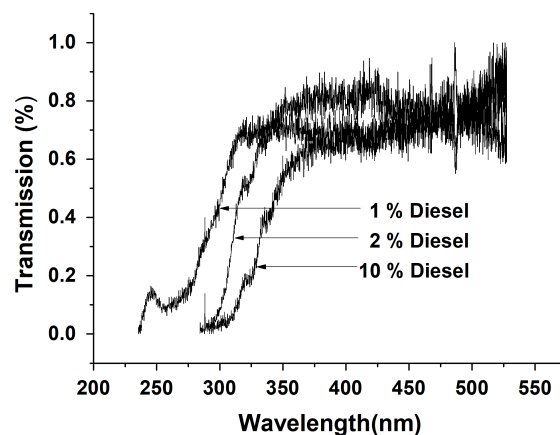


Fig. 9. Transmission spectroscopy for different concentration of diesel in diesel/ethanol mixture used in the μ Cuv.

The results shown in Fig 8 and 9 further demonstrate that for specific fluid testing the spectrometer-based measurements can be replaced by a much simpler system where the testing will be carried out at a single wavelength. Narrowband sources of ultraviolet light based on the Light Emitting Diode configurations are currently entering the commercial market. Single wavelength photodetectors have also been reported [39], that are sensitive to a specific narrow wavelength range within the ultraviolet spectrum. This will allow the measurement to be free from spurious effects such as produced from fluorescence or background light. These LEDs and photodetectors are based on AlGaIn alloys, which are inherently robust and chemically inert, insensitive to environmental conditions. Incorporation of such a source-detector system will allow the development of compact, robust and low-cost application-specific systems.

Since our current system has been developed for testing at the field, we have employed spectrometers with relatively low sensitivity levels below 300nm, to help reduce cost and complexity. However, we have carried out simulation measurements to understand the effect of the shorter path lengths for measurements the 200nm-300nm wavelength range.

Testing of a cyclo-hexane/diesel mixture at 1000:1 dilution carried out by a commercial spectrophotometer using a standard 1cm cuvette provides viable data, as shown by the round symbols in Fig 10 (a). Using this information, the transmission curves for various other dilutions were simulated using the equation

$$I = I_0 \exp(-\alpha * d) \dots \quad (1)$$

where I and I_0 represent the incident and transmitted light, α is the absorption coefficient and d is the path length within the liquid. An assumption was made that the absorption coefficient would scale with dilution.

The calculated optical transmission shown in Fig 10 (a) curves indicate that for higher concentrations, the light is expected to be mostly absorbed and the signal at the detector will be far below the baseline noise levels, assumed to at the 1% transmission, as marked by a horizontal line. This is expected, and corresponds to both our results as well as that presented in the literature.

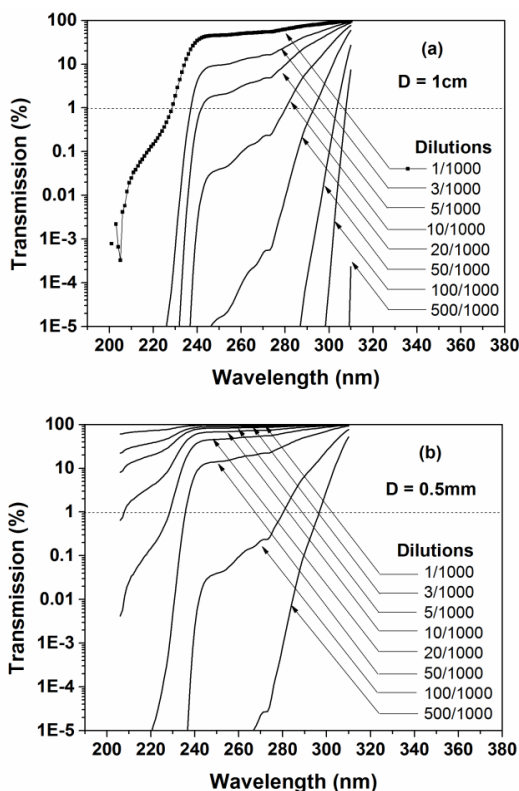


Fig. 10. Simulation of optical transmission for different concentration of diesel in diesel/cyclo-hexane mixture for (a) 1cm Cuv and (b) 0.5mm μ Cuv.

However, if a μ Cuv of 0.5mm path length is employed for the same task, the corresponding calculated optical transmission values are expected to be significantly higher, as shown in Fig 10(b). It indicates the use of the μ Cuv will allow measurements at the deep UV range without significant dilution, if an appropriate setup is employed with high sensitivity at these wavelength ranges. This is easily accomplished for single-wavelength application-specific measurements through the use to high power LED sources and appropriate single-wavelength photodetectors.

These results are expected to hold good for any fluid groups that have significant absorption at these wavelength ranges. Thus, use of a μ Cuv will cater to a wide range of applications.

V. CONCLUSION

In this work we present an optical measurement system for 100nl fluid mixtures using UV spectroscopy. While the system can be used for a wide range of applications, in this work we focus on the testing of mixtures of diesel and ethanol and diesel and kerosene.

A silicon micro-cuvette structure was fabricated using microfabrication techniques. Our results indicate that due to the very small optical path lengths in a silicon μ Cuv, mixtures of diesel with kerosene can be tested at the UV-B (\sim 325nm-355nm) range and a clear shift of absorption edge can be observed with varying composition. A similar study was carried out with diesel/ethanol mixtures at UV-C (290nm-310nm) range.

Based on our results we envision that an on-chip detection system can be developed using single wavelength sources at 300nm or 345nm and corresponding wavelength-selective photodetectors, for the testing of D/K or D/E mixtures without the need for spectroscopic measurements. While contents of such fuels are dependent on their source as well as the subsequent process steps, with appropriate calibration procedures that address such variations, this can be an useful tool for field testing of fuel mixtures without any elaborate, power consuming or expensive equipment.

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