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Silicon Microcuvette-based Testing System for Optical Absorption Spectroscopy of Vegetable Oils

Suchismita Paul, Alakananda Das, Dr. Anirban Bhattacharyya

***Abstract*— A fast and simple testing method for micro-nano liter volumes of agricultural products can be very advantageous in the food industry for quality control purposes. In this work, optical transmittance spectra of olive oils, heated to different temperatures, were compared using a custom-built test system. Results indicate that testing at two specific wavelengths of 370nm and 470nm is sufficient to determine their state of oxidation and to distinguish them from other oils. A novel microcuvette (µCUV) was designed with integrated heating as well as optical detection capabilities for this purpose. Silicon micromachining techniques were used to fabricate an inverted pyramidal structure, with an opening 750µm x 750µm, sidewall slope of 54.7˚ and an optical window of 30µm x 30µm at the bottom. To carry real-time spectroscopic measurements, while maintaining a liquid under test at controlled elevated temperatures, micro-heater structures surrounding the µCUV were designed. The design parameters were the dimensions of the silicon structure, micro heater geometry, thickness of the heating element and inter-element spacing. While several geometries were compared, simulation results indicate that due to the sloped sidewalls of the µCUV and presence of corners, multiple specially-designed heaters at the top as well as the bottom of the structure is necessary to obtain a temperature variation of less than 8% across the inner surface of the µCUV. The silicon-based system can be directly integrated into on-chip control electronics catering to a wide range of applications. We propose a fast and versatile liquid test system based on our results.**

Index Terms— Fluidic microsystems, Micromachining, Optical sensor, Real time system, Simulation modelling, Vegetable oils.

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I. INTRODUCTION

THE testing of microscopic volume of fluids is necessary for a wide range of applications, including medical diagnosis, petroleum, pharmaceutical or food processing. In many cases the identification of the fluid under test must be carried out at the field-level, using hand-held, battery powered equipment. Therefore, well-established methods such as HPLC (High pressure Liquid Chromatography) are not appropriate as they require a laboratory setting. Electrical testing, such as impedance spectroscopy measurements have proven to be effective in determination of chemical signatures, adulteration, water content, etc. [1-3]. However, this process has limitations because of the need for the immersion of electrical contacts or electrodes [4, 5], which places a limit on minimum liquid volumes, as well as opens the possibility of contamination. Optical testing methods [6, 7] have the advantage of not requiring direct contact with the liquid.

FTIR [8] and UV absorption spectroscopy have been used for testing vegetable edible oils, including canola, olive, and rice-bran [9-11]. Edible oils may exhibit undesirable chemical changes taking place upon thermal processing in air, which can have significant effects on human health [12-14]. Various spectroscopic techniques have been used to test the quality and compositional change of oil with temperature [15, 16]. In particular, UV absorption spectroscopy has been found very useful for determining their oxidation states [17, 18]. However, such measurements are typically carried out using expensive scientific equipment in a laboratory setting by trained personnel. In this work, we focus on the development of a sensing system incorporating microfluidic, optical and microheater components that can determine the oxidative state of vegetable oils as well as distinguish between various types of oils based on optical absorption spectroscopy. This system has been designed to be battery-powered, portable, robust, compact as well as low-cost.

Initial work was directed at determining the critical wavelengths at which optical transmission data is sufficient for this task for a particular oil. In a previous publication by our group, we proposed the use of a novel silicon-based microcuvette for the testing 100nl of fuels by UV absorption spectroscopy, and mixtures of fuels, specifically diesel and kerosene, were studied by this technique [19]. In order to extend the use of this system to test the oxidation rate of vegetable oils by thermal cycling,

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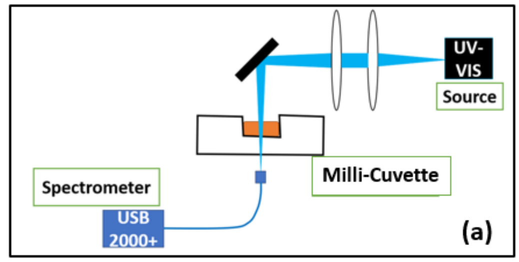
a specially configured micro-heater system must be incorporated on the chip. While micro-heaters reported in the literature typically address uniformity of temperature [20-24] on a simple plane, in this case the challenge was to achieve temperature uniformity over the sidewalls of the microcuvette in the form of an inverted truncated pyramid. In the latter half of this paper, we discussed the design and simulation of such micro-heater systems that allow the uniform heating of 100nl volume of liquid.

II. EXPERIMENTAL METHODS

The work described here consisted of two major components. The first part involved optical transmission spectroscopic studies of a series of commercially sourced vegetable oils that were kept at elevated temperatures for a duration of time to identify critical wavelength and temperature ranges where maximum change in transmittance occurs. The second part was the development of a silicon micromachined microcuvette-based fluid testing system with appropriate microheaters, and optical components designed to cover these parameter ranges in pre-defined temperature and wavelength.

A series of oil samples were processed in an oven in air through a temperature range from 20°C to 170°C and for periods extending from 15 minutes to 60 minutes. Initially the change in optical absorbance spectra of these samples were benchmarked using a Spectrophotometer (PerkinElmer Spectrophotometer Lambda 1050). These measurements were then reproduced using a custom-built cubic-shaped glass milli-cuvette with vertical sidewalls with an optical path length of 1mm, and a volume of 4 microliters. A dual-wavelength light source, consisting of a 50W Deuterium and a 50W QTH lamp was used as an excitation source for this setup, shown schematically in Fig 1(a). The light was coupled into the liquid in the microcuvette through an optical window. The transmitted light was collected through an optical fiber system into an Ocean Optics USB 2000 Plus spectrometer, with wavelength range 200nm to 500nm. In the final setup, the broadband optical source and spectrometer were replaced by LEDs of specific emission wavelengths and a photodetector for real time measurements, as shown in Fig 1(b). In this case, a photodarlington L14F1 was used, and the current measured using a Keithley 236 SMU.

Subsequently, the design of a silicon based microcuvette (µCUV) system with integrated microheater was carried out. The fabrication of the µCUV was carried out on to a 500µm thick double-side-polished, boron doped (100) silicon wafer, pre-coated with a 2µm thick thermally grown SiO_2 layer on both sides. Initially the top SiO_2 layer was selectively etched to form a pattern of 750µm x 750µm square arrays. Crystallographic wet chemical etch process was employed to produce a µCUV cavity bounded by silicon (111) planes on four sides and a bottom SiO_2 layer, which acts as an optical window. This structure can hold ~100nl fluid within. The details of the fabrication process can be found elsewhere [19].



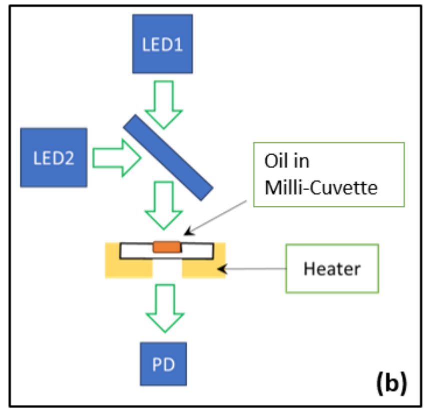


Fig. 1. Schematic diagrams of cubic milli-cuvette setups for testing oils of volume 4 µL using a spectroscopic testing system (a) and a dual-wavelength system using LEDs and a photodetector (b).

TABLE I
MATERIAL PROPERTIES

Material Properties	Silicon Dioxide	Silicon	Tungsten	Olive Oil
Electric Conductivity (σ) (S/m)	10-12	1000	27	~0
Thermal Conductivity (k) (W/m*K)	1.5	149	1440	0.16
Temperature Coefficient (α) (/°C)		-0.07	0.0045	0.00068
Density (ρ) (Kg/m³)	2180	2328	19600	917
Heat Capacity at Constant Pressure (Cp) (J/(Kg*K))	730	711.5	133.97	1790

Microheater structures were designed and simulated using COMSOL Multiphysics software using the Heat Transfer Module and Conductive Media DC Modules. The material properties considered in our work are given in table 1. The goal of the design effort was to generate a uniform temperature profile on the sidewalls of the µCUV, independent of the fluid under test. Finally, the working of the complete sensor system consisting of optical, microfluidic, and thermal elements has been discussed.

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III. Results and Discussion

A. Optical Transmission Spectroscopy of oil samples

Optical transmission measurements were carried out on a series of vegetable oils using a standard spectrophotometer to provide a benchmark. These oil samples were heated to a range of temperatures between 30°C and 170°C for durations ranging from 15 min to 60 minutes. Transmittance spectra obtained before and after the thermal treatment were compared, and selected results are presented in Fig. 2. Specifically, results for extra-virgin olive oils are presented in Fig 2(a) and 2(b) respectively. As can be seen the wavelength range of interest lies between 350nm and 500nm, where the most prominent changes occur. A careful comparison of these two figures indicates that in the wavelength range 370nm, the optical transmittance decreases after the heating process, while in the 450 to 480 nm range there is a clear enhancement of it. Fig. 2(c) and 2(d) compare the transmittance spectrum before and after heating for a series of other vegetable oils and show that no major changes occur in these samples. The clear enhancement of optical transmittance in the 450-480nm range due to increase in oil temperature is possibly due to decoloration of chlorophyll and carotenoid pigments present in EVO [25]. Similar mechanisms [26] are also responsible for the enhancement of optical transmittance at around 670nm [27], which has not been employed in the final instrument setup as no additional inferences can be drawn from variation at this wavelength. The variation of optical transmittance around 370nm range may be linked to thermal changes in phenolic acids and flavonoid compounds [28] [29].

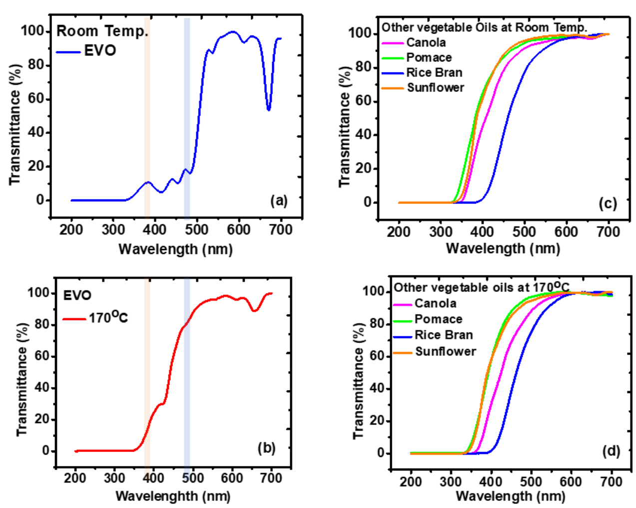

Fig. 2. Comparison of UV-VIS transmittance of EVO at Room Temperature (RT) (a), EVO on heating to 170°C (b); other vegetable oils at RT (c) and at 170°C (d). Measurements were carried out using a commercial spectrophotometer.

These results have several important features that can be utilized for the development of a test system that identifies the oxidation state of EVO and has the capacity to distinguish this particular oil from others that are otherwise visually similar. Firstly, having two independent wavelengths of reference allows the measurement to be relative immune to the drift of photodetector sensitivity over time, since the variations due to thermal processing occur in opposite directions. Secondly, using one wavelength range beyond the visible renders it insensitive to presence of leaked ambient light.

To develop a field-deployable system for oil testing, the first setup described in Fig 1(a) was used to carry out optical transmittance measurements on the EVO and POMACE olive oil samples. The glass milli-cuvettes with vertical sidewalls have an optical pathlength of 1mm and hold 4 µl oil. Transmittance spectra obtained from EVO, as shown in Fig. 3(a) indicates the presence of two wavelength regions that vary strongly with higher temperatures. From 330nm to 390nm, the transmittance of EVO shows a clear decrease, while the reverse is true at longer wavelengths, and a strong increase is observed, while the optical transmittance of pomace oil remains unchanged on thermal processing. These results are similar to those already discussed previously and establish the quality of measurements carried out using the milli-cuvette system.

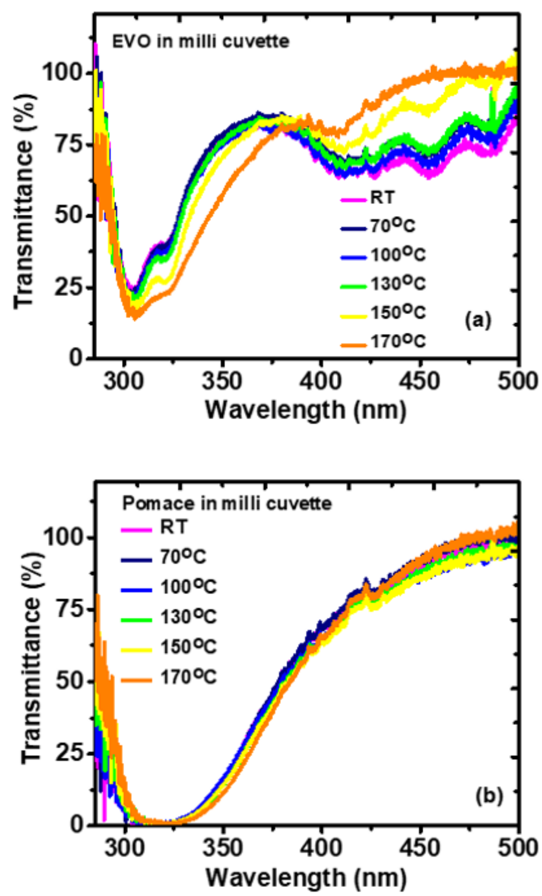

Fig.3. Variation of UV-VIS transmittance spectra measured after thermal processing using a milli-cuvette system for extra-virgin olive oil (a) and pomace olive oil (b).

Real time tests were carried out using a setup where the broadband light and spectroscopic detection is replaced with LEDs emitting at 370nm and 470nm, corresponding to the changes observed previously. The power levels of

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the LEDs were chosen to be low in order to avoid changes of oil properties due to photoexcitation. A photodarlington L14F1 biased at 10 V was employed for optical detection, and current passing through this device upon illumination, as shown schematically in Fig. 1(b) is plotted in Fig. 4, for a range of temperatures through which the oil samples are cycled. The direction of the thermal cycle is indicated by the curved arrows.

For an optical source at 370nm, increasing temperature caused a weak increase in photodetector current. The cooling cycle caused a sharp decrease in photodetector current, and the final value of photodetector current measured at room temperature after the thermal cycle shows a net decrease as indicated by the block arrow. Conversely for the optical source at 470nm, the heating cycle from room temperature to 140°C shows a sharp increase in photocurrent due to increase in optical transmission through the EVO. During the cooling cycle, the signal essentially remained the same, leading to a net increase in photodetector current as a result of the thermal cycle. These results obtained from EVO contrast with that for pomace oil, which showed no discernible variation.

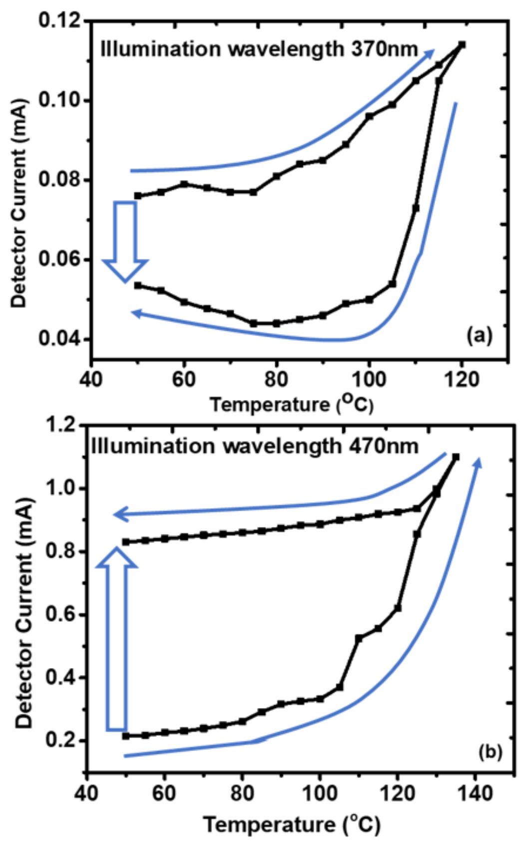

Fig. 4. Real time study of temperature dependence of optical transmission measured using a milli-cuvette, as given by photodetector current, for EVO at wavelengths 370nm (a) and 470nm (b).

From the results shown in Fig 4, it is clear that detailed spectroscopic measurements carried out by expensive benchtop equipment is not necessary, and a relatively simple setup with LEDs emitting at critical wavelengths can be used to determine the state of oxidation of olive oils, and to distinguish EVO from other oils that are visually similar. Based on these results, a sensing system was designed for testing of 100nl oils, as is described subsequently.

B. Design of Micro-cuvette with integrated heating system

The schematic of the microcuvette (μCUV) system developed by our group is presented in Fig 5(a). The cavity in the form of an inverted pyramid has been etched from a silicon wafer using wet-chemical etching process. The underlying SiO_2 layer acts as an optical window, through which light can pass, as shown. Optical testing of the ~100nl volume of liquid can thus be carried out.

Scanning electron microscopy image of an array of μCUV structures fabricated using the process described previously [30] is presented in Fig.5(b). The truncated inverted pyramidal sidewalls are clearly observed. The top opening is a square with side 750μm x 750μm, and the slope is expected to be 54.7˚. The sidewalls are found to be very smooth without any residue from the etching process. The bottom window (not seen in this picture) is 30μm x 30μm in dimension.

In Fig. 5(c) we present an optical microscope image of the top view of the μCUV after filling with a fluid. The effects of surface wettability are clearly seen from the nature of the top surface of the liquid. The bottom window is optically transparent, which is established by Fig 5(d), where the image is obtained in the top view, while a red laser is shone from the bottom. The optical window is shown here to be fully transparent, with no residue or debris that could collect at the bottom of the μCUV structure during the etch process.

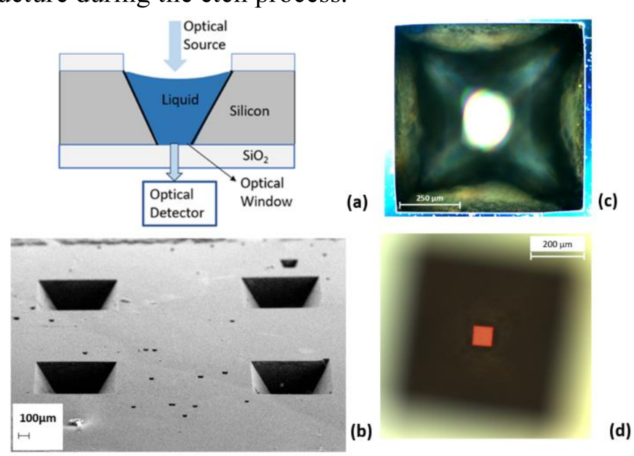

Fig.5. Schematic diagram of μCUV system with fluid under test (a); SEM image of μCUV array (b), top view optical microscope image of μCUV filled with liquid (c), and indicating optical transparency of the bottom window (d).

As established in the previous section, the oxidation of oils can be optically tested by UV absorption spectroscopy after exposure to elevated temperatures. To carry out such tests using a μCUV, it is necessary to integrate heaters that can be used for the task. Most importantly, due to the very

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small volume of the liquid the oxidation will take place within a very small time-window, and convective processes within the liquid cannot be used to establish equilibrium temperature. Therefore, it is necessary that all the walls of the μCUV be maintained at the same temperature during the process, otherwise selective oxidation will take place at hotter parts, reducing the sensitivity of the optical transmission measurements.

The major task addressed in this paper involves the design of a micro-heater system for the above-discussed μCUV structure, which will ensure that the sidewalls of the heater system, will maintain uniform temperature. The target temperature was chosen to be 170°C, based on the results on olive oils presented earlier, as the final target for the simulation effort. The dimensions and configuration of the system design was based on the optical microscope and SEM images, as shown in Fig. 5. For all our designs, tungsten is used as the heater material, due to its high thermal conductivity, high melting point and high resistivity because these are the basic properties of heater material [31]. The heating capacity of a heater is determined by length, width, thickness, and the resistivity of the heating element, and for these planar structures, uniform thicknesses of the metal is considered for sake of avoiding fabrication complexities. A wetting layer is necessary for the fabrication of microheaters in order to promote adhesion, but this layer is not considered in the simulation due to its typical thickness of several nanometers, which does not significantly affect heat transport. The heat flow from the heater element to the surface of the μCUV depends on the geometry that is the distance travelled, thermal conductivity and heat capacity of the materials, in this case silicon and silicon dioxide. As the thermal mass of the silicon is significantly larger than the liquid (EVO), we did not consider the liquid within the cuvette during the simulation.

1. Single heater configuration

Initial designs considered a single heater, fabricated on the bottom side of the μCUV, as indicted by the arrow in Fig 6(a). Two designs have been considered for simulation studies. The first, uniform heater (UH), is a relatively simple design with two distinct zones. The outer zone is composed of tungsten lines with uniform width of 5μm, spaced 5μm from each other. The thickness of the metal lines has been taken to be 5μm as well. This outer zone covers the region that is at a distance between 100 μm and 1.5mm from the center of the heater. Within the inner zone, however, the pattern is different, as can be observed in Fig. 6(b) and 6(c). Here the tungsten lines are wider (10μm) near the center of the straight paths. In this case, the spacings are also wider, which is 10μm. The second single heater, which is progressive one (PH), has three zones of design, the inner, middle, and outer. In inner and middle zone, the straight portions of each spiral contain sections of width 5μm and 10μm. Specifically, the central portion of the straight part, about one third of the length, is wider than the other parts. The distance between the spirals in inner zone is 50μm which decreases to 10μm in middle zone. The outer zone heater consists of two meander-shaped turns, with periodicity of 30 μm, separated by 5μm [Fig.6 (d, e)].

The thermal energy provided by the heater travels through the silicon to the inner walls of the microcuvette cavity, which will be in contact with the liquid under test. As the cuvette is an inverted pyramidal structure, this travel distance from the heater to this surface is shorter near the window as the Si is thinner. As we go further from the optical window, this thickness increases, affecting the heat

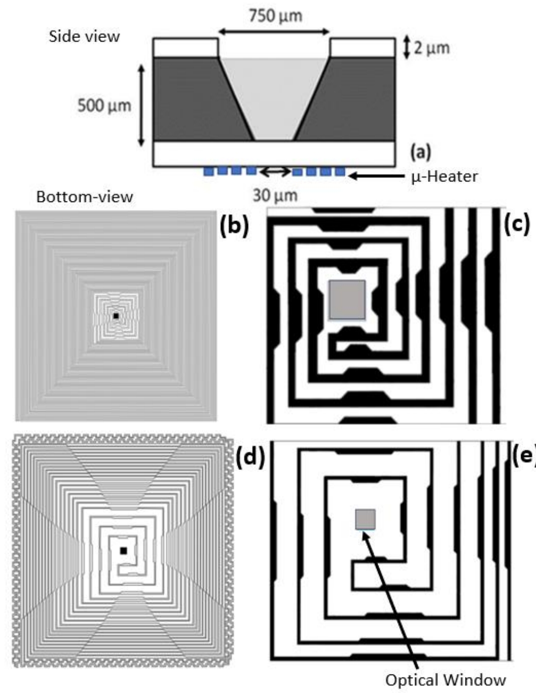

Fig. 6. Schematic diagram showing cross section of μCUV integrated with a single heater system (a); bottom view of Uniform single Heater (UH) design shown at two magnifications in (b, c); and Progressive single Heater (PH) design shown in (d, e).

flow. Since the system must be designed to be independent of the thermal conductivity and other properties of the liquid, our design attempts to ensure that the surface temperature of the silicon would be as uniform as possible. The bottom heater was therefore designed based on the heat travel distance considerations, with higher heater density near the edges, far from the window, to counter the need for longer travel distance. Furthermore, to compensate the heat loss from the corners the width of the heater is comparatively narrow there compared to the middle of the straight edges as seen in Fig 6 (d, e).

The simulation results for the temperature distribution generated by microheater system UH and PH at the bottom plane, surrounding the optical window is represented in Fig 7(a) and 7(b) respectively. It can be clearly observed that the design features employed in the progressive heater play a very important role in increasing the uniformity of the

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temperature. While the simpler structure generates more heat from the same applied voltage, the temperature distribution in that case is higher at the center, near the optical window. Typically, a meander shaped microheater is employed to increase the length of the heater, thereby generating more heat. This has been used in the second design to generate more heat, as the Silicon wafer is thicker at the edges than near the window. Similarly, the design of the corners creates higher and more uniform temperature [20, 32]. The applied voltages were selected so that the mean temperature achieved was ~170°C at the sidewalls in both cases, in connection with the requirement for oxidation of olive oil samples. The required voltages were

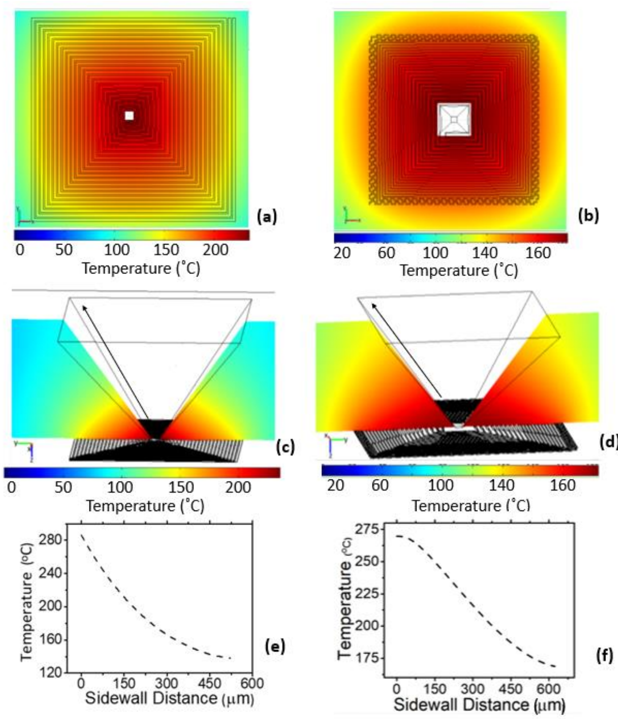

Fig. 7. Comparison of schematic and in-plane temperature variation for a Uniform single Heater (UH) (a) and Progressive single Heater (PH), (b). The temperature distribution map and plot of variations along the sidewalls are shown in (c, e) and (d, f) for the two structures, respectively. The sidewall distance is measured along the arrow shown in (c) and (d)

28V and 55V for the UH and PH designs respectively. The simulation results indicate very different temperature profiles for the sidewalls of the μCUV, as presented in Fig 7(c) and 7(d). Simulated values of temperature at various points of the sidewall are presented in Fig 7(e) and 7(f) respectively. In these and subsequent figures, the x-axis denotes distance along the sidewall, as shown by the arrow. Here the origin is located at the bottom of the cuvette, close to the optical window, and the end is at the top of the cuvette lip.

The temperature variation along the wall of the μCUV employing the UH microheater system was found to be very large. The highest temperature near the bottom of the cuvette is ~ 286°C and the lowest is at the top of that which is ~ 138°C, implying a temperature difference of ~148°C. This difference is unacceptable for carrying out olive oil based or similar experiments, as only a small portion of the oil is in contact with the surface will be heated correctly, leading to erroneous conclusions. It can be argued that the thermal conductivity or convection currents within the oil will lead to a more uniform temperature. That would however make the system dependent on the other properties of the liquid under test, further complicating the final analysis. In addition, such a measurement will likely have time variation, as the liquid temperature equilibrates.

For the μCUV with the progressive heater PH, however, the temperature distribution of the wall is significantly more uniform, ranging from 270°C to 168°C at positions near the heater and the top edge respectively. This makes the PH design more appropriate for the task, even though a higher voltage of 55V is needed to achieve the temperature range. However, the overall variation of ~102°C is still too high for the requirement of oil testing, and further modifications of the microheater design was made, as described in the following sections.

2. Multi - heater configuration

In the previous section, it was concluded that the uniformity of sidewall temperature for a μCUV structure could not be achieved using only a single heater placed at the bottom of the wafer, surrounding the window. A

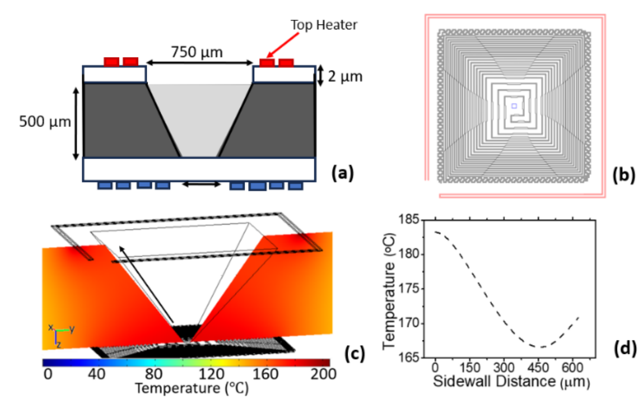

Fig. 8. Schematic of μCUV integrated with Dual Progressive Heater (DPH) system (a); top view of the bottom heater in black and top heater in red (b); simulated temperature map (c) and temperature profile across sidewall (d).

modification of the design was made by addition of a heater at the top of the wafer, surrounding the aperture of the μCUV, indicated by the red arrow in Fig 8(a). The top view schematic of the DPH, as shown in 8(b) also includes this top heater, and is shown in red. The thickness and width of this heater is 5μm, whereas the distance between their spirals is 10μm.The distance between the edge of the opening and the heater was designed to be 525μm. This heater is named as progressive dual heater (DPH), where the PH heater is used at the bottom of the wafer, and a second heater is used at the top. The simulation results are presented in Fig.8(c), where the temperature variation of a vertical slice is shown for the DPH system, and in Fig. 8(d)

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where the temperature values are plotted vs distance from the center. Due to the effect of added double spiral heater at the upper section of microreactor, the temperature difference minimizes to 16.7°C where maximum temperature is around 183.3°C and minimum temperature is around 166.6°C. The voltages applied to the top and bottom heaters are 21V and 32V respectively. While the reduction in voltage variation is significant, our results on heating of olive oils indicate that it is still too large for correct analysis of oil content.

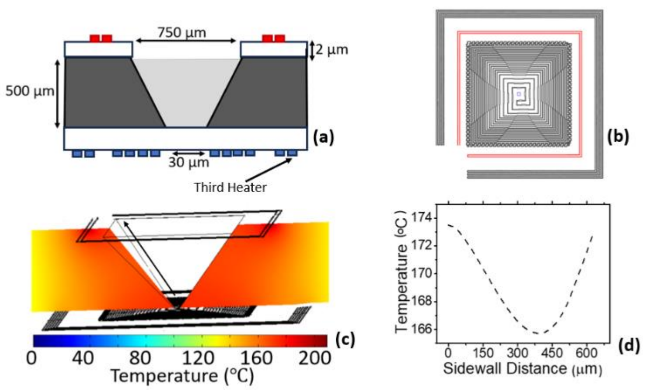

Fig.9. Schematic of µCUV integrated with Triple Progressive Heater (TPH) system (a); top view of the bottom heater in black and top heater in red (b); simulated temperature map (c) and temperature profile across sidewall vs distance from bottom (d).

Further reduction in temperature difference was achieved using a third heater [33], spaced 80µm away from the bottom heater. This design is named as progressive triple heater (TPH). The thickness and width of this heater is 5µm. It has four spirals with 10µm gap from each other. Fig.9 (a, b, c) shows the schematic diagram, design, and temperature profile along sidewall of the µCUV with triple progressive heater (TPH). As can be seen from Fig. 9(d), the temperature difference along the side wall of the cuvette is reduced to 7.25°C, where maximum temperature is around 173°C and minimum temperature is 167.75°C. The applied voltages for the top, bottom inner and bottom outer heaters are ~21V, 23V and 20V respectively.

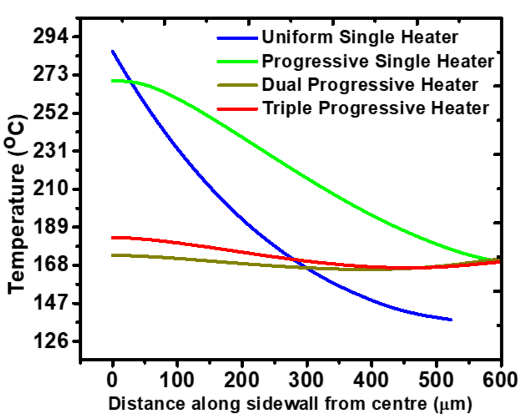

Fig.10. Comparison of temperature along sidewall for different microheater configurations, as measured from bottom center to the top lip of the µCUV.

Fig.10 summarizes the simulation results. The temperature variation along the slanted surface of the µCUV structure, from near to the top aperture to the bottom window is plotted for the various designs, the uniform, progressive, dual progressive and triple progressive systems. The results indicate that while the triple progressive system is relatively complex. It can achieve the necessary uniformity of less than 8% across the walls. The addition of the third heater does not add any fabrication steps, as both the bottom heaters have the same material and thickness and therefore can be fabricated using the same mask.

IV PROPOSED TEST INSTRUMENT

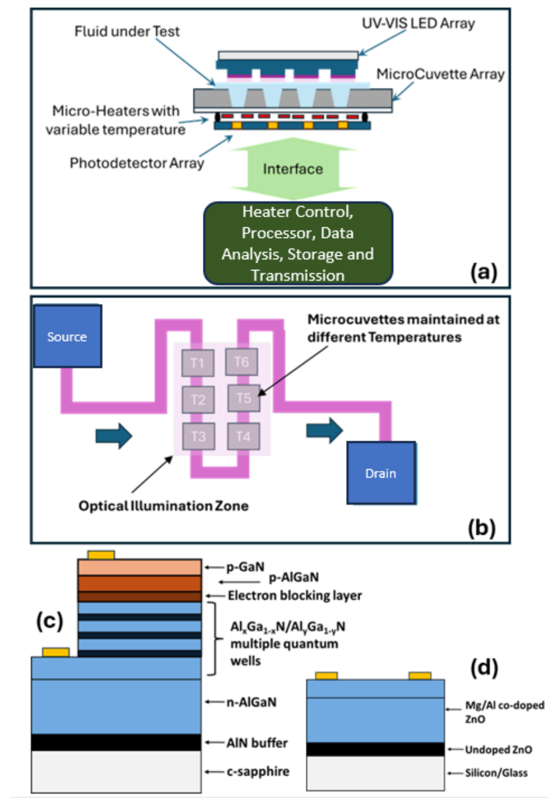

Fig.11. Schematic of side (a) and top view (b) of Proposed Test Instrument for field-testing of oils and other liquids. Schematic of fabricated AlGaN based UV-LEDs (c) and ZnO based detectors to be used in the system (d).

Based on the above results, it can be envisioned that with the appropriate design of the micro-heaters, uniform temperature can be maintained across the sidewall surfaces of the fluid inside the microcuvette. With additional design steps, it will be possible to design structures where with variation of the configuration of the microheaters, different fluid temperatures can be obtained for the same applied voltage. This opens the possibility that an array of such microcuvettes can be fabricated along with the surrounding microheaters, where application of the same external bias voltage will generate a temperature gradient along the chip, and the entire range of 70°C to 180°C can be obtained at various predesignated structures.

If light at specific wavelengths, such as 370nm and 470 nm, is made incident sequentially on the array chip, as shown in the schematic in Fig. 11, it will pass through the liquid present in the microcuvettes before being absorbed by the photodetector array placed underneath. Since the liquid is maintained at different temperatures along the array chip, scanning the photodetector signals will generate the temperature scans as shown in Fig.11(a) and 11(b). This setup allows fast measurements by avoiding temperature transients that may be difficult to minimize or eliminate during heating and cooling of the microcuvette to and from a specific target. Schematics of LED structures based on III-Nitride materials and Photodetector structures based on ZnO are shown in 11(c, d). Such devices have already been developed by our group and published previously [34, 35]. This will enable a fast and reliable test of the liquids, without necessitating expensive instrumentation or a laboratory setting. Furthermore, by changing the excitation wavelengths and temperature ranges, this setup can be made versatile and able to cater to a wide range of fluid test requirements.

V. Conclusion

In the first section of this paper, we report on the optical transmission spectroscopy results on various oils and determine the effect of thermal cycling on these properties. We identify that for extra-virgin olive oil, testing at two specific wavelengths of 370nm and 470nm allows the determination of the oxidation state as well as to distinguish this oil from other oils which are otherwise visually similar. We carry out this measurement using a test system employing glass milli-cuvette with 4µl volume capacity.

In the second part of the paper, we report on the design and simulation studies of a micro reactor for 100nl volume capacity with uniform heating facility. Specifically, uniform temperature has been achieved across the sidewalls of the microcuvette having a truncated inverted pyramidal structure. Our final design with three specially configured microheaters brings down the temperature difference to less than 8% along the sidewall of the cuvette. The average temperature was compatible with oxidation studies of olive oils, but can be varied depending on the applied voltages, catering to a wide range of applications.

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References

1. B. P. Vempatapu, D Tripathi, J Kumar, P. K. Kanaujia "Determination of kerosene as an adulterant in diesel through chromatography and high-resolution mass spectrometry." SN Applied Sciences, vol.1, pp.614-626,2019, https://doi.org/10.1007/s42452-019-0637-7
2. M. S. Rocha, J. R. Simes-Moreira "A simple impedance method for determining ethanol and regular gasoline mixtures mass contents," Fuel, vol. 84, pp. 447-452, 2005, doi: 10.1016/s0140-6701(05)82938-8.
3. T. Hofmann, K. Schrder, J. Zacheja, J. Binder "Fluid characterization using sensor elements based on interdigitated electrodes". Sensors Actuators B Chem., vol.37, pp.37–42, 1996, https://doi.org/10.1016/s0925-4005(96)01966-1
4. H. G. L. Coster, T. C. Chilcott, A. C. F. Coster "Impedance spectroscopy of interfaces, membranes and ultrastructures." Bioelectrochemistry and Bioenergetics, vol.40, pp.79-98, 1996, https://doi.org/10.1016/0302-4598(96)05064-7
5. J. Hwang, K. S. Kirkpatrick, T. O. Mason, E. J. Garboczi "Experimental limitations in impedance spectroscopy: Part IV. Electrode contact effects." Solid State Ionics. vol.98, pp.93–104, 1997, https://doi.org/10.1016/s0167-2738(97)00075-1
6. M. Didham, V. K. Truong, J. Chapman, D. Cozzolino "Sensing the Addition of Vegetable Oils to Olive Oil: The Ability of UV–VIS and MIR Spectroscopy Coupled with Chemometric Analysis." Food Analytical Methods. vol.13, pp.601–607, 2020, https://doi.org/10.1007/s12161-019-01680-8
7. J. N. Conceição, B. S. Marangoni, F. S. Michels, I. P. Oliveira, W. E. Passos, M. A. G. Trindade, S. L. Oliveira, A. R. L. Caires "Evaluation of molecular spectroscopy for predicting oxidative degradation of biodiesel and vegetable oil: Correlation analysis between acid value and UV–V is absorbance and fluorescence." Fuel Processing Technology. vol.183, pp.1–7, 2019, https://doi.org/10.1016/j.fuproc.2018.10.022
8. N. Vlachos, Y. Skopelitis, M. Psaroudaki, V. Konstantinidou, A. Chatzilazarou, E. Tegou, "Applications of Fourier transform-infrared spectroscopy to edible oils." Analytica Chimica Acta, vol. 573–574, pp. 459–465, 2006, https://doi.org/10.1016/j.aca.2006.05.034
9. M. Y. Talpur, S. T. H. Sherazi, S. A. Mahesar, A. A. Bhutto "A simplified UV spectrometric method for determination of peroxide value in thermally oxidized canola oil." Talanta. vol.80, pp.0–1826, 2010, https://doi.org/10.1016/j.talanta.2009.10.028
10. U. Oguz, O. Banu, T. Figen "Use of FTIR and UV–visible spectroscopy in determination of chemical characteristics of olive oils." Talanta. vol. 201, pp. 65-73, 2019, https://doi.org/10.1016/j.talanta.2019.03.116
11. Z. Xu, J. S. Godber, "Purification and Identification of Components of γ-Oryzanol in Rice Bran Oil." Journal of Agricultural and Food Chemistry. vol.47, pp.2724–2728, 1999, https://doi.org/10.1021/jf981175j
12. P. R. Gonçalves, H. P. Março, P. Valderrama. "Thermal edible oil evaluation by UV–Vis spectroscopy and chemometrics." Food Chemistry. vol.163, pp.83–86, 2014, https://doi.org/10.1016/j.foodchem.2014.04.109
13. A. Malvis, P. Šimon, T. Dubaj, A. Sládková, A. Ház, M. Jablonský, S. Sekretár, S. Schmidt, F. Kreps, Z. Burčová, G. Hodaifa, I. Šurina. "Determination of the Thermal Oxidation Stability and the Kinetic Parameters of Commercial Extra Virgin Olive Oils from Different Varieties." Journal of Chemistry, vol. 2019, pp. 1–8, 2019, https://doi.org/10.1155/2019/4567973
14. K. Ganesan, K. Sukalingam, B. Xu. "Impact of consumption and cooking manners of vegetable oils on cardiovascular diseases- A critical review." Trends in Food Science & Technology, vol.71, pp.132–154, 2018, https://doi.org/10.1016/j.tifs.2017.11.003
15. M. E. Khosroshahi "Effect of Temperature on Optical Properties of Vegetable Oils Using UV-Vis and Laser Fluorescence Spectroscopy." Optics and Photonics Journal, vol.8, pp.247-263, 2018, https://doi.org/10.4236/opj.2018.87021
16. Y Li, S Chen, H Chen, P Guo, T Li, Q Xu "Effect of thermal oxidation on detection of adulteration at low concentrations in extra virgin olive oil: Study based on laser-induced fluorescence spectroscopy combined with KPCA–LDA." Food Chemistry. vol.309,pp.125669,2020,https://doi.org/10.1016/j.foodchem.2019.125669
17. J. E. de Albuquerquea, L. H. C. Mattosob, R. M. Fariac, J. G. Mastersd, A. G. MacDiarmidd. "Study of the interconversion of polyaniline oxidation states by optical absorption spectroscopy."

Synthetic Metals. Vol.146, pp.0–10, 2004, https://doi.org /10.1016/j.synthmet.2004.05.019

18. Y. Wang, X. Jing "Effect of solution concentration on the UV–vis spectroscopy measured oxidation state of polyaniline base." Polymer Testing vol.24, pp.153–156, 2005, https://doi.org /10.1016/j.polymertesting.2004.09.013
19. A. Das, S. K. Mitra, A. Bhattacharyya "Determining the Composition of 100 Nano-Liter Fuel Mixture in a Silicon Micro-Cuvette Using Ultraviolet Absorption Spectroscopy." Journal of Microelectromechanical Systems. vol.30, pp.315-321,2021, https://doi.org /10.1109/JMEMS.2020.3047938
20. G. Velmathi, N. Ramshanker, S. Mohan. "Design, Electro-Thermal Simulation and Geometrical Optimization of Double Spiral Shaped Microheater on a Suspended Membrane for Gas Sensing."36th Annual Conference on IEEE Industrial Electronics Society. 2010, doi:10.1109/iecon.2010.5675550 10.1109/iecon.2010.5675550
21. G. Velmathi, N. Ramshanker, S. Mohan. "2D Simulations and Electro-Thermal Analysis of Micro-Heater Designs Using COMSOLTM for Gas Sensor Applications." COMSOL Conference 2010.
22. C. Moldovan, O. Nedelcu, P. Johander, I. Goenaga, D. Gomez, P. Petkov, K. Persson. "Ceramic Micro Heater Technology for Gas Sensors." International Semiconductor Conference.2006, doi:10.1109/smicnd.2006.283967.
23. S. Wu, Q. Lin, Y. Yuen, Y.C. Tai. "Mems Flow Sensors for Nano-Fluidic Applications." Proceedings IEEE Thirteenth Annual International Conference on Micro Electro Mechanical Systems. pp.745-750, 2000, doi:10.1109/memsys.2000.838611.
24. D. Briand, S. Colin, A. Gangadharaiah, E. Vela, P. Dubois, L. Thiery, N.F. de Rooij. "Micro-hotplates on polymide for sensors and actuators." Sensors and Actuators A: Physical. vol.132(1), pp.317-324,2006, https://doi.org/10.1016/j.sna.2006.06.003.
25. A.A. Giuliani, A. Cichellia, L. Tonuccib, N. d'Alessandroc, "Chlorophyll photosensitized oxidation of virgin olive oil: A comparison between selected unsaturated model esters and real oil samples". Rivista Italiana Delle Sostanze Grasse · January 2015.
26. Ramón Aparicio-Ruiz a, Beatriz Gandul-Rojas, "Decoloration kinetics of chlorophylls and carotenoids in virgin olive oil by autoxidation." Food Research International 65 (2014), pp.199–206, http://dx.doi.org/10.1016/j.foodres.2014.05.046.
27. J. S. Torrecilla, S. Vidal, R. A-Santos, S. C. Wang, J. C. Cancilla, "Spectroscopic determination of the photodegradation of monovarietal extra virgin olive oils and their binary mixtures through intelligent systems". Talanta, 144, 363–368. doi: 10.1016/j.talanta.2015.06.042.
28. O. S. Jolayemi, F. Tokatli, B. Ozen, "UV–Vis spectroscopy for the estimation of variety and chemical parameters of olive oils", Journal of Food Measurement and Characterization (2021) 15:4138–4149 https://doi.org/10.1007/s11694-021-00986-8.
29. P. P. Vachali, B. Li, B. M. Besch and P. S. Bernstein, "Protein-Flavonoid Interaction Studies by a Taylor Dispersion Surface Plasmon Resonance (SPR) Technique: A Novel Method to Assess Biomolecular Interactions". Biosensors 2016, 6, 6; doi:10.3390/bios6010006.
30. A Das, C Singha, A Bhattacharyya "Development of silicon microneedle arrays with spontaneously generated micro-cavity ring for transdermal drug delivery." Microelectronic Engineering. vol.210, pp.14–18, 2019, https://doi.org /10.1016/j.mee.2019.03.019
31. S. S. Srikanth, B. R. Kumar, V. Suresh, V. Jyothi. "Design and Simulation of Platinum Micro Heater for VOC sensing Applications." IJITEE, vol.9(2S3), pp145-148, 2019, doi: 10.35940/ijitee. B1038.1292S319.
32. L. Sujatha, V. S. Selvakumar, S. Aravind, R. Padamapriya, B. Preethi "Design and Analysis of Micro-Heaters using COMSOL Multiphysics For MEMS Based Gas Sensor." COMSOL Conference, Bangalore. 2012.
33. M. Horade, M. Kojima, K. Kamiyama, Y. Mae, T. Arai. "Development of a Novel 2-Dimensional Micro-Heater Array Device with Regional Selective Heating." Mechanical Engineering Research; vol.6(1), pp.66-74, 2016, http://dx.doi.org/10.5539/mer.v6n1p66.
34. P. G. Roy, S. Sen, A. Bhattacharyya, "Wavelength-switchable ultraviolet light-emitting diodes." Optics Letters, Vol. 48, No. 11 / 1 June 2023, https://doi.org/10.1364/OL.490036
35. A. Das, P.G. Roy, A. Dutta, S. Sen, P. Pramanik, D. Das, A. Banerjee, A. Bhattacharyya, Mg and Al co-doping of ZnO thin films: Effect on ultraviolet photoconductivity, Mater. Sci.Semicond. Process. 54, 36–41 (2016). https://doi.org/10.1016/j.mssp.2016.06.018

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