

Testing edible oil authenticity by using smartphone based spectrometer

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Abstract

In recent years, there has been an increasing interest in the classification of edible vegetable oils, examining authenticity and in detecting possible adulteration of high quality, expensive extra virgin olive oils with low-cost edible oils. Classical methods such as gas chromatography, liquid chromatography, Fourier transform infrared and nuclear magnetic resonance, mass spectrometry, and Raman spectroscopy have been widely applied to examine the authenticity of edible oils. Despite of their high sensitivity and accuracy, these methods are significantly expensive for daily life testing, especially in resource-poor regions. Furthermore, they are time-consuming as samples have to be analyzed in dedicated laboratories. In this paper, we propose a compact, low-cost, portable smartphone-based spectrometer for testing edible oil authenticity. Using simple laboratory optical components and a smartphone, we developed a compact spectrometer which can function in the wavelength range of 400–700 nm with the spectrum/pixel resolution of 0.334 nm/pixel. The images captured by the smartphone were converted into intensity distribution plots versus wavelength. As a proof of concept, the smartphone based spectrometer was utilized to measure the variations in fluorescent intensity of the mixed oils of expensive extra virgin olive oil and low-cost rice oil with different percentages. The results obtained the spectrometer were in good agreement with that from a laboratory spectrometer, thus, confirmed its adequate sensitivity and accuracy. Due to the cost effectiveness, the adequate sensitivity, and the portability, the smartphone based spectrometer can be applied in numerous applications such as in-field testing, lifestyle monitoring, and home diagnostics.

Keywords: spectroscopy, fluorescence and luminescence, image processing, sensors, smartphone.

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Introduction

Extra virgin olive oil (EVOO) is the most valuable and health promoting among edible vegetable oils, and thus is more expensive. This makes it susceptible to be adulterated with less expensive oils in order to increase profits. For example, some refineries mix EVOO with low-cost oils distilled from soybean, sunflower, corn, or rice, yet still label the final product as EVOO. Others even go so far as to mix vegetable oils with beta carotene and chlorophyll in order to produce completely fake olive oil that they then label as authentic. Therefore, the adulteration of EVOO is considered a major issue of the edible oil industry, which undermines the confidence of consumers and also decreases the profit of honest refineries. For this reason, effective analytical techniques to identify the quality and authenticity of EVOOs are always of high demand.

To deter oil adulteration, numerous studies have been carried out in order to develop new rapid and precise methods for quality assessment of oil products, including: gas chromatography [1], liquid chromatography [2], Fourier transform infrared spectroscopy [3], and nuclear magnetic resonance [4], mass spectrometry [5], Raman spec-

troscopy [6], and fluorescent spectroscopy [7–11]. Among these methods, fluorescent spectroscopy is a rapid, sensitive, non-destructive, and relatively simple method for characterizing edible oils without the need for pre-treatment of the sample. In fact, it has been widely used to classify, and analyze edible oils. For example, Mu et al. used 532-nm-laser-induced fluorescence combined with support vector machine to characterize and distinguish the difference between several edible oils from soybean, olive, grapeseed, rapeseed, corn, peanut, sunflower, canola, and walnut [9]. Kongbonga group, on the other hand, recorded the fluorescent spectra of several vegetable oils at excitation wavelength of 370 nm, and noticed a distinction between the refined and unrefined oils. They also denoted the possibility to detect alteration of some coveted oils such as argan and EVOOs [10]. In order to identify different vegetable oils, Nikolova et al. proposed a quick fluorescent method to analyze edible oil contents such as fatty acid, tocopherol, beta-carotene, and chlorophyll [11]. They showed that depending on the types of oils, the intensity of the fluorescent peaks originating from the oil contents varies significantly. However, despite the potential in providing detailed constituent analysis, the applications of fluo-

rescent method in oil industry and in daily life are still very limited due to the available fluorescent devices are still bulky and expensive. Therefore, a low-cost, portable, sensitive and rapid detection device for edible oil authentication is always of high interest.

In recent years, the increasing popularity of smart phone with internet connectivity, high-resolution cameras, touch-screen displays, and high-performance CPUs has resulted in the rapid development of new smartphone-based devices. In comparison with laboratory devices, a smartphone-based device has significant advantages in term of low-cost, portability, ease of use, and flexibility for everyday applications, especially in resource-poor regions. As a result, many research groups have actively engaged in developing or converting smartphones into optical sensing devices such as optical microscopes [12], spectroscopy [13–15], surface plasmon resonance biosensors [16, 17], crystal integrated label-free biosensors [18, 19], blood glucose monitors [20], or pH sensors [21, 22]. These devices have been applied for food quality analysis, for diagnosing disease, for monitoring of nutritional status and water quality, or for determining the presence of environmental contaminants.

An important advantage of smartphones is their compactness and accessibility. It should be noted that recently in hyperspectral equipment there has also been a trend towards reduction and lightening of devices, including through the use of diffractive optics [23-27]. Taking into account new effects arising from the use of diffractive optical elements new hyperspectral image processing methods are proposed in the scientific school of V.A. Soifer [28-34].

Although being low cost, and having small size, smartphone based devices still maintain an adequate accuracy which can be comparable with that of laboratory devices. For example, Long et al. developed smartphone based instruments to measure Enzyme Linked Assays (ELISA) with identical or better detection limit than conventional ELISA microplate reader [35]. Yu et al., on the other hand, reported a quantitative analysis of pharmaceutical compounds using a mobile phone thin-layer chromatography analyzer [36]. Accordingly, their smartphone based instrument can provide measurements that are equivalent to those obtained with a lab-based desktop thin-layer chromatography densitometer. Recently, researchers at the Sandia National Laboratories have developed a smartphone-based diagnostic device that can detect Zika and other mosquito-borne viruses in 30 minutes or less [37]. Such studies have opened up the possibility of replacing expensive laboratory testing equipment with low-cost, portable smartphone based devices. In the field of identifying food authentication, to the best of our knowledge, there has not been any smartphone based device for testing edible oil authenticity.

In this study, we present a low-cost, portable, sensitive smartphone based spectrometer for testing edible oil authenticity. Our spectrometer includes an entrance slit, a single lens, a diffraction grating, and a smartphone. This

CMOS camera is a wavelength-independent photon collector that can function as a detector. The images captured by the spectrometer are then converted into intensity distribution plots versus wavelength. The cradle held the smartphone and the optical elements were made by 3D printing. As a proof of concept for testing edible oil authenticity, we measured the variation in fluorescent intensity of mixed oils of EVOO and rice oil with different mixing percentages. The obtained results were then compared with that of a conventional laboratory spectrometer to determine the accuracy, and the sensitivity of our spectrometer.

1. Material and methods

1.1. Oil samples

Samples of commercially EVOO and rice oil were purchased from supermarket and kept at room temperature in the dark before use. The measurements were performed immediately after opening the bottles to prevent sample oxidation. Every data was averaged from three times measurements to ensure the accuracy of the measurement. Fluorescence spectra were recorded without pretreatment and dilution of the samples. The measurement time of 1 minute was fixed for all the measurements.

To examine the effects of EVOO adulteration, six samples of different dilution percentages of EVOO and refined rice oil were prepared by weighing as shown in Table 1. The mixed oils were then stored in a transparent quartz cuvette for fluorescent measurements. All sample were prepared with fixed volume of 3 ml.

Table 1. Samples with different mixing percentages *s* of EVOO and rice oil (ranging from 16.67% to 100%)

Sample	Percentage of olive oil	Percentage of rice oil
S1	100 %	0 %
S2	83.33 %	16.67 %
S3	66.67 %	33.34 %
S4	50.00 %	50.00 %
S5	33.34 %	66.67 %
S6	16.67 %	83.33 %

1.2. Optical setup

The smartphone-based spectrometer was designed to interface with an iPhone 5s smartphone by the Apple Inc., of which the camera can function as a digital light detector. A polylactic acid (PLA) plastic cradle was printed by a 3D imprint machine with the resolution of 0.05 mm. It was installed to hold all the optics including a cuvette holder, an entrance slit (50 μm, Thorlab), a collimating lens (focal length of 50 mm, Thorlab), and a diffraction grating (1300 grooves/mm, Edmund Optics). It is robust and can exclude light from external sources. On top of the cradle, the smartphone camera was fixed firmly. For fluorescent measurement, a laser pointer with the wavelength of 405 nm was used to illuminate the sample cuvette. Only fluorescent light collected at 90 degrees with respect to the incident beam was allowed to pass through the entrance slit. Light that entered the optical chamber was then colli-

mated by the collimating lens, and guided to the diffraction grating. The diffraction grating, aligned at an angle of ~ 47 degrees with respect to the fluorescent beam so as only the first-order diffracted light was directed onto the CMOS camera (8 MP, 3264×2448 pixels) of the smartphone. The captured images were then analyzed and converted into intensity distribution plots versus wavelength using ImageJ software. Due to the spectral responsibility of the Si-based sensor and internal infrared cut-off filters within the camera optics, the spectrometer can function in a wavelength range from 400 nm to 700 nm. A schematic diagram of our designed detection system is shown in Fig. 1.

Prior to studying the sensor characteristics, the pixel information of the captured fluorescent spectrum was calibrated in wavelength scale. To do this, we followed established procedure for calibrating smartphone based optical devices [13]. In detail, the entrance slit was illuminated by two lasers: HeCd and HeNe laser with known wavelength of 442 nm and 532.8 nm respectively. The known wavelengths of the two lasers and their wavelength separation were used to set the wavelength span corresponding to the pixel scale along the illumination direction. In this work, the wavelength span of 190.8 nm corresponds to 571 pixels, leads to a spectrum/pixel resolution of 0.334 nm/pixel. In comparison with other works on smartphone based devices, our smartphone based spectrometer exhibits compatible spectrum/pixel resolution [13, 19, 22].

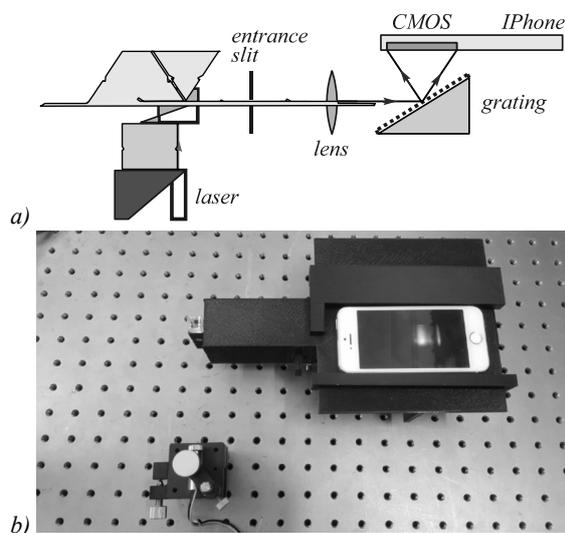


Fig. 1. (a) The model and (b) the assembled setup of the smartphone based spectrometer

2. Results and discussion

To study the adulteration effect, low-cost rice oil was chosen to mix with EVOO. Such selection was due to the fact that rice oil can be dissolved completely in EVOO. Additionally, the fluorescent emissions of both rice and EVOO are in visible range which fit well to the sensing range of our spectrometer. Generally speaking, the fluorescence of edible oils can be grouped in three regions associated to the emission of i) oxidation products (region from 400 to 500 nm); ii) tocopherol or vitamin E (region

from 500 to 650 nm); and iii) chlorophyll (region from 650 to 780 nm). The fluorescence spectra of 100% EVOO and 100% rice oil measured by a laboratory spectrometer (AvaSpec-ULS2048, Avantes) are presented in Fig. 2. The sample were excited by 405 nm wavelength laser pointer. The laboratory spectrometer's detector is a CCD having a spectral resolution of 0.5 nm. Similarly with previous published reports, fluorescent spectrum of EVOO has a strong peak at 675 nm which demonstrates the present of chlorophyll. The fluorescent emission of rice oil, on the other hand, exhibits a broad, high intensity band from 400 nm to 650 nm which is attributed to the fluorescent emission of tocopherol or vitamin E. The 675 nm peak does not appear in the rice oil's spectrum. This may come from the fact that the used rice oil is refined oil which was normally experienced a heating process during making product. Due to the heating, chlorophyll was diluted, thus, leads to the disappearance of 675 nm peak.

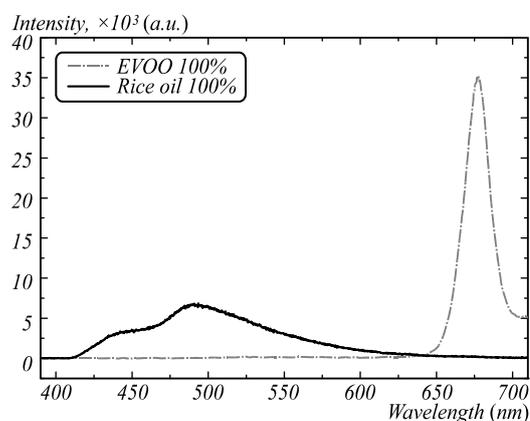


Fig. 2. Fluorescent spectra of 100% EVOO and 100% rice oil measured by a laboratory spectrometer

Fig. 3 demonstrates the fluorescent spectra of the mixed oils taken by the laboratory spectrometer. As the concentration of EVOO decrease from 100% to 16.67%, a gradual drop of intensity at 675 nm is also observed. As the high peak intensity of chlorophyll emission is usually used to distinguish EVOO with other edible oils, this again confirms the ability of the fluorescent spectroscopy in detecting the adulteration of EVOO with other edible oils. In accordance with the intensity drop at 675 nm the band shape changes with respect to the oil percentage variation is also recorded.

The fluorescence spectra of the same mixed oils taken from our smartphone spectrometer are presented in Fig. 4. As seen in the figure, our spectrometer exhibited similar fluorescent responses in comparison with that of the laboratory spectrometer. We also observed a clear quenching at 675 nm with respect to the decrease of EVOO percentages. A change from the typical band shape of EVOO to the band shape of rice oil was also noticeable. The high intensity in the range of 400 nm to 500 nm can be attributed to the high sensitivity of the CMOS in this range. Similarly, a valley appeared at the wavelength of 575 nm was due to the low sensitivity of

the CMOS at that wavelength, which results in an intensity drop [38].

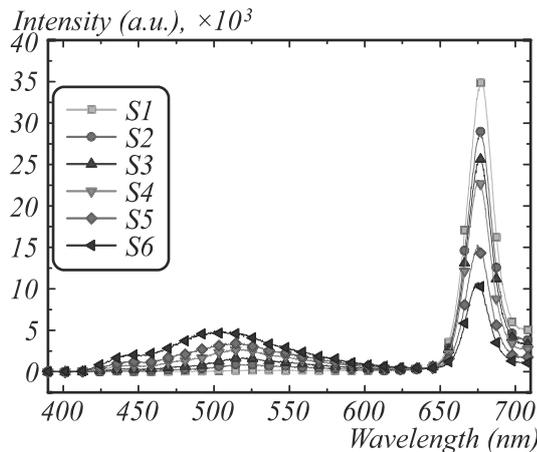


Fig. 3. Fluorescent spectra of the six samples of different dilution percentages of EVOO and refined rice oil (ranging from 16.67% to 100%) taken by the laboratory spectrometer

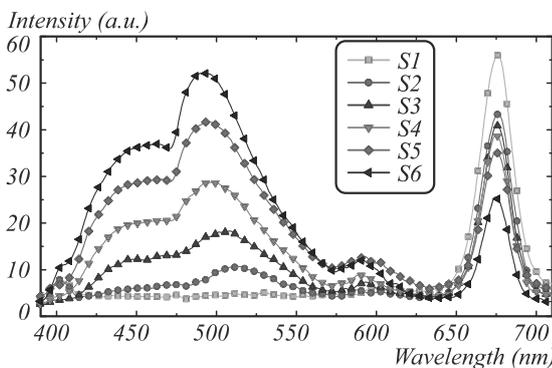


Fig. 4. Fluorescent spectra of the six samples of different dilution percentages of EVOO and refined rice oil (ranging from 16.67% to 100%) taken by the smartphone based spectrometer

In order to evaluate the accuracy and the sensitivity of the smartphone based spectrometer, calibration curves which demonstrate the dependence of peak intensities at 675 nm as a function of the EVOO concentrations were taken into account (Fig. 5). The slopes of the two calibration curves which demonstrate the sensitivity of the two spectrometers are of 0.3 for our smartphone based spectrometer and of 0.29 for our Avantes laboratory spectrometer. The linear correlations (R^2) are relatively relevant of 0.91 and of 0.97. The error of the laboratory spectrometer is of 3%, while the error of the smartphone based spectrometer is 7%. The correlation between the percentage of EVOO in a mixed oil solution measured by the laboratory spectrometer and by the smartphone based spectrometer is shown in Fig. 6. These positive agreements confirmed the sensitivity, the accuracy of our smartphone based spectrometer. In addition, the smartphone based spectrometer was made from inexpensive components. It is also compact and portable, making it suitable for safety food inspection and in-field testing.

Conclusions

We have developed a low-cost, accurate, and portable smartphone based spectrometer for testing edible oil authenticity. The spectrometer uses a laser pointer as the light source, a CMOS camera of a smartphone as the detector, and a grating as the dispersive unit. The spectrum/pixel resolution is 0.334 nm/pixel. As a proof of concept, we utilized this smartphone based spectrometer to measure the variations in fluorescent intensity of mixed oils with different percentages of an expensive EVOO and a low-cost, refined rice oil. Experiment results showed that if the percentage of EVOO decreased, a linear quenching in fluorescent intensity at wavelength of 675 nm occurred, thus indicated the reduced amount of chlorophyll. The smartphone based spectrometer showed a comparable sensitivity with that of a laboratory spectrometer. We believe that smartphone based spectrometers can be effectively applied in numerous applications such as in-field testing, lifestyle monitoring, and home diagnostics.

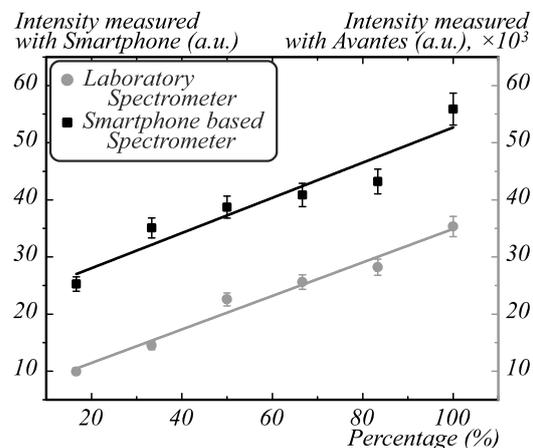


Fig. 5. Calibration curves demonstrating the dependence of peak intensity at 675 nm as a function of EVOO percentages taken from the smartphone based spectrometer and from the laboratory spectrometer

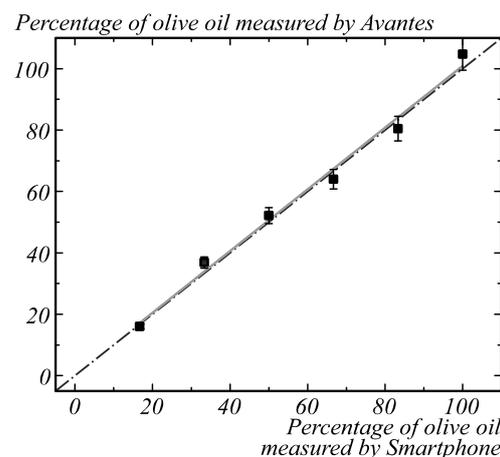


Fig. 6. Correlation of the EVOO concentrations as measured by the smartphone based spectrometer and by the laboratory spectrometer

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