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## Stimulated Brillouin scattering in combination with visible absorption spectroscopy for authentication of vegetable oils and detection of olive oil adulteration



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### ABSTRACT

Vegetable oils provide high nutritional value in the human diet. Specifically, extra virgin olive oil (EVOO) possesses a higher price than that of other vegetable oils. Adulteration of pure EVOO with other types of vegetable oils has attracted increasing attentions. In this work, a stimulated Brillouin scattering (SBS) combined with visible absorption spectroscopy method is proposed for authentication of vegetable oils and detection of olive oil adulteration. The results provided here have demonstrated that the different vegetable oils and adulteration oils exhibit significant differences in normalized absorbance values of two relevant wavelengths (455 and 670 nm) and frequency shifts of SBS. The normalized absorbance values of all spectra at the two relevant wavelengths of 670 nm and 455 nm linearly decrease with the increase of the adulteration concentration. The Brillouin frequency shifts exponentially increase with the increase of the adulteration concentration. Due to non-destructive and requiring no sample pretreatment procedure, this method can be effectively employed for authentication and detection of oils adulteration.

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### 1. Introduction

Edible vegetable oils are widely used in cooking at home and in the food industry, and are one of the important foodstuffs in our daily life. Vegetable oils provide some important components for human nutrition and health benefits that including but not limited to essential fatty acids (EFA), vitamins, minerals, and so on [1–3]. In China, vegetable oils are the primary cooking oil, especially the edible oilseed oils, such as soybean, rapeseed, peanut, and tea-seed oils have recently become more popular than traditional animal fats [4,5]. Compared with the common seed oils, olive oil is greatly appreciated by consumers and gourmets owing to its unique taste and aroma as well as the high nutritional value [6,7]. In recent years, the consumption of high-grade olive oil is becoming more and more popular and possesses a potential huge market in China. With the increasing consumption demand for edible vegetable oils, the adulteration is quite prevalent. The vegetable oils are frequent subjects of fraud in which a relatively expensive oil is substituted

with cheap or inferior quality oils. Because of its sensory quality and nutritional benefits olive oil is often adulterated with less expensive oils such as soybean, rapeseed, peanut, sunflower and corn oils [8–10]. It is an emphasis of great concern on determining the authenticity of olive oil by consumers or local regulatory authorities since these frauds can disturb the market order and harm the economic benefit of consumers. Except for the economic fraud, adulteration of olive oil can bring potential threats to consumer health. Therefore, it is desirable to develop rapid and simple methods to authenticate the vegetable oils and to detect the adulteration of olive oil.

The edible oils widely employed in olive oil adulteration in China can be olive oil blended with other edible vegetable oils in a definite proportion. The most common adulterant oils used for olive oil are seed oils, such as sunflower, soybean, corn, peanut and rapeseed oils. The diversity of oil on the market has made it necessary to develop reliable analytical methods for authenticating the olive oil from other types of vegetable oils and detecting the adulteration. Therefore, there still has been an increasing interest in the effective analytical methods for authenticating the vegetable oils and detecting possible adulteration of olive oil, and numerous research and review articles have been published with regards to the analysis of olive oil [11–17].

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The traditional methods are based on the determination and quantification of free acid radicals and fatty acid composition through wet chemical or chromatographic analysis for authenticating the categories of vegetable oil or adulterants [18,19]. For example, high-performance liquid chromatography (HPLC) method can sensitively detect the adulteration of vegetable oils [20,21]; Gas chromatography-mass spectrometer (GC-MS) has also been successfully applied in the oils authentication and adulteration [22–24]. These traditional methods are usually used as an official detection method. However, they have several obvious drawbacks such as time-consuming, complicated sample pretreatments, hazardous chemical residues, and highly trained professionals.

Developments in detection technology have undoubtedly led to greater success in the fight against adulteration over the years, but it is also an inevitable problem that the same developments of adulteration have also been used by some insincere persons to invalidate the usefulness of official methods. In recent years, spectroscopy techniques provide an alternative to the traditional methods and they have increased in importance in vegetable oils authenticating. In this respect, a lot of methods have been proposed based on Raman spectroscopy [25,26], fluorescence spectroscopy [27,28], mid-infrared spectroscopy [9], nuclear magnetic resonance spectrometry [29,30], Fourier transform near-infrared spectroscopy [31,32]. These spectroscopic methods now have the potential to replace or at least complement the traditional methods. Stimulated Brillouin scattering has been used in many fields [33–37]. However, to the best of our knowledge, no articles have been reported on stimulated Brillouin scattering combined with visible absorption spectroscopy used as a technique for discriminating edible vegetable oils or authenticating vegetable oil adulteration.

SBS is an inelastic scattering process arising from the fluctuation in density or acoustic phonons in the medium [38]. The changes of Brillouin frequency shift are closely related to the medium properties such as density, refractive index, and acoustic velocity, and so on. The aim of the work described here is to develop a new method for the authentication of vegetable oils and the detection of olive oil adulteration by carrying out visible absorption spectroscopy on different oil samples in combination with SBS spectrum. In the following, we present and discuss our results.

## 2. Samples and Methods

### 2.1. Sample Description

In order to examine the applicability of SBS combined with visible absorption spectroscopy for authentication of vegetable oils and detection of olive oil adulteration, four types of Chinese edible seed oils (rapeseed, soybean, peanut, and sunflower oils) and extra virgin olive oil (EVOO) were selected as the experimental samples. Four types of oilseed oils were produced by a famous producer of edible oil in China, and EVOO was an import commodity produced by a Spanish company. The oil samples used in this work were purchased as commercial products from a local supermarket in Nanchang, China. All the selected oil samples were labeled with the label of non-genetically modified organism (non-GMO), and the botanical origin and quality were guaranteed by the manufacturers and the franchisers. In order to maintain realistic testing conditions, all the oil samples were stored in the dark at the room temperature until the time of measurement.

### 2.2. Visible Absorption Spectra

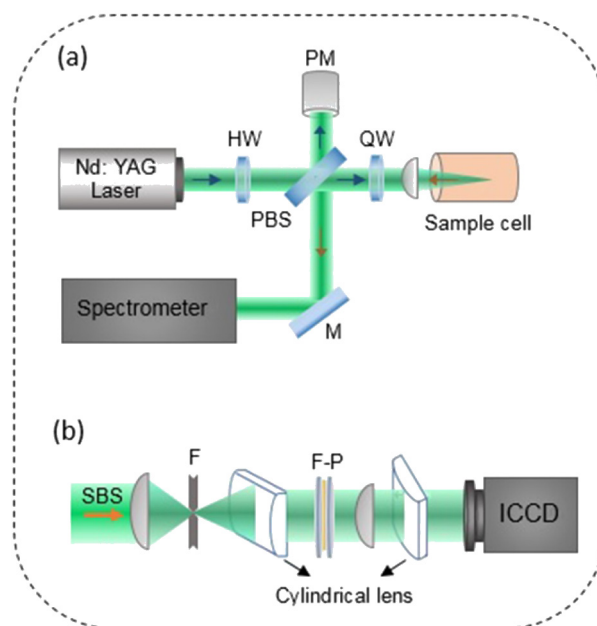
All the visible absorption spectra were measured by employing an UV-VIS-NIR scanning spectrophotometer (SHIMADZU Solidspec-3700) with the optimum resolution of 0.1 nm. The spectral region

ranges from 320 to 750 nm. Each experimental sample was put in a quadrature optical cuvette with 10 mm path-length for corresponding absorption spectra measurement. In order to maintain the comparability, the spectrophotometer was set in the same scan speed, same sampling interval, and same integration time. Each spectrum was routinely measured at least five times to reduce as much as possible the potential operational errors and sample deterioration. Finally, the collected absorption spectra of different oil samples were analyzed by a computer.

### 2.3. Experimental Apparatus for Measuring SBS Spectrum

SBS spectra were measured by employing the optical schematic shown in Fig. 1. The laser source used was an injection-seeded Nd:YAG laser with a linewidth of 90 MHz and 30 GHz obtained by switching a seed laser on and off, respectively. The laser source possesses the following parameters: repetition frequency 10 Hz, pulse duration  $\sim 7$  ns (full width at half maximum, FWHM), Gaussian spatial profile with  $1/e^2$  diameter of 12 mm, divergence angle  $\sim 0.45$  mrad. The output laser beams from the laser source with the vertical polarization were focused by an achromatic lens with focal length of 60 mm into a sample cell filled with the oil sample after passing through half-wave plate (HW), PBS, and quarter-wave plate (QW) in turn. All SBS spectra were taken in the  $180^\circ$  backscattering refractive geometry and collected into the spectrometer. The laser energy was monitored by using the power meter (PM).

Because of the Brillouin shift is the order of GHz and the signal intensity of SBS of oil samples is too weak, it is difficult to extract the Brillouin spectrum by a conventional spectrometer. In order to circumvent this problem, we developed a spectrometer system based on Fabry-Perot (FP) etalon and cylindrical lenses, as shown in Fig. 1b. In the spectrometer, the circular interference spectra of backward scattering signals formed by the F-P etalon with a free spectral range (FSR) of 20.1 GHz. The energy distributed on the whole circle spectrum was concentrated



**Fig. 1.** Experimental setup for measuring the SBS spectra of different oil samples. HW and QW denote the half-wave plate and quarter-wave plate, respectively; PBS is the polarization beam splitter, PM is the power meter, F is the filter, and F-P is the Fabry-Perot etalon.

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