Т. 89, № 1 **ЖУРНАЛ ПРИКЛАДНОЙ СПЕКТРОСКОПИИ** ЯНВАРЬ — ФЕВРАЛЬ 2022

V. 89, N 1 **JOURNAL OF APPLIED SPECTROSCOPY** JANUARY — FEBRUARY 2022

DETECTION OF MULTIPLE-HEATED EDIBLE OIL BASED ON FLUORESCENCE SPECTROSCOPY AND LIFETIME METHOD[**]

M. Chen*, W. Ma, L. Shi, Y. Lai, M. Wang, X. Wang

Shanghai Institute for Interventional medical devices,
Key Laboratory of Medical Optical Technology and Instrumentation
of Ministry of Education, University of Shanghai for Science and Technology,
Shanghai, China, e-mail: cmhui.43@163.com

This study analyzed the fluorescence intensity, integrated intensity of the spectral area, and fluorescence peak of vegetable oil samples subjected to different heating conditions. The photon lifetime of the vegetable oil was then calculated using the fluorescence lifetime method, which can determine whether the sample has been repeatedly heated at high temperatures. Combining these two methods improves the accuracy of detection and can help solve the problem of false and missed detection, providing a strong basis for the detection of high-temperature heated vegetable oil.

Keywords: *fluorescence spectroscopy, integral intensity, fluorescence lifetime, high-temperature heated vegetable oil.*

ОБНАРУЖЕНИЕ МНОГОКРАТНОГО НАГРЕВА ПИЩЕВОГО МАСЛА МЕТОДАМИ ФЛУОРЕСЦЕНТНОЙ СПЕКТРОСКОПИИ И ОПРЕДЕЛЕНИЯ ВРЕМЕНИ ЖИЗНИ ФЛУОРЕСЦЕНЦИИ

M. Chen*, W. Ma, L. Shi, Y. Lai, M. Wang, X. Wang

УДК 535.372

Шанхайский университет науки и техники,
Шанхай, Китай, e-mail: cmhui.43@163.com

(Поступила 21 декабря 2020)

Проанализированы интенсивность флуоресценции, интегральная интенсивность спектральной области и максимум флуоресценции образцов растительного масла, подвергнутых нагреву в различных условиях. По времени жизни флуоресценции рассчитано время жизни фотонов растительного масла, с помощью которого можно определить, подвергался ли образец многократному нагреву при высоких температурах. Сочетание методов повышает точность обнаружения многократного нагрева, а также решает проблему ложного и пропущенного обнаружения, обеспечивая основу для обнаружения нагретого до высокой температуры растительного масла.

Ключевые слова: *спектроскопия флуоресценции, интегральная интенсивность, время жизни флуоресценции, высокотемпературный нагрев растительного масла.*

Introduction. Some restaurants in Asian and African countries, especially China, use multiple-heated waste cooking oil, which can damage human health, instead of vegetable oil. Therefore, the rapid and accurate detection of multiple-heated vegetable oil is important. Fluorescence spectroscopy and fluorescence lifetime are used to detect vegetable oil repeatedly heated at high temperatures. Vegetable oils subjected to different heating conditions have significantly different fluorescence spectra with fingerprint specificity. Therefore, fluorescence spectroscopy can be used to detect vegetable oil samples. Vegetable oils are triglyceride-based oils derived from plants, with examples including cooking oils such as sunflower oil. Esters formed from

---

[**] Full text is published in JAS V. 89, No. 1 (http://springer.com/journal/10812) and in electronic version of ZhPS V. 89, No. 1 (http://www.elibrary.ru/title_about.asp?id=7318; sales@elibrary.ru).

straight-chain higher fatty acids and glycerin are the main components of vegetable oil. Other components include vitamins E and K, calcium, iron, phosphorus, potassium, and other minerals that help maintain elemental balance in the body. However, edible vegetable oil undergoes overheating and oxidation after repeated high-temperature heating, with unsaturated fatty acids and vitamins in vegetable oil exhibiting oxidative instability. After repeated high-temperature heating, a series of chemical reactions occur in vegetable oil, namely, oxidation, polymerization, isomerization, cyclization, and hydrolysis, that destroy the unsaturated fatty acids and vitamins. This not only reduces the nutritional value of edible oil but can also produce carcinogens as a serious threat to consumer health [1–3]. To reduce costs, some unscrupulous vendors repeatedly heat vegetable oil used for frying at high temperatures, which can damage consumer health. Therefore, identifying a convenient, fast, and green method for detecting vegetable oil heated at high temperatures multiple times is of great importance.

The detection of multiple high-temperature heated vegetable oil using fluorescence spectroscopy and fluorescence lifetime methods has received little research attention. Wu et al. [4] measured the fluorescence spectra of 42 oil samples, determining the characteristic excitation wavelengths of vegetable oil and a matrix analysis model. Comprehensive analysis of the contour spectrum and characteristic emission spectrum of vegetable oil in a specific range, by evaluating its fluorescence spectrum characteristics and matrix model, confirmed that the fluorescence spectroscopy and matrix analysis methods were effective.

Li et al. [5] used linseed oil, soybean oil, rapeseed oil, and sunflower oil as raw materials to measure and analyze synchronous fluorescence spectra and changes in fluorescent substances under different temperature conditions. The results showed that synchronous fluorescence spectrum peak changes in the four vegetable oils at different temperatures were mainly concentrated in the region of 300–415 nm, with fluctuations observed with extended heating time. Fluorescence peaks were present under heating and oxidation conditions of 50 and 150°C. Obvious fluctuations were observed, with different rates of change in the fluorescence peak intensity observed, and different oils giving quite different results. Changes in these peaks were related to changes in the fluorescent substances and degree of oil oxidation during heating. These characteristic changes in the synchronous fluorescence spectrum can be used to monitor oil oxidation.

In the experiment, fluorescence spectral data of multiple high-temperature heated and unheated sunflower oils were initially collected using a steady-state fluorescence spectrometer, with Origin2020b software used to analyze the emission and excitation spectra, allowing multiple high-temperature heating to be detected from changes in the fluorescence spectrum intensity. The vegetable oil was heated, and variations in the integrated intensity of the spectral area of the vegetable oil sample and the full width at half maximum of the fluorescence peak were evaluated simultaneously. Time-resolved fluorescence spectra of the sunflower oils subjected to different heating conditions were then obtained using a transient fluorescence spectrometer. The data were imported into Origin2020b for analysis, and the fluorescence lifetime of the fluorescent photons was calculated. Changes in the fluorescence lifetime were used to detect multiple high-temperature heating. The fluorescence spectrum analysis method has advantages of comprehensive sample information, good reproducibility, and small sampling volume [6], but is only qualitative. Therefore, misdetection and leakage are likely to occur for small changes in fluorescence intensity. In contrast, the fluorescence lifetime analysis method detects high-temperature heated oil by calculating the lifetime of fluorescent photons in the heated vegetable oil and comparing it with the fluorescence lifetime of the unheated oil. This is a quantitative analysis that provides more accurate detection results [7–9]. Combining both of these techniques improves the experiment reliability, making detection more accurate and reliable and providing a powerful reference for the detection of high-temperature heated oil.

Materials and methods. *Analytical methods.* The fluorescence spectra refer to the three dimensions of fluorescence intensity, excitation wavelength (λ_{ex}), and fluorescence emission wavelength (λ_{em}). This mainly reflects changes in fluorescence spectrum intensity with changes in excitation wavelength and emission wavelength. The fluorescence spectra contain rich information, with good selectivity and high sensitivity [10, 11]. After repeated high-temperature heating of the vegetable oil, the fluorescence peak at fixed excitation and emission wavelengths changes. According to changes in the fluorescence spectrum peak information, the vegetable oil can be heated multiple times at high temperatures and under different heating conditions and with fixed excitation. Changes in the integrated intensity of the fluorescence peak spectral area at a wavelength showed a certain trend.

Vegetable oils contain high concentrations of unsaturated fatty acids and vitamin E. Vitamin E is an effective antioxidant for protecting unsaturated fatty acids and other easily oxidizable substances. When irradiated with excitation light, these substances absorb energy in different transition states. When irradiation is stopped, electrons are in an excited state. The time taken to transition to the ground state is different for each substance, resulting in different fluorescence lifetimes. Data obtained from the experiment were fitted using Origin2020b software, and the fluorescence lifetimes of multiple high-temperature heated and unheated vegetable oils were calculated. Accordingly, multiple high-temperature heated vegetable oil was detected.

The fluorescence lifetime measurement method collects sample data using the time-dependent single-photon counting method. This method excites the sample using a light pulse with as short as possible a duration and, under single-photon counting conditions, at a certain instant. The probability of a photon emitted at a time being the first photon to reach the detector in a certain time interval is proportional to the instantaneous photon emission intensity. A fluorescence intensity decay curve can be obtained through multiple repeat measurements, allowing the fluorescence lifetime to be calculated [12].

Sample preparation. Pure-bred oils were used as edible oil samples instead of blended oils. First-grade pressed JinLongYu sunflower oil was selected as the experimental sample. The average contents of unsaturated fatty acids, linoleic acid, and vitamin E were up to 87, 67%, and 55 mg/100 g, respectively, with trace amounts of cholesterol, carbohydrates, metal ions, and other minerals detected. The samples were divided into four test groups, as follows: (i) unheated vegetable oil; (ii) vegetable oil heated at 95℃ for 10 repeat cycles; (iii) vegetable oil heated at 95°C for 20 repeat cycles; and (iv) vegetable oil heated at 95°C for 30 repeat cycles. Each heating cycle was conducted by heating to 95°C, holding for 10 min and then rapidly cooling to 20°C, then holding for 5 min. These steps were repeated to achieve the desired number of cycles, with each cycle taking about 15 min. The heating cycle was controlled using a PCR amplifier.

Instruments. Fluorescence spectra were recorded using a QuantMaster 40 steady-state fluorescence spectrometer and its supporting software. Fluorescence lifetime measurements were performed using a Time Master 3 transient fluorescence spectrometer provided by Photon Technology International. This instrument adopts advanced flash frequency time-sharing measurement and nonlinear time-scale data acquisition technologies and has advantages of fast measurement speed, high accuracy, high sensitivity, practicality, convenience, and flexible preparation, providing the fastest measurement speed currently available. This fast advance fluorescence lifetime measurement system can detect the lifetime of 7-pm fluorescein, withthea shortest measurement lifetime of 100 ps.

The experiment parameters of the QuantMaster 40 fluorescence spectrometer were set as follows: Excitation wavelength range, 310–420 nm; emission wavelength range, 315–580 nm; excitation slit width, 1.6 nm; emission slit width, 2.0 nm; step length, 1 nm; scanning rate, 1200 nm/min.

The experiment parameters of the Time Master 3 transient fluorescence spectrometer were set as follows: excitation wavelength of 337 nm and emission wavelength of 380 nm for multiple heated vegetable oil; excitation wavelength of 337 nm and emission wavelength of 430 nm for unheated vegetable oil.

Data processing. A QuantMaster 40 fluorescence spectrometer was used to detect four vegetable oil samples subjected to different heating conditions. The excitation wavelength range was 310–420 nm, and the emission wavelength range was 315–580 nm. After setting these parameters, the vegetable oil sample was placed into the fluorescence spectrophotometer for detection, with the obtained detection data imported into Origin2020b software and converted into a matrix. From the matrix data, an intensity change curve was constructed for the fluorescence spectrum of vegetable oil at a fixed excitation wavelength and fixed emission wavelength under different heating conditions, and the fluorescence spectrum at a fixed excitation wavelength was calculated for comparison. Change trends in the peak area integral intensity and full width at half maximum of the fluorescence spectrum peak were then evaluated.

The three groups of vegetable oil subjected to different heating cycles were placed into the transient fluorescence spectrometer for detection using the set parameters, the detection data were imported into Origin2020b software for data processing, and fluorescence attenuation curves were drawn for all samples. Each group was calculated in Origin according to the original data fluorescence lifetime of the vegetable oil samples.

Results and discussion. *Fluorescence spectrum analysis.* From the fluorescence spectrum matrix data, unheated vegetable oil exhibited the strongest fluorescence intensity at λ_{ex} = 359 nm and λ_{em} = 368 nm. Figure 1a shows the fluorescence spectrum intensity of vegetable oils subjected to different heating conditions at λ_{ex} = 359 nm. The fluorescence intensity curve under different heating conditions contained only one fluorescence peak, namely, the characteristic peak of vitamin E. Figure 1b shows the change curve of the fluorescence spectrum intensity at λ_{em} = 368 nm, in which the fluorescence intensity decreased with increasing number of heating cycles.

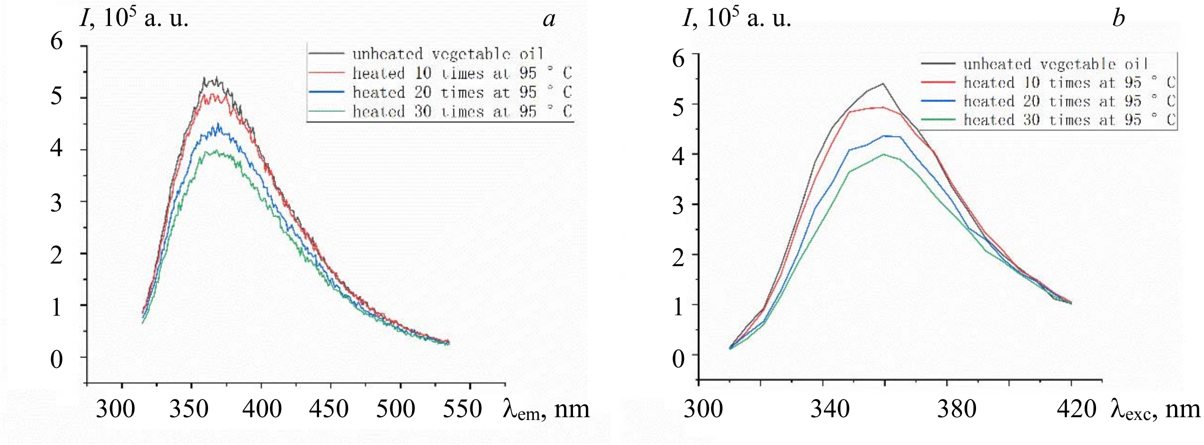

Fig. 1. Fluorescence spectra under different heating conditions: a) $\lambda_{ex} = 359$ nm; b) $\lambda_{em} = 368$ nm.

Fluorescence spectrum peak area integral intensity and full width at half maximum. Figure 2a shows the fluorescence spectrum of unheated vegetable oil at $\lambda_{ex} = 359$ nm. The fluorescence intensity was strongest at $\lambda_{ex} = 359$ nm and $\lambda_{em} = 368$ nm. The integral intensity of the fluorescence spectrum peak area was calculated at $\lambda_{ex} = 359$ nm. Figure 2b shows the fluorescence spectrum intensity of vegetable oil heated 10 times at $\lambda_{ex} = 357$ nm. The maximum fluorescence peak of the vegetable oil sample appeared at $\lambda_{ex} = 357$ nm and $\lambda_{em} = 367$ nm. The intensity of the fluorescence peak was 5% less than that of the unheated vegetable oil. This was due to substances in the vegetable oil undergoing a series of complex chemical reactions, such as oxidation and polymerization, during heating that destroy fluorescent components and vitamins, resulting in fluorescence quenching.

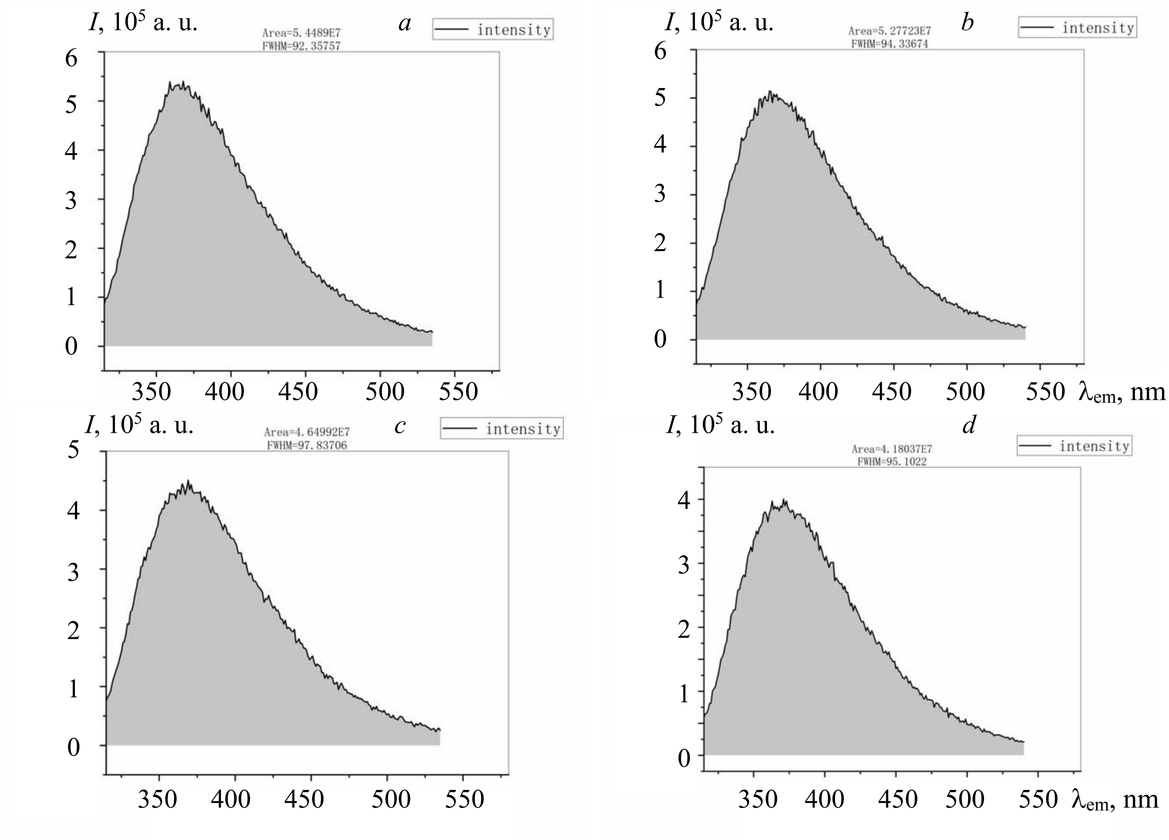

Fig. 2. Fluorescence spectrum of vegetable oils: a) unheated, $\lambda_{ex} = 359$ nm; b) heated 10 times, $\lambda_{ex} = 357$ nm; c) heated 20 times, $\lambda_{ex} = 360$ nm; d) heated 30 times, $\lambda_{ex} = 360$ nm.

Figure 2c shows the fluorescence spectrum intensity of vegetable oil heated 20 times at 95°C for λ_{ex} = 360 nm. The fluorescence peak appeared at λ_{ex} = 360 nm and λ_{em} =369 nm. Compared with the position of the fluorescence peak in the unheated oil sample, both the excitation and emission wavelengths were slightly redshifted by 1 nm, indicating that cyclic heating under these experimental conditions did not change the main fluorescent substances in the vegetable oil. Compared with unheated vegetable oil, the fluorescence intensity had decreased by 16%, indicating that the fluorescent substances in vegetable oil decreased as the number of heating cycles was increased. Figure 2d shows the fluorescence spectrum of vegetable oil heated 30 times at 95°C under λ_{ex} = 360 nm. The fluorescence peak appeared at λ_{ex} = 360 nm and λ_{em} = 368 nm. Compared with unheated vegetable oil, the fluorescence peak position was redshifted by 1 nm, the emission wavelength was not shifted, and the fluorescence intensity was 27% lower. As the number of heating cycles increased, the fluorescence peak intensity of the vegetable oil decreased. No new fluorescence peak was observed, indicating that no new fluorescent substances were produced during heating cycles under these experimental conditions, with the fluorescence intensity only decreasing. Therefore, comparison of fluorescence intensity can be used to detect multiple high-temperature heated vegetable oil.

Table 1 shows fluorescence peak positions and relative fluorescence intensity information for the four sample groups. The fluorescence intensity of the unheated sample was set to 1, and the percentage fluorescence intensity of the other samples was compared with the unheated sample. Compared with the characteristic peak of the unheated vegetable oil, samples heated 10, 20, and 30 times showed excitation wavelengths with an average redshift of 0, 2, and 3 nm, and emission wavelengths with an average redshift of 1, 2, and 2 nm, respectively, while the fluorescence peak location was unchanged. As the number of heating cycles increased, the fluorescence intensity of the characteristic peaks decreased, with the three heating groups exhibiting averages decreases of 10, 17, and 25%, respectively, compared with the unheated vegetable oil. However, the excitation and emission wavelengths corresponding to the fluorescence characteristic peaks showed only small redshifts, which might be due to the heating temperature being insufficiently high to fry food.

TABLE 1. Fluorescence Peak Position and Relative Fluorescence Intensity

Condition	λ_{ex}, nm			λ_{em}, nm			Relative fluorescence intensity		
	Expt. 1	Expt. 2	Average	Expt. 1	Expt. 2	Average	Expt. 1	Expt. 2	Average
Unheated	359	353	356	368	362	365	Normalized	Normalized	Normalized
Heated 10 times	357	355	356	367	365	366	−5%	−14%	−10%
Heated 10 times	360	356	358	369	365	367	−16%	−18%	−17%
Heated 10 times	360	358	359	368	366	367	−27%	−23%	−25%

TABLE 2. Integral Intensity of Fluorescence Peak Spectral Area

Condition	Integrated intensity of fluorescence area			Relative integrated intensity of fluorescence area		
	Expt. 1	Expt. 2	Average	Expt. 1	Expt. 2	Average
Unheated	5.45×10^{7}	5.41×10^{7}	5.43×10^{7}	Normalized	Normalized	Normalized
Heated 10 times	5.28×10^{7}	5.27×10^{7}	5.275×10^{7}	−3.1%	−2.6%	−2.9%
Heated 20 times	4.65×10^{7}	4.62×10^{7}	4.635×10^{7}	−14.7%	−14.6%	−14.6%
Heated 30 times	4.18×10^{7}	4.14×10^{7}	4.16×10^{7}	−23.3%	−23.4%	−23.4%

Table 2 shows the integrated intensity of the fluorescence peak area for the four sample groups at a fixed excitation wavelength. The integrated intensity of the fluorescence peak area for the unheated sample was set as 1 for comparison. The integrated intensity of the fluorescence peak area for the three heating groups decreased with increasing number of heating cycles, with average decreases of 2.9, 14.6, and 23.4%, respectively, compared with unheated vegetable oil. The total fluorescence intensity at a fixed excitation wavelength showed a downward trend. The full width at half maximum of the fluorescence peaks of the four sample groups was obtained using the integral intensity map of fluorescence peak area, as 92.36, 94.34, 97.84, and 95.1, respectively. From the experimental data, the full width at half maximum of the fluorescence peak showed no obvious change under different conditions.

The decrease in fluorescence intensity was due to multiple heating cycles destroying fluorescent substance vitamin E in the vegetable oil through decomposition and oxidation, gradually generating a nonrigid planar product, with weak or no fluorescence. As the number of heating cycles increased, the amount of this product gradually increased, resulting in a gradual decrease in the fluorescence peak. The position of the fluorescence peak remained unchanged, and no new fluorescence peak was produced, indicating that simple heating at 95°C did not change the main fluorescent substances in the vegetable oil nor produce new fluorescent substances.

Fluorescence lifetime method. The experimental data processing results are divided into four groups, as shown in Fig. 3. The time-resolved fluorescence spectra and fitting results of the unheated sunflower oil sample under excitation at 337 nm and emission at 430 nm are shown in Fig. 3a. The three-exponential decay method was used to fit the time-resolved fluorescence spectrum data. The goodness of fit (R^2) was 0.989, which is close to 1, indicating a good fit. The average fluorescence lifetime (t) of the fluorescence attenuating substance can be calculated using the equation

$$\tau = \frac{A_1 t_1^2 + A_2 t_2^2 + A_3 t_3^2}{A_1 t_1 + A_2 t_2 + A_3 t_3}, \tag{1}$$

where t_1, t_2, and t_3 are the fitting lifetimes, and A_1, A_2, and A_3 are the corresponding proportions of the fluorescence lifetimes. From the fitting equation, A_1, A_2, A_3 and t_1, t_2, and t_3 were obtained and substituted into Eq. (1) to obtain $t = 3.25$ ns.

The time-resolved fluorescence spectra and fitting results for the sunflower oil sample heated 10 times at 95°C, measured at λ_{ex} = 337 nm and λ_{em} =380 nm, are shown in Fig. 3b. The three-exponential decay method was used to fit the time-resolved fluorescence spectrum data. The R^2 value was 0.992, which is close to 1, indicating a good fit. From the fitting equation, A_1, A_2, A_3 and t_1, t_2, and t_3 were obtained and substituted into Eq. (1) to obtain $t = 2.62$ ns.

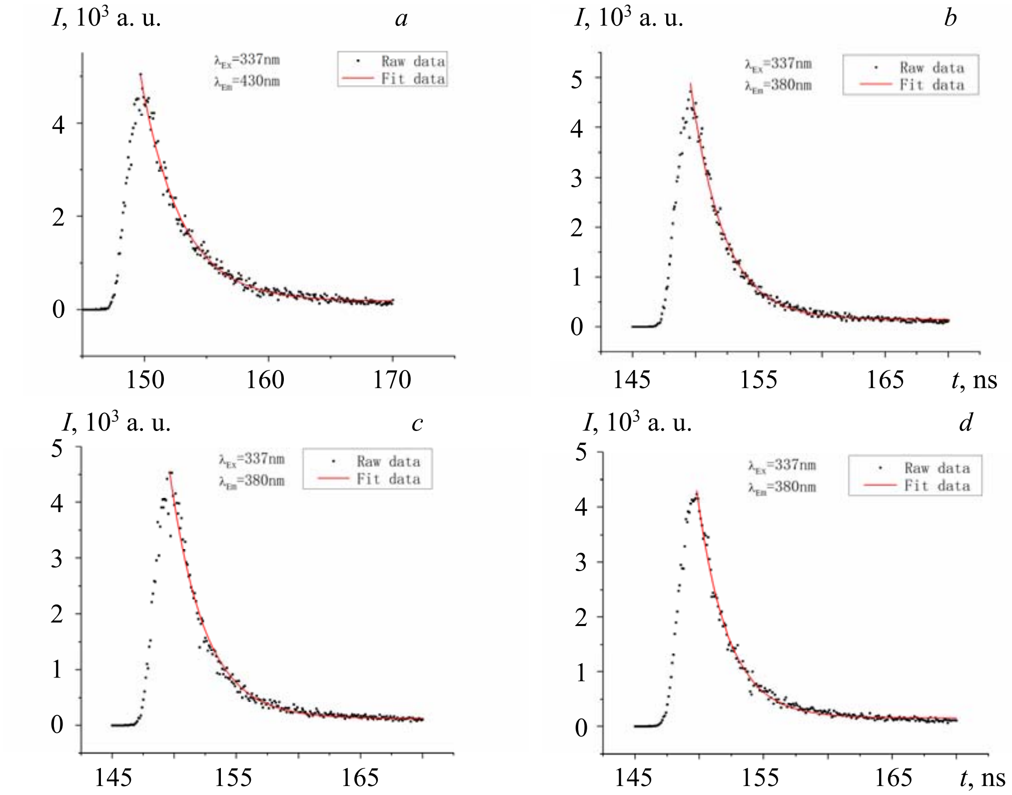

Fig. 3. Time-resolved spectra and fitting results for vegetable oil sample: a) unheated; b) heated 10 times; c) heated 20 times; d) heated 30 times.

The time-resolved fluorescence spectra and fitting results for the sunflower oil sample heated 20 times at 95°C, measured at λ_{ex} = 337 nm and λ_{em} = 380 nm, are shown in Fig.3c. The three-exponential decay method was used to fit the time-resolved fluorescence spectrum data. The R^2 value was 0.986, which is close to 1, indicating a good fit. From the fitting equation, A_1, A_2, A_3 and t_1, t_2, and t_3 were obtained and substituted into Eq. (1) to obtain $t = 2.64$ ns.

The time-resolved fluorescence spectra and fitting results of the sunflower oil sample heated 30 times at 95°C, measured at λ_{ex} = 337 nm and λ_{em} = 380 nm, are shown in Fig. 3d. The three-exponential decay method was used to fit the time-resolved fluorescence spectrum data. The R^2 value was 0.992, which is close to 1, indicating a good fit. From the fitting equation, A_1, A_2, A_3, and t_1, t_2, and t_3 were obtained and substituted into Eq. (1) to obtain $t = 2.41$ ns.

After the fluorescence lifetime output of the above four sample groups was processed, they were subjected to three repeated tests, with the resulting measured average lifetimes shown in Table 3.

TABLE 3. Calculated Average Lifetime Results

Condition	$\lambda_{ex\ 337\ nm}/\lambda_{em\ 380\ nm}$			Fit (R^2)		
	Expt. 1, ns	Expt. 2, ns	Average, ns	Expt. 1	Expt. 2	Average
Unheated	3.25±0.02	3.33±0.02	3.29±0.02	0.989	0.973	0.981
Heated 10 times	2.62±0.02	2.76±0.02	2.69±0.02	0.992	0.981	0.987
Heated 20 times	2.64±0.03	2.66±0.03	2.65±0.03	0.986	0.979	0.983
Heated 30 times	2.41±0.03	2.64±0.03	2.53±0.03	0.992	0.974	0.983

Table 3 shows that the R^2 values of all samples were around 0.98, indicating that the fitting results were good. The fluorescence lifetimes of unheated vegetable oil were all greater than 3 ns, while the fluorescence lifetimes of the heated vegetable oil samples were lower, at less than 3 ns, with most around 2.62 ns. The fluorescence lifetime of the vegetable oil sample heated 10 times was similar to that of the vegetable oil sample heated 20 times. The fluorescence lifetime of the vegetable oil sample heated 30 times in Experiment 1 was slightly shorter than those of the samples heated 10 and 20 times. This might be due to the high temperature or experimental error. The experimental results showed that the fluorescence lifetime of vegetable oil was reduced by repeated high-temperature heating but did not easily distinguish oils heated 10, 20, and 30 times. The fluorescence lifetime method has higher sensitivity and accuracy than the fluorescence spectroscopy method and requires a smaller sample amount. The test results of the fluorescence lifetime method are more accurate and reliable than those of fluorescence spectrometry when there is no significant change in fluorescence intensity.

Conclusions. This study used two analysis methods to detect vegetable oil heated repeatedly at high temperatures. First, fluorescence spectroscopy was used for detection by comparing the fluorescence peaks at fixed excitation and emission wavelengths of high-temperature heated vegetable oil and unheated vegetable oil. The fluorescence integral area and full width at half maximum of the fluorescence peak were also measured under a fixed excitation wavelength. With increasing number of heating cycles, the integral intensity of the fluorescence spectrum of vegetable oil gradually decreased, while the full width at half maximum of the fluorescence peak did not change significantly. Second, the fluorescence lifetime method was used to detect the lifetime of fluorescent photons in vegetable oil samples by comparing the results for oils before and after high-temperature heating. The fluorescence lifetime of vegetable oil was concluded to decrease after repeated high-temperature heating. The fluorescence spectroscopy method intuitively and comprehensively reflects changes in the fluorescence intensity of vegetable oil samples before and after high-temperature heating, and involves relatively simple data processing, making it a reliable method for vegetable oils exhibiting large differences in fluorescence intensity before and after heating. However, if the fluorescence intensity change is small, incorrect or missed detection can occur. The fluorescence lifetime method quantitatively analyzes differences in the fluorescence lifetime of vegetable oil samples before and after high-temperature heating, giving highly accurate and reliable test results. The combination of these two methods provides a strong basis for accurate and reliable detection of high-temperature heated vegetable oil and reduces missed and false detections.

Acknowledgments. This work was supported by the National Natural Science Foundation of China (Grant No. 61308115), and a Shanghai Science and Technology Commission Project (Grant No. 15DZ1940400).

We thank Simon Partridge, PhD, from Edanz Group (https://en-author-services.edanz.com/ac) for editing a draft of this manuscript.

REFERENCES

1. L. Xue, *Study on the Composition and Distribution of Characteristic Nutrients of Edible Vegetable Oil [D]*, Beijing, Graduate School of Chinese Academy of Agricultural Sciences (2018).
2. Y. Wen, Y. Liu, L. Wang, et al. *China Oil Fats*, **42**, No. 3, 35–39 (2017).
3. A. M. Giuffrè, M. Capocasale, C. Zappia, et al. *J. Oleo Sci.*, **66**, No. 11, 1193–1205 (2017).
4. X. Wu, R. Tian, M. Sun, et al. *Spectrosc. Spectral Anal.*, **36**, No. 7, 2155–2161 (2016).
5. Y. Li, Y. Gu, T. Gong, et al. *Agric. Prod. Process*, No. 3, 14 (2020).
6. H. Gu, Y. Sun, S. Liu, et al. *Food Anal. Methods*, **11**, No. 12, 3464–3470 (2018).
7. B. Chen, L. Guo, H. Chen, et al. *Mod. Food Sci. Technol.*, No. 6, 44 (2017).
8. H. Chen, B. Chen, D. Lu. *Sci. Rep.*, **8**, No. 1, 1–6 (2018).
9. M. Saleem, N. Ahmad, H. Ali, et al. *Laser Phys.*, **27**, No. 12, 125602 (2017).
10. J. Xu, X. F. Liu, Y. T. Wang. *Food Chem.*, **212**, 72–77 (2016).
11. J. Cao, C. Li, R. Liu, et al. *Food Anal. Methods*, **10**, No. 3, 649–658 (2017).
12. J. R. Lazzari-Dean, A. M. M. Gest, E. W. Miller. *Elife*, **8**, e44522 (2019).