American Journal of Analytical Chemistry, 2015, 6, 395-401
Published Online April 2015 in SciRes. http://www.scirp.org/journal/ajac
http://dx.doi.org/10.4236/ajac.2015.65038

Application of Two-Dimensional Correlation UV-Vis Spectroscopy in Chinese Liquor Moutai Discrimination

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Received 21 February 2015; accepted 27 March 2015; published 31 March 2015

Abstract

Chinese liquor Moutai is the "National alcoholic drink" in China and plays a very important role of social activities in Chinese people's life. In pursuit of high profits, some illegal counterfeit Moutai liquors have begun to appear in the market. Therefore, it is an urgent need for new techniques to discriminate the genuine and counterfeit Moutai liquor. In this work, the conventional Ultraviolet-Visible (UV-Vis) spectroscopy and two-dimensional correlation UV-Vis spectroscopy are applied to obtain the UV-Vis characteristic of Moutai liquor and counterfeit one, respectively. The experimental results reveal that the conventional UV-Vis spectra of the genuine and counterfeit Moutai liquor are similar. However, the two-dimensional correlation UV-Vis spectra of them are different and this method would be applied to differentiate the counterfeit Moutai liquor from the genuine Moutai liquor. Compared with conventional methods, this novel method has the advantages of easy operation, simple instrumentation and direct recognition, which make it a potential tool in the fields of food safety.

Keywords

Two-Dimensional Correlation Spectra, UV-Vis Spectroscopy, Moutai Liquor, Discrimination

1. Introduction

Chinese liquor is one of the oldest distilled spirits in the world and plays a significant role in Chinese people's

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How to cite this paper: Zhang, Z.Y., Jiang, J.Y., Wang, G.X., Wang, H., Liu, J. and Wang, H.Y. (2015) Application of Two-Dimensional Correlation UV-Vis Spectroscopy in Chinese Liquor Moutai Discrimination. *American Journal of Analytical Chemistry*, **6**, 395-401. http://dx.doi.org/10.4236/ajac.2015.65038

life [1]. The Chinese liquor Moutai (or Maotai) is the most well-known Chinese liquor, owing to its long history of distillation, excellent and unique qualities and complex production processes [2]. It is usually fermented from grains, mainly involving sorghum and wheat, and after several months or years of fermentation, the liquor is distilled out with steam. Then the fresh distillates are aged for several months or years in order to balance the different aromatic compounds, and finally, the aged liquors are diluted with water and blended into the products [3]-[5]. Additionally, based on the unique geographical environment of Moutai town in China, the Moutai liquor owns the unusual fragrance [6], and it has ever won the gold medal at Panama Pan-world Fair in 1915 [7]. Right now it has been dubbed the title of the "National alcoholic drink" in China and has great demands in more than one hundred countries in the world [8] [9]. Under the tremendous economic temptation, some illegal traders start using counterfeit Moutai liquors to cheat the customers. Hence, it is highly required to authenticate the Moutai liquor [10]. However, the most common detection strategy for analysis of Chinese liquors is integrity by component approach, such as gas chromatography, mass spectrometry and gas chromatograph-mass spectrometry [11]-[14]. These techniques enable identification and quantification of hundreds of compounds of Chinese liquors, but the experimental processes are time-consuming and the instruments are expensive and large-scale, which limit their practical applications [15]-[17]. Therefore, the quality management of Chinese liquors still mainly depends on a traditional technique, by hiring the oenophiles to taste the aroma and flavor of Chinese liquors and identify their differences [18]. But the accuracy and objectivity cannot be always guaranteed because oenophiles may be affected by their own healthy status, feeling and external environment. Especially, counterfeit liquors, which may be produced by mixing industrial alcohol with water, are not suitable for tasting and discriminating by oenophiles [19].

In recent years, the rapid development has been gained in the applications of spectroscopic techniques in Chinese liquors discrimination. Particularly, the Infrared (IR) spectroscopy is usually employed to identify the Chinese liquors owing to its simple pretreatment for a sample [20]. However, the water in the liquor also has an IR adsorption peak which may interfere with the IR results of the sample. Ultraviolet-Visible (UV-Vis) spectroscopy and two-dimensional correlation spectroscopy could be used as another kind of tool for identification and discrimination of Chinese liquors, and up to now, there have been still few studies on this. The two-dimensional correlation spectroscopy method was developed by Noda *et al*. [21]-[23], and it has become a very useful analytical technique for complex mixture systems, because the technique is based on a series of perturbation-induced dynamic spectra and a simple scheme of correlation analysis, then generates clearly and easily identifiable graphical representation of spectral fingerprints [24]-[26]. Herein, we evaluate and identify the genuine Moutai liquor and counterfeit Moutai liquor by using the two-dimensional correlation UV-Vis spectroscopy technique.

2. Experimental

2.1. Reagents and Instruments

Absolute ethanol and sodium hydroxide were purchased from Xilong Chemical Co., Ltd. (Shantou, China). Moutai liquor was obtained from Kweichow Moutai Co., Ltd. (Guizhou, China). Counterfeit Moutai liquor was obtained from China National Research Institute of Food and Fermentation Industries. Double distilled water was used throughout.

The UV-Visible absorption data were recorded from a UV-Vis spectrometer, T6 New century, Purkinje General Instrument Co., Ltd. (Beijing, China).

2.2. Methods

2.2.1. Sodium Hydroxide Concentration Optimization

2 mL liquor sample was added into a glass of bottle, then different concentrations of 400 μL sodium hydroxide solution (0.2625 mol/L, 0.4375 mol/L, 0.875 mol/L, 1.3125 mol/L, 1.75 mol/L, 2.1875 mol/L, 2.625 mol/L, 3.0625 mol/L, 3.5 mol/L) was added to each bottle, respectively. Next, the mixture solution was left to react in an oven at 70˚C for 4 h. At last, the UV-Vis spectrum of the reaction solution was collected by a UV-Vis spectrometer, respectively.

2.2.2. UV-Vis Spectroscopy

2 mL liquor sample was added into a glass of bottle, then 400 μL sodium hydroxide solution (1.75 mol/L) was

added to the liquor, and the mixture solution was left to react in an oven at 70˚C, Next, the UV-Vis spectrum of the reaction solution was collected once by a UV-Vis spectrometer every 30 min for 4 h or 5 min for 1 h. Those collected spectra were used as the dynamic spectra in the next two-dimensional correlation analysis.

2.2.3. Two-Dimensional UV-Vis Correlation Spectroscopy

The graphs of two-dimensional correlation UV-Vis spectra are obtained by the treatment of the series of UV-Vis dynamic spectra with the software "2D-shige", which is being developed by Shigeaki Morita in Professor Yukihiro Ozaki's group at Kwansei-Gakuin University. In the two-dimensional correlation analysis, the average spectrum is used as the reference spectrum, the positive and negative correlation intensities are denoted by the unshaded regions and shaded regions [27].

3. Results and Discussion

The sample of Chinese liquor Moutai is a complex mixture system, which usually contains hundreds of fragrant and flavor compounds [28]. Further studies reveal that the complexity of Chinese liquor Moutai in the chemical classes is mainly including alcohols, esters, acids, aldehydes, ketones, phenols and nitrogen-heterocyclic and so on [29] [30]. Hence, it is difficult to well identify the quality of Moutai liquor by using the current commercial separation and analysis techniques. In contrast, the spectrum detection technique is expected to provide a new testing strategy [31]. **Figure 1** shows the UV-Vis spectra of the Moutai liquor and counterfeit Moutai liquor. By comparing their UV-Vis spectra, the similar absorption peaks of them at 278 nm were shown in the results, which were mainly attributed to absorption of the furfural molecule [32]. The difference of them was only reflected on the absorption intensity, thus it is uneasy to get the extract useful information for directly identification of the samples from the conventional UV-Vis results.

Recently, the two-dimensional correlation spectroscopy technique has been developed because it can improve the resolution of a spectrum through the difference in responses of various molecular groups of a system to a given external perturbation [33]-[35]. Besides, it can provide some new information, which cannot be obtained from the conventional spectrum. Therefore, there is a huge advantage by using two-dimensional correlation spectroscopy to identify and discriminate the complex system [36]. In this paper, two-dimensional correlation UV-Vis spectra were obtained based on the sodium hydroxide induced chemical reaction. Firstly, the UV-Vis spectra of sample's mixture solution during the reaction process were measured as the dynamic spectra. Subsequently, the collected spectra were converted into a two-dimensional correlation UV-Vis spectrum. It has been reported that the Moutai liquor could occur color reaction at 70˚C for 4 h under the alkaline solutions [37]. As shown in the **Figure 2**, different concentration of sodium hydroxide solutions were mixed with Moutai liquor, respectively, and the corresponding reaction results were measured by a UV-Vis spectrometer. The results display that the UV-Vis adsorption spectra is red-shift with the increase of sodium hydroxide solution, and when the amount of the added sodium hydroxide solution reached 1.75 mol/L, the reaction tends to be stable.

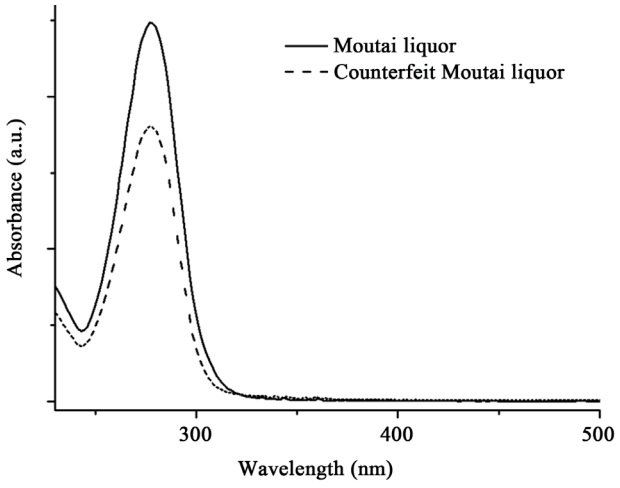

Figure 1. UV-Vis spectra of Moutai liquor (solid line) and counterfeit Moutai liquor (dash line).

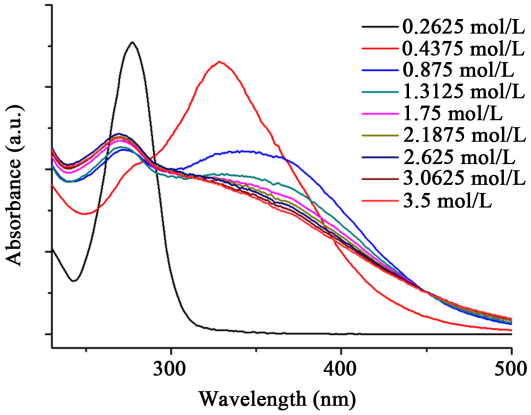

Figure 2. UV-Vis spectra of Moutai liquor with different concentration sodium hydroxide solutions.

Figure 3 shows the dynamic UV-Vis spectra and two-dimensional correlation synchronous spectra of Moutai liquor and counterfeit Mourtai liquor in the wavelength range of 250 - 500 nm. As shown in the conventional UV-Vis spectra (**Figure 3(a)** and **Figure 3(c)**), which are collected of the react system under the alkaline solutions every 30 min for 4 h, it can be found that the absorption peaks of both of Moutai liquor and counterfeit Moutai liquor have a similar shift trend, but the changed peaks are around 350 nm and 330 nm, respectively. The spectra change of the reaction (color change of Moutai liquor under the alkaline condition) was mainly induced by the organic compounds including furfural, diacetyl, acetoin, and 2,3-diketone, etc. It has been reported that most of these compounds contained carbonyl or conjugate π bond, after heating under alkaline condition, intramolecular electron transfer and rearrangement of them would form cross conjugated system and further prolong π bond. Plus, heterocyclic compound in the liquor such as furan and pyran would form multiple electron conjugated system for multi π aromatic heterocycles, so that the reaction results display apparent spectra changes finally [38]-[40].

From the two-dimensional correlation UV-Vis spectra, the minimal spectra changes (color change of Moutai liquor under the alkaline condition) have been expanded, which is directly shown in the **Figure 3(b)** and **Figure 3(d)**, respectively. In the two-dimensional correlation graphs unshaded regions indicate positive correlation signs, while shaded regions indicate negative correlation signs. The Moutai liquor has one strong auto-peak at 345 nm and another auto-peak at 278 nm with positive intensities, and a pair of cross peaks at (278 nm, 345 nm) and (345 nm, 278 nm) with negative intensities. For the counterfeit Moutai liquor, it has one strong auto-peak at 328 nm and an auto-peak at 278 nm with positive intensities, and a pair of cross-peak at (278 nm, 328 nm) and (328 nm, 278 nm) with negative intensities. Additionally, the positive range of them is in 300 - 460 nm and 300 - 410 nm, respectively. Hence, by using such different two-dimensional correlation UV-Vis spectra, a new interesting tool for the discrimination of counterfeit Moutai liquor from genuine Moutai liquor was developed.

Furthermore, the dynamic UV-Vis spectra in the range of 0 - 30 min and 0 - 1 h were evaluated in this work, respectively, as shown in **Figure 4**. For the conventional UV-Vis spectra (**Figure 4(a)** and **Figure 4(d)**), the absorption spectra of Moutai liquor were changed rapider than the counterfeit Moutai liquor at the same condition, and the red-shift trend was similar as above mentioned in the **Figure 3(a)** and **Figure 3(c)**, but their two-dimensional correlation spectra are significantly different. Specifically, **Figure 4(b)** and **Figure 4(e)** (t = 0 - 30 min) have the similar differences as the above mentioned in **Figure 3(b)** and **Figure 3(d)**. However, the **Figure 4(c)** and **Figure 4(f)** (t = 0 - 1 h) display new differences. Moutai liquor has a unique auto-peak around 345 nm and another auto-peak at 277 nm with positive intensities in the **Figure 4(c)**, the cross peaks of Moutai liquor were situated at (277 nm, 333 nm) and (333 nm, 277 nm) with negative intensities, respectively. For counterfeit Moutai liquor (**Figure 4(f)**), there are two obvious auto-peaks at 277 nm and 328 nm with positive intensities, and a pair of cross peaks at (277 nm, 328 nm) and (328 nm, 277 nm) with negative intensities. The peak positions in the two-dimensional correlation UV-Vis spectra of counterfeit Moutai liquor are different from genuine Moutai liquor. In addition, 0 - 1 h could be chose as the optimum reaction time in this work. These characteristics of the two-dimensional correlation UV-Vis spectra are visually so evident, so that this method has the potential in directly used for the quality analysis of Moutai liquor.

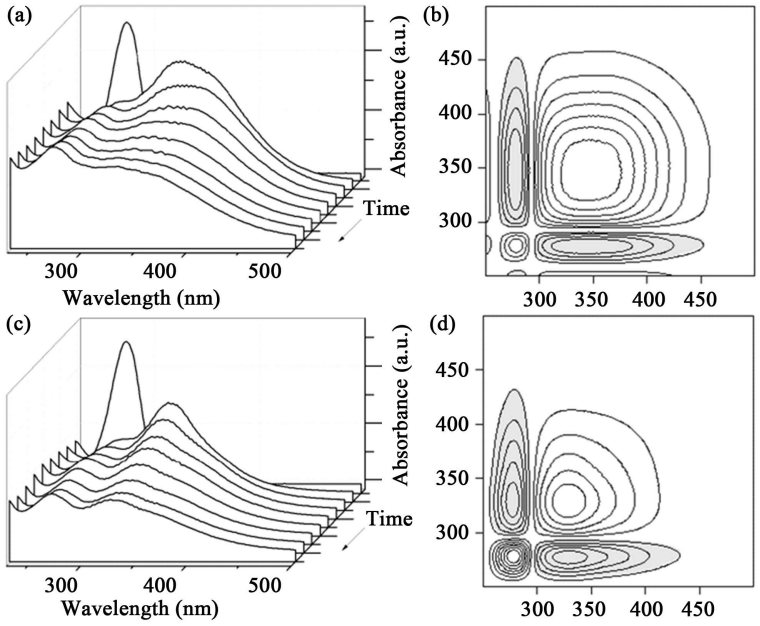

Figure 3. UV-Vis spectra of Moutai liquor ((a), t = 0 - 4 h, Δt = 0.5 h) and counterfeit Moutai liquor ((c), t = 0 - 4 h, Δt = 0.5 h), two-dimensional correlation UV-Vis spectra of Moutai liquor (b) and counterfeit Moutai liquor (d) based on the previous dynamic UV-Vis spectra, respectively.

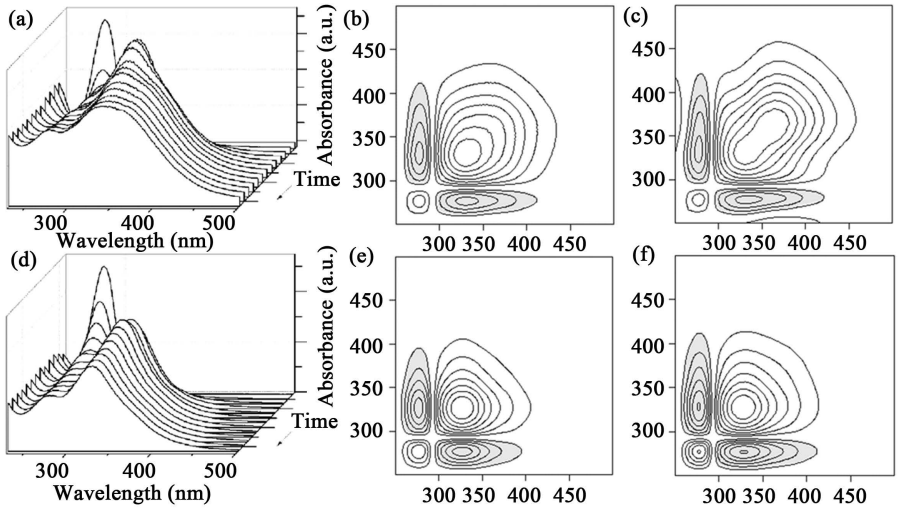

Figure 4. UV-Vis spectra of Moutai liquor ((a), t = 0 - 1 h, Δt = 5 min) and counterfeit Moutai liquor ((d), t = 0 - 1 h, Δt = 5 min), two-dimensional correlation UV-Vis spectra of Moutai liquor ((b), t = 0 - 30 min and (c), t = 0 - 1 h) and counterfeit Moutai liquor ((e), t = 0 - 30 min and (f), t = 0 - 1 h) based on the previous dynamic UV-Vis spectra, respectively.

4. Conclusion

In summary, the conventional UV-Vis spectroscopy and two-dimensional correlation UV-Vis spectroscopy are applied to the identification and discrimination of the authentic and adulterated samples of Chinese liquor Moutai in this work, respectively. The obtained results suggested that the novel two-dimensional correlation UV-Vis spectroscopy analytical method has the potential application in the identification and discrimination of the counterfeit Moutai liquor from genuine Moutai liquor. The potential applications of this strategy also could be most likely extended to the evaluation of other fields.

Acknowledgements

We gratefully acknowledge the financial support of this research by National Natural Science Foundation of China (61373058, 71433006) and the scientific research foundation of General Administration of Quality Supervision, Inspection and Quarantine of China (2012104009-2, 201410173), and the Scientific Research Foundation for Advanced Talent of Nanjing University of Finance & Economics.

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