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Application of Modern Detection Technology in the Detection of Liquor Components

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Abstract

Liquor, as a traditional brewing industry in China, has a history of more than 4000 years, and is also one of the six major distilled liquors in the world. However, the complexity of trace components in liquor make the research on detection difficult. It has only been more than 40 years. With the progress of social science, technology and economic development, people's requirements for liquor quality have been improved. It is particularly necessary to detect the conventional physical and chemical indicators, sensory indicators, safety indicators and flavor indicators that affect liquor quality. This paper summarized the application of detection and analysis technology in liquor, which provided new ideas for the application of new methods and technologies in liquor detection.

Keywords

Liquor; Detection technology; Chromatography; Spectroscopy; Electrochemistry.

1. Introduction

Liquor is a general term for Chinese liquor (except wine and rice wine), also known as "shao Jiu", "Lao Bai Gan" and "burning knife". It is one of the six famous distilled spirits in the world which has a history of over 4000 years. Chinese liquor is made from starch (sugar) by cooking, saccharification, fermentation, distillation, aging and blending with koji and liquor mother as saccharifying starter. The main components of liquor are ethanol and water, accounting for about 98%. Other trace components include aldehydes, alcohols, acids, lipids, ketones, etc., accounting for about 2% [1]. Although trace elements are few, they have great influence on liquor quality, flavor and security.

As early as the 1960s, people began to use modern scientific and technological means to analyze and detect liquor. So far, thousands of trace components in liquor have been detected. Liquor detection and analysis technology can be divided into two categories: traditional detection and analysis technology and modern new detection technology. Although the traditional detection technology has been widely used, it has some limitations. It is necessary to improve the function of existing equipment based on the traditional detection technology or to propose new detection technology, which has great social and economic value.

2. Chromatography and Mass Spectrometry

2.1 Gas Chromatography

Gas Chromatography (GC) is a chromatographic method using gas as mobile phase. The separation of mixtures is mainly realized by the difference of boiling point, polarity and adsorption properties ofsubstances.After being vaporized in the vaporizing chamber, the sample is carried into thechromatographic column by mobile phase. The column contains liquid or solid stationary phase. Because of the difference of boiling point, polarity or adsorption property of each component, each

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component tends to form a distribution or adsorption equilibrium between the mobile phase and the stationary phase.The column contains liquid or solid stationary phase. Because of the difference of boiling point, polarity or adsorption property of each component, each component tends to form a distribution or adsorption equilibrium between the mobile phase and the stationary phase. While the carrier gas is flowing, it is difficult to establish such a balance in practice. It is precisely because of due to the flow of carrier gas that the sample components are repeatedly distributed, adsorbed or desorbed during the movement. The result is that the components with high concentration in the carrier gas first flow out of the chromatographic column, while the components distributed with high concentration in the stationary phase flow out next. When the component flows out of the chromatographic column, it enters the detector immediately. The detector can convert the presence or absence of sample components into electrical signals, and the size of the electrical signals is proportional to the amount or concentration of the component under test. When these signals are amplified and recorded, the gas chromatograms have been finished [2], which are used for qualitative and quantitative analysis. It has the characteristics of high efficiency, rapidity, sensitivity, wide application range but poor qualitative ability.

Yaqi Zhang et al. [3] used HT-930 capillary column GC to optimize the chromatographic separation conditions of alcohols, esters and organic acids in liquor mixtures, and simultaneously detected 31 trace components of liquor. In 2016, Jialing Huang et al. [4] used headspace solid-phase microextraction-gas chromatography to detect pyrazines in liquor . The results showed that it has good linear relationship when the range was 0.05~20 mg/L, and the correlation coefficient r > 0.99.The detection limits (S/N =3) of eight pyrazines were 1.37~6.78 μg/L, the recoveries were 87.6% ~105.4%, and the relative standard deviations were 4.61%~6.37%. In 2018, Guohui Wu [5] simultaneously determined 10 common trace components in liquor by GC, including methanol, n-propanol, n-butanol, isobutanol, isoamyl alcohol, ethyl acetate, ethyl propionate, ethyl lactate, ethyl caproate and beta-phenylethanol. In 2019, Lin Sun et al. [6] used GC to quantitatively detect esters, alcohols and acids in liquor. The results showed that ethyl butyrate was 0.04~1.06, ethyl lactate was 0.44~2.24, ethyl acetate was 0.28~3.26, and methanol was 0.01~0.52.

2.2 Gas Chromatography-Mass Spectrometry

Gas Chromatography-Mass Spectrometry (GC-MS) is a method which combines the characteristics of gas chromatography and mass spectrometry to identify different substances in samples. The organic mixture is separated by a chromatographic column, and then enters the ion source through the interface to ionize into ions. Before entering the mass spectrometer, the ion has a total ion flow detector between the ion source and the mass analyzer to intercept part of the ion flow signal. The total ion current intensity with the time interval or the scan number change curve are the total ion flow chromatography of mixtures [7]. According to the information provided by mass spectrometry, qualitative and quantitative analysis of organic or inorganic substances as well as structural analysis of complex compounds can be carried out. It not only exerts the high separation ability of chromatography, but also exerts the high identification ability of mass spectrometry.

In 2016, Qian Zhang [8] used liquid-liquid extraction-GC/MS to determine volatile phenols in liquor. This method is beneficial to the accurate qualitative and quantitative analysis of volatile phenols in liquor, and provides theoretical basis for the analysis of liquor flavor components. In 2018, Yutong Chang et al. [9] established a headspace solid-phase microextraction-GC-MS for the determination of flavor substances in liquor. The flavor components of Fuyu-flavor liquor were analyzed and 74 components were obtained, including 50 esters, 8 alcohols, 6 acids, 3 ketones, 2 phenols, 2 aldehydes and 3 sulfur and nitrogen compounds. The relative standard deviation was 2.9%~15.7%. In 2019, Meilin Deng et al. [10] established an analytical method for the simultaneous determination of 10 plasticizers in liquor by gas chromatography-tandem mass spectrometry based on liquid-liquid extraction. The method has the advantages of simple pretreatment, high sensitivity, linear relationship, accuracy and precision. The detection limit of the method can meet the requirements of the statutory limit, and effectively improve the detection speed and flux.

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2.3 High Performance Liquid Chromatography

On the basis of classical chromatographic methods, High Performance Liquid Chromatography (HPLC) is based on the theory of gas chromatography. In technology, it adopt high pressure pump, high efficiency stationary phase and high sensitivity detector. It has the characteristics of high column efficiency, high selectivity, fast analysis speed, high sensitivity, good repeatability and wide adaptability [11]. HPLC can be divided into liquid-liquid distribution chromatography and chemical bonded phase chromatography, liquid-solid chromatography, ion exchange chromatography, ion pair chromatography, ion chromatography, space exclusion chromatography. Now HPLC is widely used in chemical, chemical, pharmaceutical, biochemical, environmental protection, agriculture and other scientific fields[12].

In 2018, Fang Wang et al. [13] used HPLC to simultaneously detect four major acids in liquor: lactic acid, butyric acid, hexanoacetic acid and acetic acid. In 2019, Bailizhen et al. [14] detect lactic acid in liquor by ultra-high performance liquid chromatography. Within the linear range, lactic acid has a good linear relationship, R2 is 0.9997, RSD is 0.23%, recovery rate is 90%-110%, detection limit is 1 mg/L. In 2020, Huilin Liu [15] used Dansyl chloride (DNS CL) as the derivatization reagent,thermo hypersil gold C18 column (250 mm × 4.6 mm, 5 μ m) as the separation column and Acetonitrile and 0.01 mol/L ammonium acetate solution containing 0.1% formic acid as mobile phases a and B with the flow rate of 0.8 ml / min and UV detector wavelength of 254 nm to nine kinds of biogenic amines in liquor and yellow rice wine. The results showed that the linear correlation between biogenic amines concentration and peak area was good (R2 > 0.998) in the range of 0.5-50 mg/L. This method is fast, simple and easy to operate, which provides technical support for the development of liquor.

2.4 Triple Quadrupole Mass Spectrometry

The vaporized material enters the mass spectrometer as a molecular state.As it bombarded by the electrons emitted by the filament, it becomes various fragments. Then these fragments enter the quadrupole, the quadrupole changes its direction through different electric directions. When these fragments pass through the quadrupole, due to the different mass and mass to charge ratio of the fragments, the forward direction changes with the change of the electric direction of the quadrupole. The time of the chip arriving at the receiving end is different. If the mass to charge ratio is too small or too large, the direction change of the charged fragment will be too fast or too slow, and it will hit the quadrupole and can not be detected. The fragments in the middle will arrive at the receiving end successively in the order of the mass to charge ratio from small to large, and they will be detected[16].

In 2015, Shengjun Wang et al. [17] Used isotope dilution gas chromatography triple quadrupole tandem mass spectrometry to determine the content of plasticizer in liquor. The results showed that the detection limits of DMP and DEP were 0.25 and 0.1 ng/ml, and the limits of quantification were 0.75 and 0.375 ng/ml, respectively. The average recoveries were 96.5% and 80.0%, and the precision was less than 5.4%, which could meet the detection requirements of DMP and DEP plasticizers in liquor. In 2016, Yinhui Wang [18] and others simultaneously determined gallic acid, ferulic acid, catechin, caffeic acid, luteolin, chlorogenic acid and p-coumaric acid in liquor by ultra performance liquid chromatography triple quadrupole tandem mass spectrometry. The linear range of the seven organic acids was 20~200ng/ml, and the linear correlation coefficients were all greater than 0.992. Under the three spiked levels, the average recoveries were 97.1%~111.3%, and the relative standard deviation (RSD) was controlled within 5%. In 2019, Shuangli Wang [19] used high performance liquid chromatography triple quadrupole mass spectrometry (hplc-qqq) to analyze aromatic esters in Chinese liquor, which could simultaneously detect the content of ethyl phenylacetate and isobutyl phenylacetate in liquor. This method has the advantages of simple to operate, short time to respond ,and high accuracy.

2.5 Two-dimensional Gas Chromatography-Time of Flight Mass Spectrometry

Two-dimensional Gas Chromatography-Time of Flight Mass Spectrometry(GC×GC-TOFMS) is a separation and identification technology with high resolution, high sensitivity and peak capacity

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developed in recent years. Full two dimensional gas chromatography (GC×GC) is a new separation system proposed by Liu et al in the early 1990s .Compared with the traditional one-dimensional gas chromatography (1D GC), GC×GC has the advantages of large peak capacity,obvious structural chromatographic behavior, fast separation speed and high sensitivity[20]. In 2007,Keliang Ji [21]et al. first introduced GC *GC-TOFMS into the field of liquor flavor compounds analysis.

In 2018, Zechun Hong [22] measured the trace components of a Maotai-flavor liquor by using two-dimensional gas chromatography-time-of-flight mass spectrometry. From the three liquor samples, 737, 783 and 794 effective flavor compounds were identified, including esters, acids, alcohols, ketones, aldehydes, nitrogen and sulfur compounds. In 2017, the characteristics of volatile components in typical sesame-flavor liquor were studied by headspace solid-phase microextraction (HSSPME) combined with TGC-TOF-MS. The volatile components identified in sesame-flavor liquor mainly include 130 esters, 26 alcohols, 15 organic acids, 88 aldehydes, ketones and acetals, 16 nitrogen compounds, 20 furans, 25 sulfur compounds, 14 terpenes and 6 other types. 11 volatile sulfur compounds and 12 terpene compounds were identified for the first time[23].

3. Spectrometry

3.1 Near Infrared Spectroscopy

Near Infrared Spectroscopy (NIRS) is an efficient and non-destructive fast detection technology, which can realize on-line detection and on-site monitoring. NIRS is mainly used to analyze the components of compounds containing hydrogen chemical bonds, and to identify the structure of the tested compounds by using the absorption characteristics of near infrared light[24]. According to the propagation mode of light in the medium, it can be divided into near-infrared spectral transmission method and near-infrared spectral reflection method. The transmission method is suitable for the detection of liquid with less impurities; the reflection method is mostly diffuse reflection, which is suitable for the detection of liquid and solid substances with high viscosity. The application of NIRS in wine industry is mainly aimed at qualitative analysis and quantitative detection of liquor products and it is little applied on the detection of base liquor[25,26]. Due to the complexity of liquor components and less content, it is difficult to quantitatively detect trace components.

In 2016, Shijiao Dai [27] determined the content of total esters in fermented grains by NIRS. By analyzing the near-infrared spectra of different fermented grains samples, MF and NCL spectral pretreatment methods were used to smooth and denoise the spectra. The near-infrared model of total esters in fermented grains was established by partial least squares (PLS) algorithm. The coefficient of determination R~2 was 97.15%, and the prediction standard deviation (RMSEP) was 0.07%. In 2019, Jinling Zhang [28] used NIRS to detect the content of methanol in liquor-based liquor, which ensured the stability and precision of the analysis. In 2019, Yuhong Tian et al. [29] used NIRS to detect the alcohol content, total acid content and total ester content in liquor production. More than 680 standard liquor samples were detected. Using PLS regression analysis to establish the corresponding model. The correlation coefficients of alcohol content, total acid and total ester in this model were 0.9995, 0.9576 and 0.9910. Then the model was verified by the blind samples, and the En values were -1 between +1.

3.2 Raman Spectroscopy

Raman spectrum is a kind of scattering spectrum. Raman spectrum analysis is based on the Raman scattering effect discovered by Indian scientist C.V. Raman. It analyzes the scattering spectrum with different frequency from the incident light to obtain the information of molecular vibration and rotation, and is applied to the study of molecular structure. It has the characteristics of fast and accurate, good reproducibility, simple sample pretreatment, compact and portable, and widely used[30]. Yijian Jiang [31] first tried to use Raman spectroscopy in the identification of true and false wine and concentration measurement In 1993. The results showed that laser Raman scattering could be an effective method to detect liquor quality, and could also be used to determine liquor age and guide liquor blending.

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In 2017, Si Chen et al. [32] optimized the experimental conditions of colloidal gold, volume ratio of solution to be tested and sodium chloride solution, mixing time and pH value of determination system with colloidal gold as reinforcement substrate, and used surface enhanced Raman spectroscopy to rapidly analyze saccharin sodium sweetener in liquor. The results showed that the surface enhanced Raman spectroscopy (SERS) method could quickly and accurately analyze saccharin sodium sweeteners in liquor, which could provide method support for the development of real-time and rapid detection device for sweeteners in liquor. In 2018, Xiuxing Zhang [33] used LRS-2 laser Raman spectrometer to measure and analyze absolute ethanol and four different brands of liquor, and obtained their Raman spectra. The results showed that the peak value of Raman spectrum characteristic peak of liquor changed with the change of concentration. The higher the concentration was, the greater the peak value was, and the position of Raman spectrum peak shifted with the change of concentration. It can be inferred that the concentration affects the activity of chemical bond in ethanol molecule.

4. Electrochemical Sensor Technology

Electrochemical sensor is composed of one or more sensitive elements which can produce electrical signals related to the chemical properties of the components to be measured. Its principle is to convert the chemical information generated by the interaction between the measured object and the sensitive material into measurable electrical signals. According to the detection object, it can be divided into biosensor, gas sensor and ion selector. According to different working modes, it can be divided into potential type sensor, current type sensor and conductance type sensor [34]. The potential sensor is based on the ion exchange mechanism of phase boundary model to detect the potential change caused by the change of different charge quantity on the interface between taste solution and sensor. The Voltammetric sensor measures the response current between the working electrode and the counter electrode under the potential excitation signal relative to the reference electrode. The conductivity sensor takes the change of conductivity of electrolyte solution after oxidation or reduction as output[35]. At present, electrochemical sensors have been widely used in food, medicine, environment, agriculture, industrial production and other fields [35,37,38,39].

4.1 Electronic Nose/Tongue Detection Technology

Electronic nose and electronic tongue are bionic instruments that simulate human olfactory and taste organs in the middle and late 20th century. After signal processing and pattern recognition, the whole olfactory information of various solutions is finally obtained, which realizes the quantitative and qualitative detection of complex liquids [40]. Electronic tongue uses materials similar to biological system as the sensitive lipid membrane of sensor. When one side of lipid membrane contacts with taste substance, the membrane potential changes, resulting in response. The quantitative relationship of various taste substances is detected, and the taste sense of acid, sweet, bitter, salty and fresh can be analyzed by matching with human taste sense [41]. At present, electronic nose, electronic tongue and other new rapid detection technologies have the advantages of accuracy, rapidity, simple sample pretreatment, convenient operation, good repeatability, etc., and have broad development prospects. They can respond to all substances in liquor as a whole, and are widely used in the food industry, but they have not yet formed a unified evaluation standard [42, 43].

At present,TS-5000z electronic tongue system of Japanese insent company and astree electronic tongue of French alpha m.o.s company are widely used. Astree electronic tongue electrochemical sensor array consists of a standard reference electrode (Ag/AgCl) and seven cross selective sensors. Its analytical principle and method are completely similar to that of human tongue. It is used to test the comprehensive taste of complex soluble organic compounds and inorganic compounds. Ting Tian [44] used Gemini electronic nose system produced by alpha MOS to detect the first to seventh rounds of Maotai flavor liquor brewed by solid grain. The electronic nose system contains six sensors. Each sensor has different characteristics and sensitivity to substances, and each sensor can only produce sensitive response to some specific compounds. The results showed that the method could well

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distinguish and identify the Maotai flavor liquor of different rounds, and had good reproducibility. In 2017, Huimin Zhou et al. [45] constructed an electronic nose system for rapid prediction of the total sugar content of Shaoxing yellow rice wine. At the same time, the total sugar content of rice wine samples was tested. The characteristic values of electronic nose detection data were extracted by nonlinear double superposition stochastic resonance, and the prediction model of total sugar content was established by combining the characteristic value with the test results of total sugar content of yellow rice wine samples. The model can not only predict the total sugar content of rice wine samples, but also realize the type detection of rice wine samples. In 2018, Zhou Xiaoyang et al. [46] carried out DFA analysis on yellow water by using the sensor relaxation characteristics of electronic tongue, and modeled the sensor data through BP neural network, and obtained a method for rapid detection of wine precision, acidity, reducing sugar and residual starch in yellow water. In 2019, Li Qiang [47] proposed a layer by layer self-assembly technology to prepare self-assembled monolayer functionalized compound sensitive coating of quartz crystal microbalance by controlling the concentration of precursor solution at room temperature, and developed a high-precision and low-cost quartz piezoelectric sensor. Three kinds of pattern recognition systems based on multi-dimensional scale analysis support vector machine, random forest and error back-propagation neural network are proposed and constructed.

4.2 Other Electrochemical Sensors

In addition to the electronic nose / tongue, there are almost no other ready-made electrochemical instruments which can directly detect the substances in liquor. In the experiment, people usually use self-made or self-assembled electrochemical sensors to detect the substances in liquor.

Xinyue Jiang used graphene electrode sensor to measure plasticizer in liquor by AC impedance method. It not only absorbed diethyl phthalate in liquor, but also absorbed other plasticizers in liquor. It can be seen that this method is lack of specificity in the detection of plasticizer content in liquor, and it is suitable for the detection of total plasticizer content in liquor, but not for specific plasticizer [48]. Lijuan Yi used Polypyrrole Modified Glassy Carbon Electrode to detect tyramine in two kinds of liquor samples, and verified the determination by HPLC. The results showed that the modified electrode for the determination of tyramine content in liquor was close to that of HPLC, and its preparation was simple, low-cost, with good repeatability, stability and selectivity, which could be used for the determination of tyramine in liquor [49].

5. Conclusion

After more than 40 years of development of liquor detection technology and update of detection equipment, traditional physical and chemical analysis technology has been widely used in liquor detection. However, most of the traditional detection technologies have some disadvantages, such as large equipment, complex sample pretreatment, relatively slow analysis time and insufficient field test ability, which can not meet the current requirements of liquor industry for detection technology. Therefore, considering the detection of components and quality in liquor production process and liquor products, it is of great significance to continuously research new technologies, constantly integrate new technologies, enhance the research of detection methods and improve the detection performance. Chromatography and mass spectrometry can effectively separate various components for detection and analysis, which is also the reason why they can be used in industrial production, but the detection time is longer than spectroscopic and electrochemical methods. In recent years, a large number of scholars have studied its application in liquor component detection and liquor classification recognition. Spectral method needs a large amount of data to establish the model, and it requires high data analysis technology. Electrochemical sensors can achieve on-line detection with rapid response, but most of them have cross sensitivity, and the detection accuracy of single component is not as good as chromatography. Therefore, if the combination of rapid separation technology and electrochemical sensor can improve the selectivity of electrochemical sensor for single substance, the problem of low detection accuracy caused by complex components in liquor can be effectively avoided, and the

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detection speed can be accelerated It is believed that in the near future, with the development of science and technology, the material components in the process of liquor production can be monitored in real time, liquor quality control and component detection can be promoted to a new height.

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