CHEMICAL ENGINEERING TRANSACTIONS
VOL. 71, 2018
A publication of AIDIC
The Italian Association of Chemical Engineering
Online at www.aidic.it/cet
Guest Editors: Xiantang Zhang, Songrong Qian, Jianmin Xu

ISBN 978-88-95608-68-6; **ISSN** 2283-9216
DOI: 10.3303/CET1871073

Analysis of Detecting Process of Chemical Composition in Black Foods--A Case Study of Fatty Acids

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In this paper, based on the research of fatty acids in black foods, black rice and white rice were used as the subjects. Soxhlet extraction method and GC/MS were used to quantitatively analyze the content of fatty acids in black rice. The study found that the contents of monounsaturated, polyunsaturated, and saturated fatty acids in the 10 fatty acids detected by black rice were 35.36%, 36.94%, and 25.02% respectively, and the total detection rate was 97.32%. In comparison, white rice detected three types of fatty acid compounds, of which the saturated fatty acid content was 27.37%, while the unsaturated fatty acid was 45.04%, and the polyunsaturated fatty acid was 27.59%. It can be seen that black rice contains higher levels of fatty acids and polyunsaturated fatty acids than white rice does, and black rice has a higher nutritional value. This study on the content of fatty acids in black rice can provide corresponding reference for subsequent studies on fatty acids in other black foods.

1. Introduction

1.1 Literature review

In recent years, as the development and research of black foods has intensified, these products have gradually formed an industrial value chain. Clinical practice has proven that black foods can regulate the physiological functions of the human body and also prevent many types of diseases. In this regard, further research into the chemical composition of black foods has become a hot spot. In the case of lycium ruthenicum, the detection limit of fatty acids was found to reach 0.42-1.84 ng/mL by means of external standard method and online mass spectrometry. At the same time, Pre-column derivatization detection of fatty acids by high-performance liquid chromatography revealed that unsaturated fatty acids accounted for approximately 64% of the total fatty acids in lycium ruthenicum (Hu et al., 2014; Yao, 2018).
According to the detection and analysis of the main fatty acids in grape seed oil, among the fresh food species, the stearic acid contents of the phoenix and Jingyu cultivars are higher; in the white brewing, the oleic acid content of Chardonnay is smaller and higher in Munson; in red brewing, the contents of stearic acid, linolenic acid in phoenix, Jingyu, Jufeng were significantly different (Zhang et al., 2012). In the fatty acid analysis of blackberry seed oil by ultrasound-assisted method, the best process parameter of blackberry seed oil was extracted as petroleum ether. After 10 minutes of ultrasonic extraction with a power of 400 W, the extraction rate of blackberry seed oil was 17.06% (Hu et al., 2018; Suarez et al., 2018; Zhu et al., 2010). Many scholars have also analyzed the growth areas of wild lycium ruthenicum. There is a considerable gap in the chemical composition of lycium ruthenicum in different regions. The contents of carbohydrates, proteins, fatty acids and ash content range from 69.55% to 77.14% and 10.76% to 14.72 respectively. %, 3.90% to 6.89%, and 6.63% to 10.99% (Shuang et al., 2017).
At the same time, in order to optimize the Soxhlet extraction process, some scholars have analyzed that linoleic acid, oleic acid, palmitic acid and stearic acid are the main fatty acid constituents of the seed oil of the

Please cite this article as: Yang C., Chen S., Qin Q., 2018, Analysis of detecting process of chemical composition in black foods--a case study of fatty acids, Chemical Engineering Transactions, 71, 433-438 DOI:10.3303/CET1871073

Canarium pimel, and concluded that the Canarium pimel seed has high oil yield, and its fatty acid composition is similar to palm oil but it has a higher content of unsaturated fatty acids, so that the nutritive value is superior to palm (Guo et al., 2010).

1.2 Research background

At present, with the deepening of the concept of health, how to apply appropriately black products such as blackcurrants, black rice, black beans, and comfrey has become a focus of attention (Hou et al., 2011). For this reason, many scholars have conducted many studies on black food ingredients, extraction techniques, safety of use, and nutritional value. In this context, the chemical composition of black foods has also been applied to the production of various fields in society. For example, black rice is a good raw material for wine making to make black rice (Gongmi) wine, black milk and black rice wine, black glutinous rice wine, etc.; in the case of non-alcoholic drinking products, beverage series such as Ganoderma lucidum, mulberry, black currant, violet hyacinth, and black ant have been developed (Wei et al., 2013).

It can be seen that the development of black food as a raw material has become a new attempt in the new generation of scientific and technological circles. In addition, practice has shown that black foods have the effect of promoting the body's various circulatory systems and delaying the aging of body functions. And different dietary systems with black foods can further improve the body's immune system and play its role in health care (Ma et al., 2011). Therefore, it is of great practical significance to study the content of chemical components in black foods. In this paper, taking fatty acids as an example, we will conduct further research on black foods and think that black foods can be used in a wider range of applications.

2. Black food and fatty acid theory

Nowadays, there are two general meanings that are common to black foods. First, foods that contain melanin coats, such as black rice, black sesame, black chicken, and fungus, can be called brown foods. Second, they contain crude fiber content. Foods with more ingredients can also be listed as black foods (Lu et al., 2018). At the same time, black foods generally include two main characteristics: first, natural edible pigments cause blackness in black foods; second, black foods contain more amino acids, fatty acids, mineral elements, and vitamins than similar products. B group and other ingredients.

Chapter 2 Fatty acids are the main components of the nutrients such as glycolipids and neutral fats. They are compounds composed of oxygen, hydrogen and carbon (Yang et al., 2015). They are also the main components of fats and are important nutrients that humans need. In general, fatty acids include long-chain, medium-chain, and short-chain fatty acids, while black foods mostly contain long-chain fatty acids. In addition, according to the number of carbon atoms in the double bonds, the fatty acids include monounsaturated fatty acids, polyunsaturated fatty acids, and saturated fatty acids (Hu et al., 2014).

Chapter 3 In addition, fatty acids are classified into trans- and cis-unsaturated fatty acids according to the same or different hydrogen atoms. Under this classification, fatty acids that do not contain double bonds are saturated fatty acids (Wang et al., 2013). For example, animal fats are all saturated fatty acids. Monounsaturated fatty acids are the most representative of oleic acid, and natural oils and oils include oleic acid (Chen et al., 2017). Trans fatty acids are not present in vegetable oils, and most of these fatty acids are present in animal fats or in hydroprocessed oils and fats. Polyunsaturated fatty acids contain more than two double bonds and have very special biological activity (Du et al., 2010).

3. Detecting process of fatty acid in black food

3.1 Periment materials

In order to ensure a more rigorous and scientific detection process, this paper selected the common black rice and black sesame for detection analysis, and at the same time, in order to highlight the differences in fatty acids in black food, we select samples of white rice and white sesame as control materials for testing.

3.2 The main reagents and equipment

A total of six reagents were used in this study, namely methanol, anhydrous ethanol, potassium hydroxide, n-hexane, anhydrous sodium sulfate, and petroleum ether, and all were purchased from Changzhou Zhongao Chemical Co., Ltd. Among them, the boiling range of petroleum ether is 60 to 90 degrees Celsius, and all reagents are analytically pure.

The equipment used during the test is as follows:

GC-MS 6800 Gas Chromatograph Mass Spectrometer, Jiangsu Tianrui Instrument Co., Ltd.;

20μL microsampler, Shanghai Yuanquan Biotechnology Co., Ltd.;

FB 224 electronic balance, Shanghai Shunyu Hengping Scientific Instrument Co., Ltd.;

N-1300D-WB Rotary Evaporator, Shanghai Assistant Industry Co., Ltd.;
BIOBASE electric constant temperature drying oven, Shandong Boke Biological Industry Co., Ltd.;
Z216MK Desktop Centrifuge, Germany HERMLE Labortechnik GmbH
QFN-BSXT-02 Soxhlet extractor, Suzhou Xuan Warui Intelligent Technology Co., Ltd.;
TSD-8000 ultrasonic cleaner, Tianshi Zhongmei Technology Co., Ltd.
WF-300A Chinese medicine grinder, Jiangyin Tianfeng Pharmaceutical Machinery Co., Ltd.
SYD-265B thermostatic water bath, Jiangsu Yiyong Instrument Equipment Co., Ltd.;
SHZ-D (III) circulating water vacuum pump, Shanghai Cang Mao Enterprise Co., Ltd.

3.3 Detection methods and process

3.3.1. Sample preparation

For Fatty acid detection in black foods, the food oil content needs to be measured simultaneously. Therefore, the selected four kinds of samples were placed in a traditional Chinese medicine grinder for crushing, and were packed in a sealed container, and the samples were stored in a refrigerator freezer area (Yan et al., 2012).

3.3.2. Fat oil extraction treatment

In general, the methods for the detection of food oil content include Soxhlet extraction, acid hydrolysis, and alkali hydrolysis. The specific choice depends on the main components of the sample (Wang et al., 2017). Considering the high protein content of the samples selected in this study, Soxhlet extraction is mainly used for extraction and determination of oil content. The four kinds of sample was sieved through a 0.45 mm aperture, and 5 g of the crushed sample was precisely weighed and placed in a conical flask. Then we added 150 ml of petroleum ether with a funnel, which must maintain a boiling range of 60 to 90 degrees Celsius. After stirring with a glass rod, heating and reflux treatment was performed for 5 hours. Then, the extracted liquid was subjected to reduced pressure rotation and evaporation and concentrated treatment. Finally, petroleum ether was recovered.

3.3.3 Fatty acid methylation treatment

For the fatty oil in the second step, a 0.5 ml sample was accurately pipetted and dropped into a round bottom flask. Then, 0.5 ml of a 0.5 mol/L blended potassium hydroxide-methanol solution and 8 ml of n-hexane were added, and then refluxed in a water bath of 70 degrees Celsius for 20 minutes and allowed to stand for cooling. After cooling, the solution in the flask was pipetted into a centrifuge tube, 10 ml of distilled water was added and discharged, and sonication was completed. After the completion of the centrifugation step, the sodium sulphate solution was added to the drying process. After standing for a period of time, the supernatant was extracted and sealed, and further GC/MS analysis was performed.

3.3.4. Chromatographic conditions and mass spectrometry conditions

Chromatographic conditions: gasification chamber temperature is maintained at 300 degrees Celsius; sample volume is 0.4 μL; carrier gas flow rate is 1 ml/min; split ratio is 100 to 1; chromatographic column is a 30m*0.25mm*0.33μm elastic quartz capillary column. The temperature of the program rises from 100 degrees Celsius to 280 degrees Celsius at a rate of 10 degrees Celsius per minute while maintaining the temperature of 5 minutes (Chen et al., 2016). Mass spectrometry conditions: Electron bombardment of the ion source were conducted at 230 degrees Celsius; 1988V voltage maintenance, emission current mA was at 34.6; interface temperature was at 230 degrees Celsius; and the 20-500 m/z scan range was defined (Yang et al., 2013).

3.4 Results

Fatty oil components of the four samples were identified by gas chromatography mass spectrometry. The experimental data were processed through the G170LBA ChemStation system to search the Nis98 spectral library for control, compounding and analysis. The peak area normalization method was used to determine the relative percentage of fatty acids in the sample. The specific results are shown in Tables 1 to 5.
According to Table 5, a total of 10 types of fatty acids were detected in black rice. The contents of saturated fatty acids, monounsaturated fatty acids, and polyunsaturated fatty acids were 25.02%, 35.36%, and 36.94%, respectively, and the total detection rate was 97.32%. A total of 3 compounds were detected in white rice, with a saturated fatty acid content of 27.37%, an unsaturated fatty acid of 45.04%, a polyunsaturated fatty acid of 27.59%, and a total detection rate of 100%. It can be seen that the content of polyunsaturated fatty acids in black rice is significantly higher than that of white rice. The methyl ester of octadecenoic acid, an essential fatty acid in black rice, is 36.96%, which is significantly higher than 27.63% in white rice.

Table 1: Analysis results of fatty acid in black rice

NO.	Maintenance	compounds	Relative area	Similarity
1	19.03	Methyl Lignocerate	1.45	0.99
2	17.41	Methyl Behenate	0.63	0.98
3	15.78	Methyl icosanoate	1.59	0.98
4	15.58	Methyl Cis-11-eicosenoate	0.89	0.95
5	14.02	Methyl stearate	2.99	0.98
6	13.88	9-Octadecenoic acid methyl ester	34.65	0.98
7	13.80	9,12-Octadecenoic acid methyl ester	36.96	0.99
8	12,14	14-methyl	19.77	0.99
9	11.78	Methyl Palmitoleate	0.55	0.99
10	10.05	Methyl myristate	0.52	0.96

Table 2: Analysis results of fatty acids in white rice

NO.	Maintenance	compounds	Relative area	Similarity
1	12.13	Methyl Palmitate	27.33	0.98
2	13.41	9,12-Octadecenoic acid methyl ester	27.63	0.99
3	13.78	9-Octadecenoic acid methyl ester	45.04	0.98

Table 3: Analysis results of fatty acid in black sesame

NO.	Maintenance	compounds	Relative area	Similarity
1	19.05	Methyl Lignocerate	0.45	0.99
2	15.41	Methyl Behenate	2.23	0.98
3	15.82	Methyl icosanoate	2.18	0.99
4	15.58	Methyl Cis-11-eicosenoate	1.09	0.98
5	14.12	Methyl stearate	9.65	0.99
6	14.08	9-Octadecenoic acid methyl ester	0.65	0.98
7	12.80	9,12-Octadecenoic acid methyl ester	0.96	0.88
8	5.14	14-methyl	0.77	0.96
9	11.87	Methyl Palmitoleate	0.62	0.99
10	10.03	Methyl myristate	0.08	0.95

Table 4: Analysis results of fatty acid in white sesame

NO.	Maintenance	compounds	Relative area	Similarity
1	19.04	Methyl Lignocerate	0.35	0.98
2	16.41	Methyl Behenate	0.58	0.99
3	15.81	Methyl icosanoate	2.08	0.99
4	15.62	Methyl Cis-11-eicosenoate	1.08	0.98
5	14.02	Methyl stearate	8.65	0.99
6	14.07	9-Octadecenoic acid methyl ester	12.65	0.99
7	14.80	9,12-Octadecenoic acid methyl ester	59.08	0.99
8	11.85	Methyl Palmitoleate	0.42	0.99
9	10.03	Methyl myristate	0.07	0.96

Table 5: Comparison of fatty acid composition in samples

Fatty acid	relative peak area			
	Black rice	White rice	Black sesame	White sesame
C14: 0	0.52		0.08	0.07
C16: 0	19.77	27.33	0.77	
C16: 1	0.55		0.62	0.42
C18: 0	2.99		9.65	8.65
C18: 1	34.65	45.04	0.65	12.65
C18: 2	36.96	27.63	0.96	59.08
C20: 0	1.59		2.18	2.08
C20: 1	0.89		1.09	1.08
C22: 0	0.63		2.23	0.58
C24: 0	1.45		0.45	0.35
saturated fatty acid	25.02	27.37	28.42	25.91
Monounsaturated fatty acids	35.36	45.04	70.53	14.33
Polyunsaturated fatty acids	36.94	27.59	0.58	59.22

4. Conclusion

The fatty acids in black rice were detected by Soxhlet extraction, and subjected to methyl esterification using potassium hydroxide-methanol solution. Finally, quantitative analysis of GC/MS was performed. Based on GB/T 17376-1998 animal and vegetable oil fatty acid methyl ester preparation method, this paper uses potassium hydroxide - methanol solution instead of HC1-methanol solution, improved and innovated fatty acid methyl ester preparation method.

With this method, a fast and complete derivatization reaction can be obtained, and the esterification effect is better. And the operation flow and steps are very simple and suitable for processing analysis of large batches of samples. Through analysis and testing, it can be concluded that black foods are rich in fatty acids and have good beauty and anti-aging effects. Therefore, in the later period, the comparative study and analysis of the antioxidant properties of black foods can be conducted to explore the structure-efficiency relationship of the antioxidant properties.

Acknowledgement

Lianyungang Kudzu Food Limited Company; Research about health food through biotransformation on kudzu; Jiangsu institute of Marine resources development (lianyungang) (HKK01617); “A Project Funded by the Priority Academic Program Development of Jiangsu Higher Education Institutions”.

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