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Effect of Oil Content in Tea-seed Pancake on the Determination of Tea Saponin

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Abstract. According to the national standard for the determination of tea saponin content in tea-seed pancake (GB/T 35131-2017), tea saponin was obtained by extracting oil with ethanol and removing solvent. The actual samples of tea-seed pancake did not completely remove the oil, and some of the residual oil content was as high as 9%. Due to the long period of national standard determination, people often use ethanol to extract tea saponin directly, instead of subsequent acid hydrolysis or alkali hydrolysis steps, so as to realize the rapid evaluation of tea-seed pancake quality. In this case, the residual oil has a greater impact on the results. If it was still tested according to the national standard method, the result will be much higher than the actual result. In this work, petroleum ether extraction process was selected before ethanol extraction to remove the residual oil. Experimental results showed that, the accuracy of the determination was effectively improved.

1 Introduction

Tea-seed pancake is a kind of cake left after extracting oil from camellia oil fruit, which is purple brown particles, as shown in Fig.1. Tea-seed pancake contains about 18% tea saponin, fresh cake can be as high as more than 20%, chemically refined cake only about 12%. The annual output of Camellia oleifera seeds in China has reached more than 800000 tons, and the annual output of pressed camellia oleifera seed meal can reach more than 500000 tons[1], which is the country with the largest variety, the widest distribution and the largest output of camellia oleifera seeds in the world. It is mostly produced in Jiangxi, Hunan and other places.

Fig.1 Tea-seed pancake (picture obtained from internet)

Tea saponin is a hemolytic toxin, which can dissolve the red blood cells of fish, so it can kill wild fish, loach, snails, mussels, frog eggs, tadpoles and some aquatic insects. Tea saponin is easy to dissolve in alkaline water, when used, adding a small amount of lime water, the effect is better. Due to the high protein content of tea meal, it is also a kind of high-efficiency organic fertilizer, which is widely used in crops and fruit trees with excellent effect. It can increase the fertility of the pond with less silt and poor bottom material. The use of tea-seed pancake as a pond clearing drug has more unique efficacy than other drugs.

Tea seed cake is widely used in fish pond, paddy field, high-grade lawn, earthworm, ground tiger and other pests. As a kind of green medicine, tea-seed cake can decompose by itself and remain non-toxic. It has no effect on human body and is safe to be used. The second is not to kill water plants, but also to promote the growth of water plants. Third, there are no side effects on shrimp and crab larvae, and the emergence rate of shrimp and crab larvae is higher.

Tea-seed pancake is also used in the production of feed and extraction of tea saponin from tea-seed pancake through deep processing. Tea seed powder is widely used in washing chemical industry, machine derusting, aquaculture, golf course, killing earthworms and so on.

Tea saponin is a kind of glycoside compounds extracted from tea seeds. The pure product is white columnar crystal, as shown in Fig.2. Tea saponin is a pentacyclic triterpenoid saponin [2-7], which consists of seven ligands, four glycosides and two organic carboxylic acids. It is a natural non-ionic surfactant, and has many physiological functions, such as antibacterial and anti-inflammatory, virus killing, antioxidant, anti hypertension, insecticidal and insect repellent, and promoting plant growth. Because of the above properties of tea saponin, it has a wide range of applications in

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agriculture, medicine, food, daily chemical and other fields[2-4]. Tea saponin can improve the wettability and suspension rate of wettable powder (≥ 75%) as a wetting agent of pesticide. As a natural non-ionic surfactant, tea saponin can significantly improve the physical and chemical properties of pesticide solution, increase the effective volume of pesticide on the target, and help to give full play to the efficacy of pesticide. Therefore, the application effect can be improved.

Fig.2 Tea saponin (picture obtained from internet)

China has established a number of manufacturers for the production of camellia oleifera seed oil and tea saponin. The production of tea saponin and its preparations has been greatly developed, but there are still a considerable number of resources that have not been fully utilized. Therefore, the development and extraction of tea saponin for daily chemical, building materials, agriculture, aquaculture, pharmaceutical industry, paper industry, leather industry and coating industry will produce good economic, social and ecological benefits[8].

Zhang Tuanjie [9] and others established phloroglucinol method, phenol sulfuric acid method, p-Dimethyl aminobenzaldehyde method, fluorescence spectrophotometry method, thin layer chromatography method, potassium bromate method, high performance liquid chromatography method and vanillin concentrated sulfuric acid method for the determination of tea saponins.

Tea saponin was extracted by a new technology, namely water extraction and alcohol precipitation. The optimum conditions of water extraction were as follows:

The extraction temperature was 60 °C. Liquid-solid ratio was of 10:1. The extraction time was 2 h.

Water extraction followed by alcohol precipitation. The results showed that the optimal conditions for this precipitation process were as follows: concentration of ethanol was 90%, volume ratio of ethanol to concentrated solution was 4:1, alcohol precipitation temperature was 75 °C and alcohol precipitation time was 2.5 h. Compared with water extraction and alcohol precipitation under these conditions, the extraction rate of water extraction and alcohol precipitation was 95.2%, and the purity was 69.9%, which improved the purity and was conducive to subsequent refining treatment.

With the wider use of tea-seed pancake, the price of tea-seed pancake is becoming higher and higher. Many manufacturers, after pressing the tea seed, no longer extract residual oil, but sale the tea-seed pancake directly. The residual camellia oil is mainly composed of palmitic acid, stearic acid, oleic acid, linoleic acid and linolenic acid [10], which has a great influence on the determination of crude tea saponin.

In the previous research of the extraction and determination of tea saponin from tea-seed pancake, the external factors, such as extraction solvent, solvent amount, extraction temperature and extraction time, are carefully discussed. However, oil content, as an internal factor, is relatively easy to be ignored. The influence of the oil content of the sample on the determination of tea saponin was studied in this work.

2 Experimental

2.1 Reagents and Instruments

Reagents and instruments used in the process of analysis and detection were listed in table 1.

Table1. Reagents and Instruments

Name	Specification/model	Manufacturer
anhydrous ethanol	AR	Sinopharm Group
petroleum ether	AR	Sinopharm Group
electronic balance	ME240E	Mettler Toledo
electric constant temperature drying oven	UFE500A0	Meimert company
constant temperature water bath	DK-S24	Shanghai Jing Hong company

2.2 Experimental Samples

The tea-seed pancake from different factories in Yichun area of Jiangxi Province was selected as the experimental sample, which was fully crushed and passed through 150 μm sieve, as shown in Fig.3.

Fig.3 Powders of tea-seed pancake

2.3 Experimental Method

Fig.4 shows the experimental device. Firstly, experimental sample with the weight of 5.0 g and 100 ml anhydrous ethanol was added into a 250 ml triangular ground flask sequentially. Then， a dry reflux condenser was installed, and refluxed for 2 h in a boiling water bath. After that process, the condenser was removed. The triangular grinding flask was placed on the hot water bath and let it stand for about 1 h (Fig.4 Step 1).

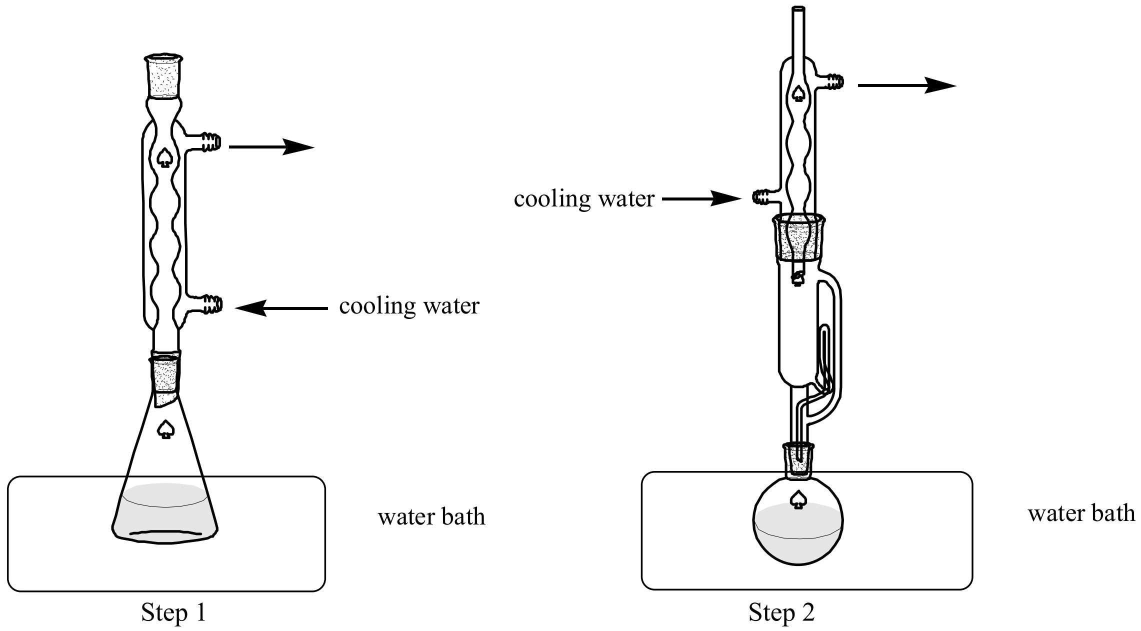

Fig.4 Experimental device

Secondly, the extract was filtered into a flat bottomed flask with constant weight at 105 °C through absorbent cotton when it was hot, and the insoluble matter was retained in the original triangular flask as far as possible. Then，40 ml anhydrous ethanol was added and refluxed on the boiling water bath for 1 h. After that, the triangular grinded flask was tilted on the hot water bath pot and left standing for about 1 h, and the extract was filtered into the flat bottomed flask through the original funnel.

Thirdly, the flat bottomed flask containing the extraction solution was installed with Soxhlet extraction tube and reflux condenser (Fig.4 step 2). Anhydrous ethanol was evaporated into the extraction tube in a water bath for recovery until the anhydrous ethanol in the flask was nearly clear.

At last, the flask was moved into a constant temperature oven, dried at 105 °C for 2 h. Then， the flask was took out and cooled in a dryer. The product was weighed.

To examine the influence of residual oil, sample without oil removal with the weight of 5.0 g was put into the filter paper cartridge, put the filter paper cartridge into the extraction cartridge of Soxhlet extractor, and added petroleum ether from the upper end of the condenser tube of the extractor to two thirds of the volume of the bottle. Sample was extracted in water bath for 10 h. After extraction, the filter cartridge and the sample were placed in a 250 ml triangular flask to extract tea saponin according to the experimental method.

3. Results and discussion

The results of crude tea saponin determination with and without oil removal were shown in Table 2.

For the three experimental samples, the oil content was 5.4%, 7.6% and 8.4%, respectively. The ea saponin content was 18.0 %, 27.7 % and 24.9 %, respectively, when the detection method without residual oil removing was selected. On the other hand, when the residual oil was removed from the raw material, the corresponding results were 12.5 %, 18.7 % and 16.5%, respectively.

The relative error was 9.0 %, 9.7 % and 10.1 % for sample 1#, sample 2# and sample 3#.

It could be analyzed from table 2 that, the relative error increased with the increase of residual oil content. Due to the principle of similar compatibility, the residual camellia oil contains polar components and was easily soluble in hot ethanol. When tea saponin was extracted by hot ethanol, the residual oil was also extracted, which made the result higher.

The higher the oil content, the greater the relative error is. Considering the uncertainty of the residual oil content in tea-seed pancake samples, it is suggested that petroleum ether be used to extract and remove the residual oil first, and then hot ethanol be used to extract tea saponin for detection, so as to improve the accuracy of tea saponin content determination.

Table 2. Effect of different oil content on the determination results of tea saponin in tea-seed pancake

Tea saponin content (%) / Method	Sample 1#	Sample 2#	Sample 3#
Oil content	5.4	7.6	8.4
Without residual oil removing	18.0	27.7	24.9
With residual oil removing	12.5	18.7	16.5
Relative error	**9.0**	**9.7**	**10.1**

4. Conclusions

1) In this work, petroleum ether extraction process was added before ethanol extraction to remove the residual oil. Experimental results showed that, the accuracy of the determination was effectively improved.
2) The oil content of tea-seed pancake itself has a great influence on the extraction and determination of tea saponin. The higher the oil content, the greater the relative error is.
3) Considering the uncertainty of the residual oil content in tea-seed pancake samples, it is suggested that petroleum ether be used to extract and remove the residual oil first, and then hot ethanol be used to extract tea saponin for detection, so as to improve the accuracy of tea saponin content determination.

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