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Exploration of the method for extracting caffeine from tea

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Abstract:

Caffeine is a widely consumed alkaloid with considerable relevance in both the food and pharmaceutical sectors, where efficient extraction methods are of great practical importance[1-5]. Among the various techniques available, the conventional Soxhlet extraction method is commonly used due to its straightforward operation, although there remains potential to improve its yield[7]. This study set out to explore strategies for enhancing caffeine yield from tea leaves by refining key parameters within the Soxhlet extraction process. The investigation examined the influence of several variables—such as extraction temperature, siphon cycle frequency, heating voltage[9], and the type of crystallization container—on the overall extraction efficiency. The results showed that adjustments to these factors led to measurable improvements in caffeine yield[6]. By comparing outcomes before and after optimization, the study further confirmed the beneficial effects of the process modifications. These findings offer practical insights for the efficient and cost-effective extraction of bioactive compounds from natural materials.

Keywords: Soxhlet extraction, caffeine, yield, extracting condition

1. Experimental Principles and Related Chemicals

1.1 Experimental Principles

Caffeine is readily soluble in organic solvents such as ethanol[2-5], has a boiling point of around 178°C, and sublimes easily. Meanwhile, other substances in tea leaves, such as proteins and cellulose, are either insoluble or poorly soluble in ethanol and will not appear in the extract. In this experiment, 99.5% pure ethanol[6] is uniformly used as the extraction solvent. It should be noted that in addition to caffeine, tea leaves also contain acidic substances such as tannin acid. After concentrating the ethanol extract, quicklime or soda is added. These alkaline substances can neutralize the acidic compounds in the concentrated solution, forming salts. Compared to the original acidic substances, these salts have a much higher boiling point, thereby preventing the acidic components from interfering with the sublimation and crystallization of caffeine. After purification by sublimation, caffeine can be obtained as needle-like crystals on the filter paper.

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1.2 Chemicals

Fenghuang Dancong (black tea), 99.5% ethanol, calcium oxide (or sodium carbonate and other alkaline substances)
2.Initial Experimental Protocol

2.1 Experimental Apparatus

Mortar, analytical balance,round-bottom flask, Soxhlet extractor[10], adjustable heating mantle, water bath heater, asbestos gauze, glass rod, beaker, evaporating dish, glass funnel, filter paper

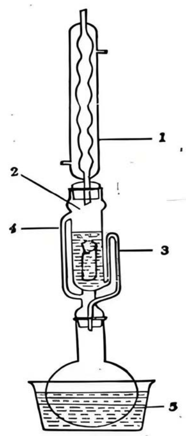

Figure2-1 Soxhlet extractor
1:condenser 2:extractor 3:siphon
4:connecting pipe 5:heater

2.2 Experimental Procedure

2.2.1 Extraction

Weigh 10.0 g of Fenghuang Dancong tea leaves, wrap them in filter paper, and place the package into the Soxhlet extractor. Add 80 mL of 99.5% ethanol to the extractor, and place boiling stones and 30 mL of 99.5% ethanol into the round-bottom flask.
Install a spherical condenser and connect it to a cooling water source. Heat to extract for 3.5 hours, during which a total of 4 siphoning cycles should occur in the extractor. Stop heating immediately after the fourth siphoning cycle is completed.

2.2.2 Concentration and Evaporation

Transfer the extract to a beaker and place it on an electric heater or in a water bath. Heat for 20 minutes until most of the ethanol has evaporated, and the mixture in the beaker becomes highly viscous or nearly solid.

2.2.3 Sublimation and Purification

Add 2 g of sodium carbonate to the mixture and heat it with an alcohol lamp while stirring with a glass rod. This step evaporates any remaining ethanol and allows the alkaline substance to react with acidic compounds in the mixture. Continue until the mixture becomes completely solid.
Transfer the solid mixture to an evaporating dish placed on asbestos gauze. Take a small piece of filter paper and pierce it evenly with a needle on one side. Then, place the filter paper on the evaporating dish from bottom to top, with the perforated side facing the inside of the dish, and cover it with an inverted glass funnel (a small wad of cotton should be placed at the top of the funnel to prevent the caffeine from escaping during sublimation). Continue heating on the asbestos wire mesh for 30 minutes. If turbid gas (brown smoke) appears in the funnel, immediately stop heating. After cooling, carefully scrape the caffeine crystals attached to the filter paper into a pre-weighed container, weigh it, and record the result.

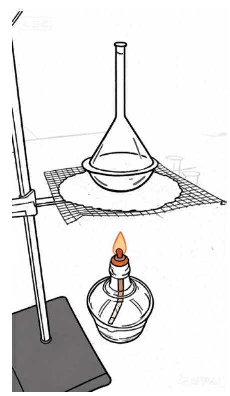

Figure2-2 sublimation setups

3. Improved Experimental Protocols

3.1 First Improvement

3.1.1 Modifications (First Set of Variables)

· Ethanol volume: 20 mL in the extractor, 80 mL in the round-bottom flask.

3.1.2 Procedure

· Weigh 10.0g of Fenghuang Dancong tea.
· Assemble the spherical condenser, connect cooling water, and apply continuous heat.
· No siphoning occurred. The experiment was terminated.

3.1.3 Result

The experiment failed as siphoning did not occur.

3.1.4 Analysis and Discussion

Analyzing the procedure and environmental conditions, the following potential causes of failure were identified:
Firstly,insufficient heating power from the ordinary heating mantle, leading to inadequate temperature in the

round-bottom flask and preventing efficient ethanol vaporization and condensation.Secondly,imbalanced ethanol distribution; excessive ethanol in the round-bottom flask may have significantly reduced heating and evaporation efficiency[3-8].Furthermore, low ambient temperature causing evaporated ethanol to condense prematurely in the connecting tubing and flow back into the round-bottom flask.

3.2 Second Improvement

3.2.1 Modifications (Second Set of Variables)

A. Ethanol volume: 80 mL in the extractor, 30 mL in the round-bottom flask.
B. Heating apparatus: Adjustable heating mantle.
C. Evaporation apparatus: Electric hotplate set to 70V.
D. Crystallization container: Evaporating dish.

3.2.2 Procedure

· Weigh 10.0g of FengHuang DanCong tea.
· After the fourth siphoning occurred ,transfer the extract to a beaker and evaporate the ethanol using an electric hotplate set to 70V. Stir with a glass rod.
· After 20 minutes of evaporation, add 2g of sodium carbonate to the mixture. Heat with an alcohol lamp to initiate crystallization.
· After approximately 30 minutes,carefully scrape the caffeine crystals formed on the filter paper into a pre-weighed container using a clean metal spoon. Weigh to obtain the final mass and result. The experiment concluded.

3.2.3 Result

Crystallization was successful. Total caffeine extracted: 0.0103g. Yield: 0.1027%.

3.2.4 Analysis and Discussion

The yield was lower than that of typical extraction experiments. By analyzing the experimental procedures and surrounding environment, the following potential reasons were identified:
Firstly,during the siphoning process, not all caffeine may have dissolved, potentially due to the chemical properties of the tea leaves, resulting in partial loss.Secondly,some caffeine may have sublimated and escaped during the evaporation of ethanol, leading to partial loss[3-8].
Furthermore,during the crystallization heating, some caffeine may have escaped or failed to crystallize from the residual material due to insufficient temperature, causing further loss.

3.3 Third Improvement

3.3.1 Modifications (Third Set of Variables)

· Evaporation apparatus: Water bath heater.

3.3.2 Procedure

· After the fourth siphoning occurred ,transfer the extract to a beaker and evaporate the ethanol using a water bath heater. Stir with a glass rod.
· After approximately 20 minutes of evaporation,add 2g of sodium carbonate to the mixture. Heat with an alcohol lamp to initiate crystallization.
· After approximately 30 minutes,carefully scrape the caffeine crystals formed on the filter paper into a pre-weighed container using a clean metal spoon. Weigh to obtain the final mass and result. The experiment concluded.

3.3.3 Result

Crystallization failed. No caffeine crystals were produced.

3.3.4 Analysis and Discussion

The experiment did not yield caffeine. Through analysis of the experimental steps and surrounding conditions, the following potential causes were identified:
Firstly,during the water bath heating process, the beaker was left uncovered, leading to the sublimation and escape of a significant amount of caffeine along with the gaseous ethanol.Secondly,during the crystallization process, the lack of a tight seal allowed some caffeine to escape.Furthermore,the tea leaves may have contained impurities or exhibited varying chemical properties, resulting in a lower-than-ideal amount of caffeine available for extraction.

3.4 Fourth Improvement

3.4.1 Modifications (Fourth Set of Variables)

·The kind of tea is the Jasmine tea

3.4.2 Procedure

· Weigh 10.0g of Jasmine tea.
· After the fourth siphoning occurred,transfer the extract to a beaker and evaporate the ethanol using a water bath heater. Stir with a glass rod.
· After approximately 20 minutes of evaporation,add 2g of sodium carbonate to the mixture. Heat with an alcohol lamp to initiate crystallization.
·After approximately 30 minutes, carefully scrape the caffeine crystals formed on the filter paper into a pre-weighed container using a clean metal spoon. Weigh to obtain the final mass and result. The experiment concluded.

3.4.3 Experimental Results

Crystallization was successful, but the quantity of caffeine extracted was extremely low.
Yield: Negligible.

3.4.4 Analysis and Discussion

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The experiment yielded almost no caffeine. Through analysis of the experimental steps and surrounding conditions, the following potential causes were identified:
Firstly,the composition of the jasmine tea leaves may have contained impurities or exhibited differing chemical properties, resulting in a lower-than-ideal amount of caffeine available for extraction.Secondly,during the ethanol evaporation process, excessive heating from the electric hotplate caused the mixture temperature to rise too high, leading to significant sublimation of caffeine. This sublimated caffeine escaped along with gaseous ethanol in the form of turbid fumes (a mixture of white and brown), resulting in substantial loss.

3.5 Fifth Improvement

3.5.1 Experimental Improvements (Fifth Set of Variables)

· Crystallization Container: Beaker

3.5.2 Experimental Procedure

· After approximately 30 minutes, immediately stop heating once brown fumes appear.Carefully scrape the caffeine crystals formed on the filter paper into a pre-weighed container using a clean metal spoon. Weigh the mass to obtain the results and conclude the experiment.

3.5.3 Experimental Results

Crystallization was successful. Total caffeine extracted: 0.0052 g. Yield: 0.052%.

3.5.4 Analysis and Discussion of Results

Although the experiment successfully produced caffeine, both the amount of crystallization and the yield were significantly lower than the ideal values. Through analysis of the experimental steps and surrounding conditions, the following potential reasons were identified:
Firstly,during the siphoning process, not all caffeine dissolved in the ethanol, resulting in partial loss.Secondly,during the transfer of the ethanol solution after siphoning, some solution remained in the round-bottom flask, leading to partial loss.Furthermore,although the voltage of the electric heating plate was reduced during the evaporation process, the heating effect remained excessive, causing a significant portion of the caffeine to sublimate and escape, resulting in noticeable loss.Additionally,since the crystallization process was conducted in a beaker, container-related factors (such as the beaker absorbing part of the heat) may have reduced the heating efficiency of the mixture, preventing some caffeine from sublimating and crystallizing, leading to partial loss.

4. Results and Discussion

The comparative results of the experiments after multiple improvements are summarized in the following table:
Experiment Tea Variety Ethanol Distribution Evaporation Apparatus Crystallization Container Result
1 Phoenix Dancong ;Extractor: 20 mL Flask: 80 mL ;Failed
2 Phoenix Dancong ;Extractor: 80 mL Flask: 30 mL ;Electric hotplate Evaporating dish ;Successful, 0.0103g crystals (0.103% yield)[4]
3 Phoenix Dancong ;Extractor: 80 mL Flask: 30 mL ;Water bath heater Evaporating dish ;Failed
4 Jasmine tea ;Extractor: 80 mL Flask: 30 mL ;Electric hotplate Evaporating dish Successful, minimal crystals (negligible yield)
5 Phoenix Dancong ;Extractor: 80 mL Flask: 30 mL ;Electric hotplate Beaker ;Successful, 0.0052g crystals (0.052% yield)[2-3]

5. Conclusion

This experiment introduced improvements in four key areas: the type of tea leaves used, the ethanol concentration, the evaporation setup, and the container for crystallization. Based on the comparative data presented earlier, these adjustments had a obvious effect on the efficiency of caffeine extraction from tea, with certain modifications increasing the yield by up to [percentage]%. More importantly, the revised protocol not only improved the yield but also played a significant role in enhancing students' hands-on skills[7], observational abilities, and critical thinking. By turning abstract theories into observable phenomena, it helped enhance their understanding of scientific concepts. At the same time, it encouraged a strict and honest approach to science, strengthened problem-solving skills, and promoted teamwork and safety awareness. These outcomes are essential for inspiring scientific curiosity and bridging the gap between theory and practice, laying a strong foundation for students' future development in experimental work.

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