An Investigation of Caffeine Content in Tea – An Abstract

Aim

1. To determine the amount of caffeine extracted by steeping different brands of commercially available tea bags in hot water.
2. To relate the caffeine content in tea with the methods of tea production.
3. To investigate the factors affecting the extent of caffeine extraction.
4. To suggest, based on experimental findings, a guideline for healthy tea drinking on behalf of tea lovers.

Introduction

Teas are mainly classified into 3 classes according to the extent of fermentation. They are Green tea, Oolong tea and Black tea. Black tea is fully fermented. Green tea does not undergo fermentation while Oolong tea is only partially fermented. For instance, Black tea contains more caffeine than Oolong tea, which contains more caffeine than Green tea. This also relates to the extent of fermentation of tea. The longer time the fermentation, the more caffeine the tea contains.

Caffeine is a naturally occurring organic compound. It is a white, odourless and prism-like crystal that is very soluble in hot water. When you brew a pot of tea, most of the caffeine in tea will dissolve in the first couple of minutes. Caffeine is stable under normal temperature and pressure showing no reactions with strong acids, alkalis or chlorine water. However, it decomposes on strong heating to give toxic oxides of carbon and nitrogen.

When you steep a cup of tea, the amount of caffeine dissolves in water depends on (1) the time and temperature of steeping, (2) the size of the tea leaf and (3) the type of tea used. The longer the brewing time and the hotter the water, the more caffeine ended up in the cup. The smaller the tea leaf, the stronger the extraction of caffeine.

In this project, the amount of caffeine extracted by brewing tea bags in hot alkaline aqueous solutions was investigated. Experimental conditions were also varied to study their effects on the extent of extraction.

Theory

Extraction of caffeine from tea requires a thorough understanding of the chemistry of caffeine and other substances, mainly tannins, present in tea. For instance, tea contains 10~30 % of tannins by mass but only 2~4 % of caffeine by mass. The separation of caffeine from tannin impurities is the most crucial step in the extracting process.

Tannins are usually divided into two groups, namely hydrolyzable tannins (HTs) and proanthocyanidins (PAs). The commonly known tannic acid is an example of hydrolyzable tannins.

HTs can be hydrolyzed by mild acids or mild bases to yield carbohydrate, phenolic acids or their salts. Under the same conditions, proanthocyanidins do not hydrolyze. Therefore, by varying the nature of the extracting solvents, caffeine extracted from tea can be purified from a mixture containing HTs and PAs.

Procedure

Preparation of Tea Infusion

1. In the first set of samples, three tea bags from each of the seven brands were steeped in 150 cm^3 of boiling deionized water for 1 minute in a beaker containing 5.5 g of sodium carbonate and 12.5 g of sodium chloride.
2. Step 1 was repeated by steeping tea samples for 5 minutes and 30 minutes respectively.

Sample	Set 1	Set 2	Set 3
Steeping Time (min)	1	5	30

3. The tea remained in the tea bags were squeezed back to the beaker by watch glasses and the used tea bags were discarded

Solvent Extraction

4. The tea samples were cooled to room temperature and then introduced into separating funnels.
5. The beakers formerly contained the tea samples were rinsed with 20cm^3 of dichloromethane. The rinsings were then added to the separating funnels.
6. The mixtures in the funnels were then shaken gently. The vapour produced was released after a few shakes. Any emulsions were broken up by agitation with a glass rod. The mixture in each funnel was allowed to separate for 10 minutes.
7. The lower layer together with the emulsion, if any, was transferred to the original beaker.
8. The tea samples left in the separating funnels were extracted twice with two 20 cm^3 portions of dichloromethane.
9. The three dichloromethane extracts were combined and washed twice with 20 cm^3 of cold 6M sodium hydroxide and once with 20 cm^3 of cold deionized water.
10. The dichloromethane extract was transferred to a clean beaker and dried over anhydrous sodium sulphate. The clear solution was then decanted into another clean beaker.

Evaporation and Sublimation

11. The dried samples were heated gently over a hot plate to evaporate the excess solvent until about 5 cm^3 of solution was left in each beaker.
12. A clean **soda glass** test tube was weighed and the mass was recorded as (α).
13. The sample was added into the pre-weighed test tube using a dropper. The beaker and the dropper were rinsed with a little dichloromethane and the rinsing was transferred to the test tube. (This is to minimize the loss of caffeine). The solvent was evaporated completely by heating the test tube over a hot plate. The test tube containing crude caffeine was then weighed again and the mass was recorded as (β). The value ($\beta - \alpha$) represents the mass of crude caffeine.
14. *A cotton wool plug was inserted at the mouth of the test tube and the test tube was heated in an oil bath for about 2 hours. Pure caffeine was condensed as sublimate at the upper part of the tube. The test tube was cut into two pieces at about 3 cm from the bottom with a red glow nichrome wire.
15. The upper part of the tube that contained pure caffeine was weighed and the mass was recorded as (ϕ).
16. A little caffeine was removed and tested for purity by melting point determination.
    The upper part of the tube was cleaned and dried completely. It was then weighed and the mass was recorded as (γ). The value ($\phi - \gamma$) represents the mass of pure caffeine obtained from the tea sample.

* In the first attempt of sublimation, cotton wool plugs were not inserted in the test tubes.
This resulted in a contamination of the final product with vapour of oil from the oil bath.
In the final set-up, this error had been avoided by insertion of cotton wool plugs that absorbed the oil.

Follow-up Experiment after Submission of Project Report

Four sets of experiments were tried out as shown below.

1. Six tea bags instead of three tea bags were used in preparing the tea infusion of **Black Tea** to see if an increase in amount of sample used would improve the percentage yield of caffeine. Since more tea would imply more difficult solvent extraction due to formation of more stubborn emulsion, **Black Tea** was chosen for the test because it gave less stubborn emulsion due to the presence of less tannins.
2. It was thought that adding sodium chloride when steeping tea would salt out more tannin salts which rendered the emulsion more stubborn. Therefore, **Green Tea**, which gave most stubborn emulsion, was steeped in deionized water without sodium chloride and sodium carbonate to see if it helped to reduce the formation of emulsion.
3. In addition to steeping the samples for 1, 5 and 30 minutes, samples of Sow Mei, Jasmine, Iron Buddha and Oolong were steeped for 15 minutes to see if the assumption that all caffeine would be extracted from tea sample in 30 minutes was correct.
4. In solvent extraction using deionized water, the mixtures in the separating funnels were allowed to stand overnight to see if it would improve the yield of caffeine.

Conlcusion

To sum up, this project has accomplished the following tasks :

1. A workable experimental procedure for extracting pure caffeine from tea has been developed which utilizes only the relatively cheap apparatus available in a secondary school laboratory.

2. The factors affecting the extent of caffeine extraction were investigated and the results were shown below.
   a. **The time factor**
   Results : The longer the steeping time, the more caffeine and tannins extracted.
   Moreover, over 50% of caffeine was extracted in the first-minute steeping.
   It was also found that the steeping time of tea should not be long, otherwise, more tannins would dissolve which formed stubborn emulsion making the solvent extraction more difficult.

   b. **The nature of tea**
   Results : The more the tea was fermented, the higher was its caffeine content.
   For example, Black Tea (fully fermented) was found to contain more caffeine than Pu Erh (partially fermented), which contained more caffeine than Green Tea (not fermented).

3. A guideline for healthy tea drinking was suggested as follows.
   a. Do not steep tea for a long time since more caffeine will be released.
   b. Discard the first infusion of tea since most caffeine will dissolve in the first couple of minutes
   c. If you only want to taste tea, you should choose Green Tea, which contains less caffeine and more tannins (the sources of flavour). If you want to increase the alertness, you should choose Black Tea, which contains more caffeine and less tannins.

4. Findings from follow-up experiments
   a. The yield of caffeine was significantly improved by allowing the extraction mixture to stand overnight.
   b. The amount of emulsion was substantially reduced when sodium chloride and sodium carbonate were not used in preparing tea infusion.
   c. The amount of caffeine extracted increased sharply in the first 5-minutes steeping and remained more or less the same in the next 25 minutes.

Experimental Results

Table 1 : Results with Caffeine Contaminated with Oil

1min. Samples	[1]Pu Erh	[2]Green Tea	[1]Black Tea	[1]Oolong	[1]Jasmine	[1]Iron Buddha	[2]Sow Mei
Yield of Caffeine	26.9mg	16.6mg	23.1mg	16.2mg	18.5mg	12.6mg	17.0mg
M.P. Range (^oC)	238-240	238-244	220-238	246-248	260-266	238-246	240-246

5min. Samples	[1]Pu Erh	[2]Green Tea	[1]Black Tea	[1]Oolong	[1]Jasmine	[1]Iron Buddha	[2]Sow Mei
Yield of Caffeine	13.9mg	13.7mg	*15.8mg	*18.5mg	18.3mg	14.1mg	14.6mg
M.P. Range (^oC)	238-246	238-248	238-242	238-242	236-242	240-244	260-266

30min.Samples	[1]Pu Erh	[2]Green Tea	[1]Black Tea	[1]Oolong	[1]Jasmine	[1]Iron Buddha	[2]Sow Mei
Yield of Caffeine	15.8mg	12.8mg	12.5mg	16.2mg	14.7mg	*17.2mg	22.9mg
M.P Range (^oC)	236-246	236-248	237-248	235-244	240-245	238-244	242-250

[1]Rickshaw tea [2] Luk Yu tea

The data in red are not reliable due to contamination of caffeine with oil

The data marked with an * are not reliable because borosilicate tubes were used instead of soda glass tube and the borosilicate tubes broke into pieces when cut. Some caffeine might be lost or the caffeine might be contaminated with small pieces of glass.

Table 2 : Results with Caffeine Not Contaminated with Oil

1 min Samples	[1]Pu Erh	[1]Black Tea	[2]Green Tea
Yield of Caffeine	3.3mg	4.2mg	*19.6mg
M.P. Range (^oC)	234-240	245-248	240-248

5 min Samples	[1]Pu Erh	[1]Black Tea	[2]Green Tea
Yield of Caffeine	5.1mg	6.0mg	2.9mg
M.P. Range (^oC)	236-248	236-246	238-245

30min Samples	[1]Pu Erh	[1]Black Tea	[2]Green Tea
Yield of Caffeine	6.6mg	7.0mg	5.6mg
M.P. Range (^oC)	235-242	244-258	238-250

*This datum is not reliable because the test tube containing caffeine was broken into piece when cut

Results of follow-up experiments

1. The effect of using more tea bags in steeping on the yield of caffeine

No. of tea bags	Type of Tea	Steeping Time/min	Yield of Caffeine/mg	Yield of Caffeine per Tea Bag/mg
3	Black Tea	5	6.0	2.0
6	Black Tea	5	15.8	*2.6

*The increase in the yield of caffeine may not be the consequence of using more tea bags since the mixture was allowed to stand overnight after shaking with cold deionized water.

2. The effect of steeping tea in deionized water without sodium chloride and sodium carbonate

Type of Steeping	Type of Tea	Steeping Time/min.	Yield of Caffeine/mg
With deionized water only	Green Tea	5	*18.1
With NaCl(aq)/Na_2CO_3(aq)	Green Tea	5	2.9

*The increase in the yield of caffeine may not be the consequence of steeping tea without sodium chloride and sodium carbonate for the same reason mentioned above. However, it was noticed that less emulsion was formed when tea was steeped in deionized water only.

3. The effect of the duration of steeping on the yield of caffeine

Type of Tea	Sow Mei				Jasmine				Iron Buddha				Oolong			
Steeping Time/min	1	5	15	30	1	5	15	30	1	5	15	30	1	5	15	30
Yield of Pure Caffeine/mg	15.0	21.3	18.7	15.7	13.7	*16.4	20.0	23.0	12.1	18.9	20.3	16.5	23.0	*12.8	18.8	19.0

The yields were much greater than those in Table 2 since all extraction mixtures were allowed to stand overnight before separation.

The data in red are not reliable since extraction with dichloromethane was only done twice.

References

1. Comprehensive Chemistry, 'HKAL' Course – Physical Chemistry II (Fourth Edition), T.M. Leung & C.C. Lee, Fillans, 1996
(P.514-520: Partition of a Solute between Two Phases)

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(P.280-282: Solvent Extraction)

3. Modern Physical Chemistry, Liptrot, Thompson & Wallser, E.L.B.S.,1982.
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(P.124-126: Sublimation)
(P.138-139: Use of drying agents)
(P.127-130: Solvent extraction, treatment of emulsions)

5. Modern Organic Chemistry (Fourth Edition), R.O.C. Norman & D.J. Wanddington, E.L.B.S., 1985.
(P.17: Recrystallisation)

6. Useful Web links

- ☞ http://www.chemicalagency.net/caffeine.html
(A modified procedure for caffeine extraction)
- ☞ http://www.teatalk.com/index.htm
- ☞ http://www.revolutiontea.com/index.html
- ☞ http://www.stashtea.com
- ☞ http://www.chinamist.com/index.htm
- ☞ http://www.ansci.cornell.edu/plants/toxicagents/tannin/index.html