IB Chemistry (SL)

Extraction and Isolation of Caffeine in Teas

RESEARCH QUESTION: WHICH TYPE OF TEA (ORGANIC OR COMMERCIAL TEA) WOULD HAVE THE GREATEST CAFFEINE RECOVERED USING EXTRACTION AND PURIFICATION METHODS?

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ABSTRACT

The purpose of this investigation is to find out the highest caffeine percent recovery using extraction and purification methods in organic and commercial teas. The hypothesis of this research is: If the caffeine from one organic and two commercial teas is extracted and measured, then the commercial teas will have the highest percent caffeine recovery because of the production of it compared to the organic (native grown) Navajo tea. In order to test the hypothesis, the procedure will be to test the ethyl acetate that was separated from the liquid tea. After extracting the caffeine from the tea, the next procedure is to purify the caffeine. In order to purify the caffeine, the ethyl acetate and caffeine are separated in extraction. Crucial to this investigation is the crystallization and sublimation in order to obtain the pure caffeine. The last procedure is to identify the amount of caffeine by using Paper Chromatography to identify the Retardation Factor, Rf:

$$Rf = \frac{distance\ sample\ travels}{distance\ solvent\ travels}$$

After calculating the Retardation Factor, Rf, the amount of caffeine obtained was calculated with this equation:

$$Percent\ of\ Caffeine\ Recovered = \frac{Mass\ of\ Caffeine\ Recovered}{Mass\ of\ Caffeine\ Available} x100\%$$

The results of the extraction and isolation of caffeine experiment present that the commercial teas, Earl Grey and Lipton, had a higher percentage of caffeine when compared to the organic Navajo Tea and the decaffeinated green tea. This is concluded that the hypothesis was acceptable to this investigation, that commercial tea has more caffeine than organic tea. The average percent recovery of caffeine for the Lipton tea was 10.5% about 0.5% higher than Navajo Tea and

Decaffeinated Green Tea. To add on, the commercial tea had a mass of 0.290 g of caffeine recovered which measures the residue that was collected from extraction. This is lower though compared to Navajo Tea. Navajo has the highest mass recovered of 0.340g. The percent recovery will tell the amount of caffeine that the mass. Even though, decaffeinated green tea was tested, caffeine was still found, with a percent caffeine recovery 3%, and 0.135g of caffeine.

INTRODUCTION

The world's most popular chemical compound is caffeine. This chemical compound is found in soda, coffee, and even pain reliever. This investigation will focus on caffeine found in a Native American tea familiar to the Navajo tribe – called Navajo tea. This tea is located in the Southwest region of the United States (Figure 2). Thelesperma Megapotomicium, also known as Navajo tea is usually hand-picked by Navajos who know how to identify and steep this delectable plant. Once Navajo tea is brewed it can be consumed as a beverage, and the Navajo traditions of Navajo tea are it can be used to soothe aches and pains of the body. In studies of Navajo tea, it was discovered Navajo tea contains Luteolin. This flavonoid has multiple biological effects such as anti-inflammation, anti-allergy and anticancer. Additionally, research suggests people with type 2 diabetes have different reactions to caffeine. For people with type 2 diabetes, caffeine raises blood sugar and insulin levels, and certain stress hormones, like epinephrine or adrenaline. Epinephrine can prevent your cells from processing as sugar and keeping the body from making as much insulin. Caffeine also blocks adenosine (protein). This molecule plays a big role in how much insulin your body makes, and how the cells respond to it. Caffeine keeps adenosine from doing its job, making it hard to clear sugar from the blood

quicker. Investigating the amount of caffeine is important because most Navajos have diabetes, and this topic would be concerning to them.

Caffeine has a similar molecular structure to adenosine, Figure 1. Adenosine is a neurotransmitter that allows the body to relax and slow activity. Caffeine affects multiple cognitive brain functions such as sleeping, memory, and modifies brain dysfunctions and diseases for example: Alzheimer's disease, Parkinson's disease, Huntington's disease, Epilepsy, Pain/Migraine, Depression, and Schizophrenia. Caffeine is soluble in many solutions like: water, and ethyl acetate, Figure 1.

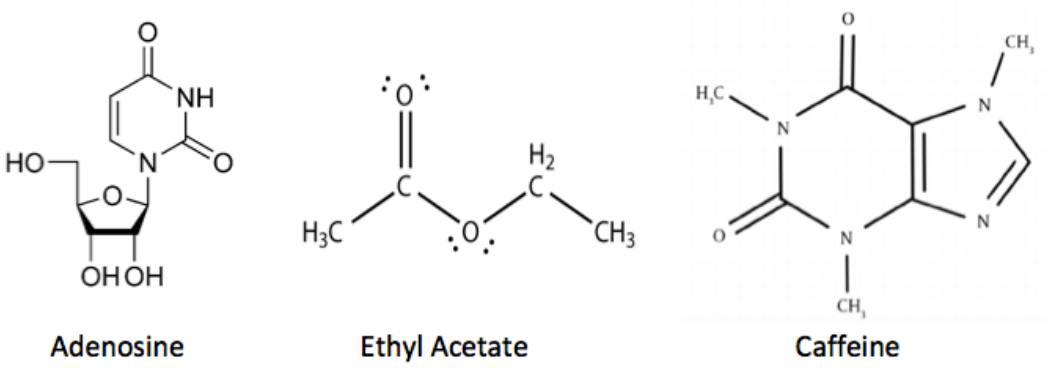

Figure 1. Three Molecules Central To This Study

RESEARCH QUESTION: Which type of tea (organic or commercial tea) would have the highest average caffeine recovered, using extraction and purification methods.

HYPOTHESIS: *If the caffeine from the one organic and two commercial teas is extracted and measured, then the commercial teas will have the highest average percent caffeine recovery because of the production of it, compared to organic and native grown Navajo tea.*

METHODOLOGY

The independent variables of this investigation are Earl Grey tea (commercial), Lipton (commercial), Organic Navajo Tea (native grown),, and Decaffeinated Green Tea (commercial). The dependent variables are the Caffeine Paper Chromatography, the R_f Value, Extraction, and percent caffeine. A Control variable will be the caffeine standard. In order to assess the results of the extraction and isolation of caffeine, a graph with the Caffeine Paper Chromatography, Retardation Factor Rf , Extraction, and percent caffeine recovered percentages is presented for Earl Grey Tea (commercial), Lipton (commercial), Organic Navajo Tea (native grown), and Decaffeinated green tea (commercial).

A primary source of information regarding the medical effects of caffeine is a report from a Cancer Therapy journal, because it is using the same herb that this investigation is trying to understand. Also, since caffeine and cancer are correlated, this may have a foreshadowing on this investigation's results. A limitation of this source is the lack of comparison to other caffeine free or caffeine dominant substances. The secondary source is another lab experiment on caffeine extraction on tea leaves. This source has a lot of information because it is similar to this investigation. A limitation of this study is the lack of information on what kind of tea the researcher used.

MATERIALS

1) 5 bundles of raw Navajo tea (organic, native grown)
2) A box of Decaffeinated Green Tea (commercial)
3) 1 package of Lipton Tea and 1 package of Earl Grey Tea (both commercial)

4) Ethyl Acetate
5) Sodium Carbonate
6) Sodium Sulfate
7) 250mL Beaker (4)
8) 100mL Beaker
9) 100mL Graduated Cylinder
10) Hot Plate
11) Funnel
12) Ethyl Alcohol
13) Water-ice mixture
14) 50mL Beaker
15) Quantative Filter Paper (9)
16) Caffeine Standard Solution
17) Pipettes (4)

PROCEDURES

1. Obtain four unopen tea bags, weigh the total mass to the nearest 0.1g and record.
2. Obtain another tea bag, empty out the contents, weigh to the nearest 0.1g and record in data table.
3. Add 100mL of distilled water to a 250mL beaker then add the four tea bags from step 1 to the same beaker.
4. Weigh 4 grams of sodium carbonate, Na_2CO_3, and add the solid to the beaker. Stir to dissolve.
5. Using a hot plate heat up the beaker for 15 minutes, just under boiling.

6. Remove beaker from heat and let cool.
7. Once cooled, remove tea bags from solution and discard.
8. Utilizing the hood, pour 25mL of ethyl acetate to the solution in the beaker.
9. Pour mixture from beaker to a 250mL separatory funnel.
10. Stopper the funnel and, holding the stopper in place lightly tip the funnel upside down.
11. While the funnel is inverted open the stopcock to vent he pressure.
12. Close stopcock and place the funnel in the ring attached to the support stand.
13. Wait for the two solutions to separate.
14. After the mixture is separated place a 250-mL beaker below the stopcock and collect the lower aqueous layer.
15. Collect remaining ethyl acetates in a second 250mL beaker.
16. Add the aqueous layer from Step 14 in the separatory funnel.
17. Add a fresh 25mL of ethyl acetate to the separatory funnel
18. Repeat procedures 10-12
19. After the layers separated again collect the bottom aqueous layer in the same 250mL beaker from step 14.
20. Collect remaining ethyl acetate.
21. Weigh four grams of sodium sulfate and add to the ethyl acetate mixture
22. Swirl mixture
23. Mass a 100mL beaker containing two boiling stones to the nearest 0.01g.
24. Pour ethyl acetate in the 100mL beaker containing the two boiling stones.
25. Heat mixture at a low setting until ethyl acetate evaporates
26. Remove beaker and let cool

27. Weigh the beaker with the crude caffeine
28. Using the same boiling stones transfer them to a 50mL beaker
29. Add 20mL of 95% ethyl alcohol to the 50mLbeaker
30. Heat beaker at low setting. **DO NOT BOIL.**
31. Once the solution is hot remove
32. Fill a 250 mL beaker of 50mL of ice water
33. Pour hot ethyl alcohol in the 100mL beaker containing the crude caffeine
34. Swirl to mix and dissolve
35. Place the 100mL beaker in the 250mL beaker and wait for crystals.
36. Once crystallization is complete remove 100mL beaker
37. Mass a quantitative filter paper and record in the data table
38. Set up gravity filtration method with quantitative filter paper
39. Filter caffeine from the cold ethyl alcohol
40. Upon the end of filtration remove filter paper and set aside to dry
41. When dry mass the filter paper
42. In a small test tube add approximately 2mL of ethyl alcohol. Add purified caffeine and dissolve. Save solution for procedure 54.
43. Mass a 50mL beaker
44. Add an ice-water mixture to the beaker
45. Place the beaker in the 100mL beaker containing the crude caffeine from part 1
46. Heat beaker at a low setting. *Caffeine will sublime from the bottom and deposit fine white crystals.*
47. Heat until sublimation process is complete then set aside to cool

48. Remove inner 50mL beaker and decant off ice-water mixture Dry the inside of the 50mL beaker. **Careful not to get crystals wet.**
49. Mass beaker and sublimed caffeine, *place beaker upside down on the balance.*
50. Scrape the caffeine crystals from the bottom of the beaker with filter paper.
51. In a small test tube add approximately 2mL of ethyl alcohol. Add purified caffeine and dissolve. Save solution for procedure 54.
52. Obtain Chromatography paper from Flinn Scientific.
53. Using a pencil and ruler gently draw a faint line 0.5cm from the bottom of the paper.
54. Gather test tubes containing the solution of caffeine and the purified sample from steps 42 and 51.
55. Use micro-tip pipet to place one small drop of caffeine standard solution on the place at the CAF location. Let spot dry completely. Place another drop on top of the now dry spot. Repeat this procedure until 6-8 drops have been placed on each other.
56. Repeat step 55 with the caffeine sample.
57. Add ethyl acetate to a jar where it covers the bottom and has a height of 0.3cm.
58. Without moving the liquid, form tent-like structures with Chromatography papers back to back.
59. Cover experiment and leave for develop process. **Must be covered for 30 minutes.**
60. When ethyl acetate has moved within 1cm from the top of the papers, remove and place on paper towel until they dry. **At the same time.**
61. Locate the solvent front location with a pencil.
62. View the paper using UV light and use a pencil to place a dot in the center of each spot
63. Measure the distances in cm starting from the bottom of the paper.

DATA ANALYSIS

Table 1 and Table 2 calculations are for Earl Grey and Lipton teas, respectively.

Table 1. Percent Caffeine Recovery from Earl Grey Tea (Commercial)

Trial 1	Sample Travel (cm)	Solvent Travel (cm)	Retardation Factor Rf
Caffeine Standard	2	4	Rf = 0.5
Caffeine Extract	2	4	Rf = 0.5
Mass Caffeine Available (g)	0.26	Percent Caffeine Recovery Trial 1	12%
Trial 2	Sample Travel (cm)	Solvent Travel (cm)	Retardation Factor Rf
Caffeine Standard	6.3	12	Rf = 0.53
Caffeine Extract	6.3	12	Rf = 0.53
Mass Caffeine Available (g)	0.27	Percent Caffeine Recovery Trial 2	12%
Average Mass Caffeine Available (g)	0.265	Average Percent Caffeine Recovery	12.0%

Table 2. Percent Caffeine Recovery from Lipton Tea (Commercial)

Trial 1	Sample Travel (cm)	Solvent Travel (cm)	Retardation Factor Rf
Caffeine Standard	5	7	Rf = 0.71
Caffeine Extract	5	7	Rf = 0.71
Mass Caffeine Available (g)	0.35	Percent Caffeine Recovery Trial 1	10%
Trial 2	Sample Travel (cm)	Solvent Travel (cm)	Retardation Factor Rf
Caffeine Standard	5.5	11	Rf = 0.5
Caffeine Extract	5.5	11	Rf = 0.5
Mass Caffeine Available (g)	0.24	Percent Caffeine Recovery Trial 2	11%
Average Mass Caffeine Available (g)	0.290	Average Percent Caffeine Recovery	10.5%

On the next page, Table 3 and Table 4 calculations are for Navajo and Decaffeinated teas, respectively.

Table 3. Percent Caffeine Recovery from Organic Navajo Tea (Native Grown)

Trial 1	Sample Travel (cm)	Solvent Travel (cm)	Retardation Factor Rf
Caffeine Standard	6	8	Rf = 0.75
Caffeine Extract	6	8	Rf = 0.75
Mass Caffeine Available (g)	0.42	Percent Caffeine Recovery Trial 1	8%
Trial 2	Sample Travel (cm)	Solvent Travel (cm)	Retardation Factor Rf
Caffeine Standard	6.2	7	Rf = 0.89
Caffeine Extract	6.2	7	Rf = 0.89
Mass Caffeine Available (g)	0.26	Percent Caffeine Recovery Trial 2	12%
Average Mass Caffeine Available (g)	0.340	Average Percent Caffeine Recovery	10%

Table 4. Percent Caffeine Recovery from Decaffeinated Green Tea (Commercial)

Trial 1	Sample Travel (cm)	Solvent Travel (cm)	Retardation Factor Rf
Caffeine Standard	2	4	Rf = 0.5
Caffeine Extract	2	4	Rf = 0.5
Mass Caffeine Available (g)	0.12	Percent Caffeine Recovery Trial 1	5%
Trial 2	Sample Travel (cm)	Solvent Travel (cm)	Retardation Factor Rf
Caffeine Standard	6.3	12	Rf = 0.53
Caffeine Extract	6.3	12	Rf = 0.53
Mass Caffeine Available (g)	0.15	Percent Caffeine Recovery Trial 2	3%
Average Mass Caffeine Available (g)	0.135	Average Percent Caffeine Recovery	4%

Table 5. Summary of Average Percent Caffeine Recovered

Type of Tea	Trial 1	Trial 2	Sample Average
Earl Grey Tea (Commercial)	12.0%	12.0%	12.0%
Lipton Tea (Commercial)	10.0%	11.0%	10.5%
Navajo Tea (Organic)	8.0%	12.0%	10.0%
Decaffeinated	5.0%	3.0%	4.0%

(Commercial)			

The caffeine recovery percentages for all Trials 1 and 2, Tables 1-4, as well as the overall averages for both trials are shown in Table 5 (above), and graphed in Figure 2 (below). From both the table of data, and the resulting bar chart of that data, it is clear that Earl Grey tea had the highest average caffeine (12.0%) followed by Lipton Tea (10.5%). Organic Navajo Tea, which is native grown, had the lowest average caffeine at only 10.0%, making it the healthy choice among caffeinated teas. The table and figure also showed that decaffeinated tea is not actually decaffeinated, but merely "reduced in caffeine," as caffeine was still recovered from the decaffeinated tea.

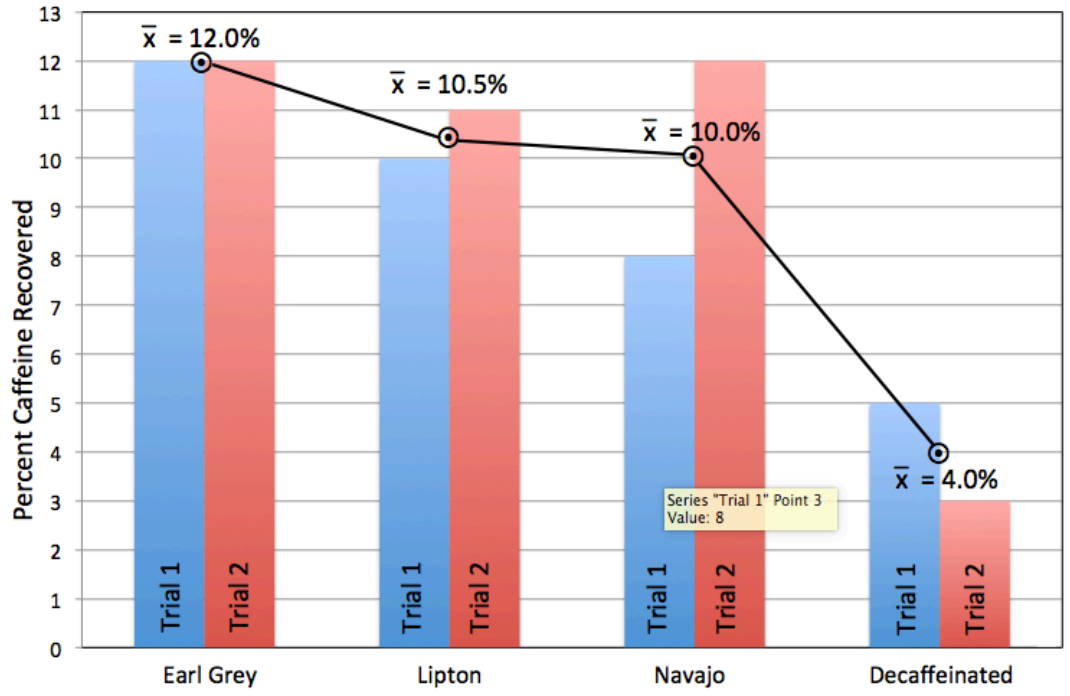

Figure 2. Percent Caffeine Recovered: Two Trials and Resulting Averages for three Caffeinated Teas and one Decaffeinated Tea

All the data in Tables 1-5 and Figure 2 is based on the retardation factor, Rf. The calculation of Rf is illustrated in Figure 3. The range of Rf is $0 \leq Rf \leq 1$.

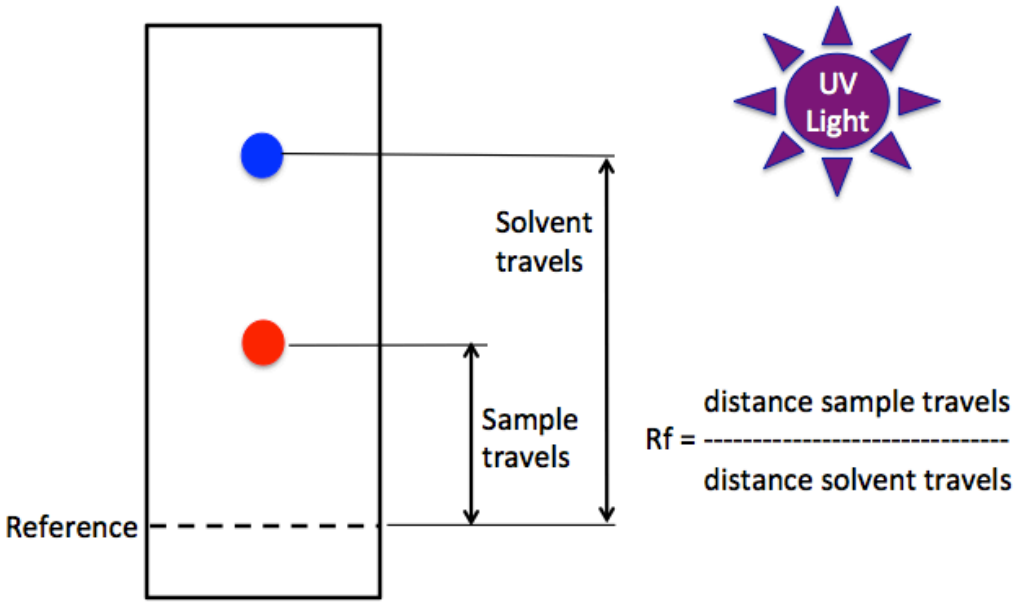

Figure 3. Chromatography Paper and Calculating the Retardation Factor Rf

RESULTS

The results of the extraction and isolation of caffeine experiment suggests that commercial teas, such as Earl Grey and Lipton, have higher average percent caffeine recovered when compared to Navajo Tea and Decaffeinated Green Tea. This is concluded from the data shown in Table 4. The average percent recovery for Earl Grey tea was 12.0% about 2.0% higher than Navajo Tea and three times (12/4) that of the Decaffeinated Green Tea. The average percent recovery for the Lipton tea was 10.5%, about 0.5% higher than Navajo Tea and about 2.6 times that of the Decaffeinated Green Tea. Likewise, Lipton Tea has a mass of 0.290 g of caffeine recovered which measures the residue that was collected from extraction. This is lower though compared to Navajo Tea. Navajo has the highest mass recovered of 0.340g. The percent recovery tells the amount of caffeine that the mass. Even though, decaffeinated green tea was tested, caffeine was still found, with an average percent caffeine recovery 4.0%, and 0.135g of caffeine.

DISCUSSION

The results found in this investigation provided an acceptance to the hypothesis. This showed that Earl Grey (commercial) had the highest average percent caffeine recovered (12.0%) followed by Lipton tea (commercial) (10.5%), and organic Navajo tea was the lowest of the three caffeinated teas at an average of 10%. Compared to other experiments like Trimle's, show that Lipton tea had an 18.8% of caffeine in one bag. This differs in this experiment with only an average of 10.5% of caffeine found in Lipton tea. However, this experiment differs from Trimble's by the amount of variables. Trimble had one variable, Lipton, and this investigation utilized 3 types of caffeinated teas. The use of sodium carbonate in the tea mixture was used to alter the gallic acid's chemical formula. If Sodium Carbonate was not used the gallic acid would change the results. By having the sodium carbonate, it also converts the tea to an inorganic salt. Limitations that would help find the highest caffeine yield would be the industrial solvent methylene chloride, which is very toxic and banned by the EPA for use in paint removers as of November 24, 2019. Other possible limitations that this investigation included the commercial teas chosen. Decaffeinated Green tea has a very little to no caffeine level according to the company, but a significant average percent of caffeine (4.0%) was detected nevertheless.

A suggestion for future caffeine studies is to test the different parts of the Navajo Tea plant, to compare which part of the plant has the most caffeine. Also, to test younger Navajo Tea plants versus older ones. And to not only use the traditional Navajo tea but other teas as well. A factor to take into consideration is that the method for picking the Navajo Tea in this experiment could have been buying it, and not picking it, since the storage time may have an effect. Another consideration is that the Navajo Tea could be picked from different locations on the Navajo Reservation, as soil conditions may have an effect.

CONCLUSIONS

The results found in this investigation accepted the hypothesis that both commercial teas (Earl Grey and Lipton) had more caffeine than Navajo tea (organic, native grown). This means that drinking Navajo tea is healthier than commercial tea, which is good for the Navajos who consume it.

However, through this investigation I found out that decaffeinated green tea does in fact contain caffeine, with an average of 4.0% percent caffeine recovered. This shows that although decaffeinated labeled products infer there is no caffeine in them, a low percentage of caffeine can still be obtained. Thus, the trade name "decaffeinated," really means "reduced caffeine."

Works Cited

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