Extraction of Caffeine

Name:

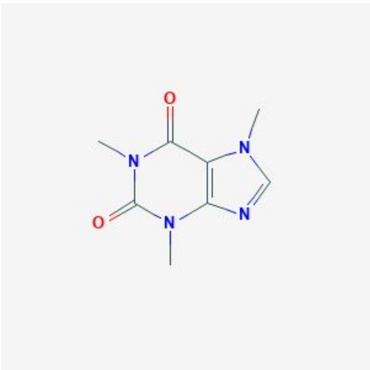
Date of Experiment:

TA:

Section:

Introduction:

The purpose of this experiment was to extract caffeine from tea leaves by separating it from other compounds found in the leaves, such as proteins and pigments. When the tea is brewed, the cellulose is not extracted as it is insoluble in water, and the other compounds are not extracted by dichloromethane due to their high water solubility.[1] By taking advantage of the different solubilities of the compounds, the caffeine can be isolated and extracted from the tea leaves.[2] The process of extracting caffeine is important as many people do not desire the effects of caffeine and prefer to drink decaffeinated beverages.[3]



Structure of caffeine[4]

Experimental Process:

Boiling distilled water (100mL) was added to a beaker along with three tea bags, which weighed 3.27g, 3.29g, and 3.31g respectively. The solution was stirred periodically for ten minutes, and the tea bags were removed and pressed to extract as much of the tea solution as

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[1] http://www.chem.ucalgary.ca/courses/351/laboratory/351expt_06_caffeine.pdf Organic Chemistry 351 Website, Isolation of Caffeine from Tea, October 10, 2019.
[2] http://www.chem.ucalgary.ca/courses/351/laboratory/351expt_06_caffeine.pdf Organic Chemistry 351 Website, Isolation of Caffeine from Tea, October 10, 2019.
[3] http://www.chem.ucalgary.ca/courses/351/laboratory/351expt_06_caffeine.pdf Organic Chemistry 351 Website, Isolation of Caffeine from Tea, October 10, 2019.
[4] https://pubchem.ncbi.nlm.nih.gov/image/imgsrv.fcgi?cid=2519&t=l, PubChem, Caffeine, October 10, 2019.

possible. The beaker was then allowed to sit in an ice water bath to cool to room temperature, and the cooled solution was then poured into a separatory funnel along with 20mL of dichloromethane. The funnel was shaken and the solution separated into an upper aqueous and lower organic phase. The lower phase was drained into a 125mL Erlenmeyer flask, and the extraction was repeated twice. The combined organic phases were then poured into the separatory funnel along with 20mL of 6M sodium hydroxide. After shaking the funnel and waiting for the layers to separate, the lower organic phase was drained and returned to the funnel along with 20mL of distilled water in order to repeat the extraction one more time. After the layers separated, the lower organic phase was drained into a 250mL Erlenmeyer flask. Anhydrous magnesium sulphate (1 teaspoon) was added to the flask and the flask was swirled. The magnesium sulphate settled at the bottom of the flask, indicating that the solution was not dry. Another teaspoon of magnesium sulphate was then added and the flask was swirled again, and the magnesium sulphate formed a fine powder. The drying agent was then removed through gravity filtration into a 250mL Erlenmeyer flask. The solution was poured into a 100mL round-bottom flask, which weighed 48.09g, and the dichloromethane was removed using a rotary evaporator. The flask was weighed again, this time containing the crude product, and had a weight of 48.123g, which meant that the crude product weighed 0.033g. Approximately 2.0mL of hot acetone was then added to the flask in order to dissolve the crude caffeine, whilst the flask was being heated in a steam bath. The flask was quickly removed and allowed to cool in an ice-water bath, however the solution did not recrystallize in time.

<u>Results:</u>

<u>Weight of tea bags:</u>

- 3.27g
- 3.29g
- 3.31g

*Paper of one tea bag weighs 0.15g

<u>Weight of tea leaves:</u>

- 3.27g - 0.15g = 3.12g
- 3.29g - 0.15g = 3.14g
- 3.31g - 0.15g = 3.16g

Total weight of tea leaves: 3.12g + 3.14g + 3.16g = 9.42g

Weight of 100mL round-bottom flask: 48.09g

Weight of 100mL round-bottom flask with crude product: 48.123g

Weight of crude product: 48.123g - 48.09g = 0.033g

Crude weight = $(0.033g \div 9.42g) \times 100\% = 0.35\%$

Discussion:

According to the experimental results that were obtained, the crude weight percentage of caffeine found in tea was 0.35%. However, a pure product of caffeine was not obtained because the solution containing caffeine and hot acetone did not crystallize, even after cooling it down in an ice bath. This was possibly because the solution was not initially heated in a steambath for the minimum duration required, or that it required more time to cool down in the ice bath. To avoid such a mistake in future experiments, one should be more concise in carrying out experimental procedures to avoid any mistakes and thus ensure that there is enough time near the end to carry out any final steps. Furthermore, when the anhydrous magnesium sulphate was added to the organic solution, it settled at the bottom of the flask in solid clumps. This indicated that the solution contained water, and thus another teaspoon of drying agent had to be added. The presence of water in the solution could have been a result of water being extracted along with dichloromethane in the liquid-liquid extraction. This is because there was an emulsion between the organic and aqueous layers which was hard to break, and therefore some of the water might have been extracted along with the organic layer into the flask.

Conclusions:

The techniques of liquid-liquid extraction and rotary evaporation were used to extract the caffeine from the tea leaves. The crude product that was collected had a weight of 0.033g, accounting for a crude weight percentage of 0.35%. A pure product was not obtained, possibly because the hot acetone solution was not heated for the right duration or it did not have enough time to cool down in the ice bath and recrystallize. This can be avoided in future experiments by ensuring that one is more prepared and concise when carrying out experimental procedures, to minimise the chances of errors and thus ensure that the entire experiment can be carried out within the time allotted.

Questions:

1. Because during the liquid-liquid extraction, some of the water might have been extracted along with the organic solution, so a drying agent is used in order to remove the water. The solution is then placed in a rotary evaporator to extract all other compounds that share a similar boiling point, leaving behind the desired product.
2. When sodium chloride is added to the solution, it dissolves in the aqueous layer and thus increases the aqueous layer's ionic strength. As a result, the water molecules in the emulsion become attracted to the aqueous layer due to its ionic strength, which causes the emulsion to break as the water molecules separate from the organic phase.

References:

1. http://www.chem.ucalgary.ca/courses/351/laboratory/351expt_06_caffeine.pdf Organic Chemistry 351 Website, Isolation of Caffeine from Tea, October 10, 2019.
2. https://pubchem.ncbi.nlm.nih.gov/image/imgsrv.fcgi?cid=2519&t=l PubChem, Caffeine, October 10, 2019.