Lab Report: Isolation Of Caffeine

Section 12-02
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Abstract:

Caffeine can be extracted from tea by its ability to be better dissolved in dichloromethane than water. By using separatory funnel, the caffeine was separated from tea and coffee dissolved in the dichloromethane solution, splitting into two layers so the dichloromethane solution was later heat to extract only caffeine.

Objective:

To extract caffeine from tea and coffee and check its purity by using Thin Layer Chromatography (TLC).

Introduction:

Caffeine, nitrogen-containing basic compounds, is alkaloid and has a bitter taste that we extracted from tea plants and coffee. Its IUPAC name is 1,3,7-trimethylpurine-2,6-dione, $C_8H_{10}N_4O_2$ [1] . In plants, caffeine are pesticides ; nicotine found in tobacco is sprayed onto other plants. Presenting in many ingested products, it affects human's metabolism ,stimulating the heart, respiration,and the central nervous system which makes you alert. [2] This substance is addictive and cause nervousness, insomnia - difficulty falling or staying sleep, and headaches when a person is about to withdraw it.

The origin of tea came from China. The small-leaved China plant and the large-leaved Assam plant are widely used ,as well as their hybrids. Fermented tea is called black tea while unfermented tea is called green tea ,and partially fermented tea is called oolong.

The majority in tea leaves are cellulose, a water-insoluble polysaccharides, and other substances containing in the leaves are caffeine, tannins, and chlorophyll. In this experiment, the water soluble material, which is caffeine, in the tea leaves are extracted. Caffeine is extracted by using dichloromethane or methylene chloride, an organic solvent that is insoluble in water, when the solution is cooled down because caffeine dissolves in this solution (140 mg/ml) more than in water (22 mg/ml). However, tannins ,phenolic compounds that have hydroxyl group attaching to an aromatic ring, are slightly soluble in dichloromethane.In order to separate tannins from caffeine that is also soluble in dichloromethane, sodium carbonate is added to the water ,turning tannins to phenolic anions which are highly dissolved in water. Therefore, tannins remain in water (the upper layer in the separatory funnel) while caffeine is mixed with dichloromethane (the lower layer). In fact, there is a drawback when tannins are converted into phenolic anions because they eventually become anionic surfactants. Anionic surfactants are molecules with nonpolar tail and negatively polar head that mix nonpolar molecule - dichloromethane in this case- with polar molecule which is water, causing a layer of emulsion in the separatory funnel. [3] Thus, during the extraction of caffeine from the water into dichloromethane, only agitate the solution without shaking it forcefully. Moreover, in case there is minor amount of water mixing in the dichloromethane , anhydrous sodium sulfate is added to the Erlenmeyer flask containing extracted caffeine to absorb the leaked water ,and it helps breaking emulsion forming in the flask as well.

Materials:

1. Coffee and Tea solution
2. 30 milliliters of dichloromethane
3. 6 teaspoons of Sodium sulfate
4. 15 mL Erlenmeyer flask
5. Separatory funnel
6. Graduated cylinder
7. Rotary evaporator
8. Melting point apparatus

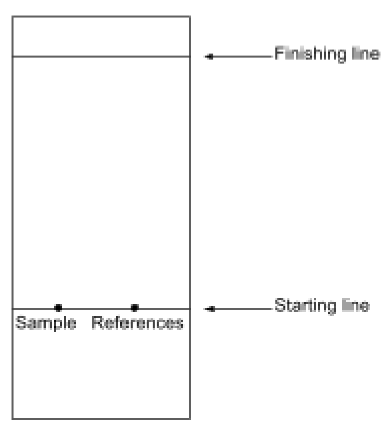
Procedure:

Caffeine Extraction

1. Set a separatory funnel
2. Put 15 mL of coffee and tea solution in a separatory funnel
3. Put 15 mL of dichloromethane in the separatory funnel with coffee and tea solution.
4. Stopper the separatory funnel. Hold the stopper into the neck of the funnel tightly and gently shake it. Then, turn the stopcock away from body and open the stopcock to release pressure. Gently shake it and release pressure for several times until the solution and dichloromethane mix together. Close the stopcock.
5. Put the funnel back to the stand and allow the mixture to settle until it separates into two layers.
6. Drain the lower layer, mixed dichloromethane layer into a Erlenmeyer flask carefully. Do not drain any drop of the upper layer in the flask.
7. Repeat step 4 to 6 one more time.
8. Add 6 spatulas of sodium sulfate into the flask with mixed dichloromethane and swirl it.
9. Filtrate mixed dichloromethane into a micro filter flask using Hirsh with filter paper.
10. Transfer mixed dichloromethane to a pot of a rotary evaporator. Turn on the instrument to separate dichloromethane and caffeine.

Thin-layer Chromatography

1. As the picture on the right, on the chromatography plate, using pencil, draw 2 lines for starting and finishing line. Draw two spot on the starting line for sample and references. (Be careful not to touch the plate surface!!)
2. Put sample and reference of pure caffeine on the spot drown.



3. Put the TLC plate into a solvent, ethyl acetate.
4. Let it the solution runs until reaching the finishing line. Take if off and let it dry.
5. Stain or expose the TLC plate to iodine vapor.

Measuring Melting point

1. Load dried caffeine reference powder into a capillary tube and tap the capillary tube on a hard surface until the sample packs into the bottom.
2. Place the capillary tube into the melting point apparatus and turn the heating stage on.
3. Carefully observe the sample through the eyepiece and note down the temperature when the sample starts to melt.

Results:

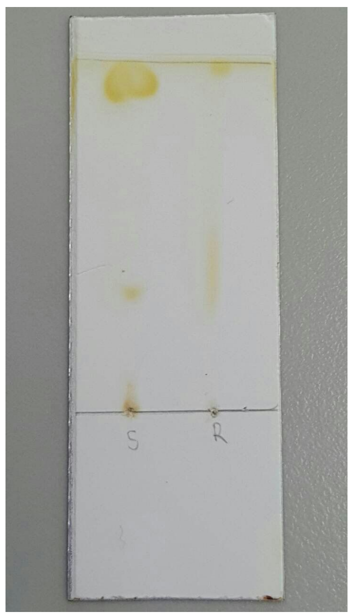


Figure 1: The result of Thin Layer Chromatography comparing the sample and the reference of extracted caffeine shows that the sample and the reference were carried up to the same position. Therefore, their abilities of being absorbed were the same, showing that the caffeine is pure.

Figure 2: After heating the sample at around its melting point (235 degree celsius), the sample was burnt before melting.

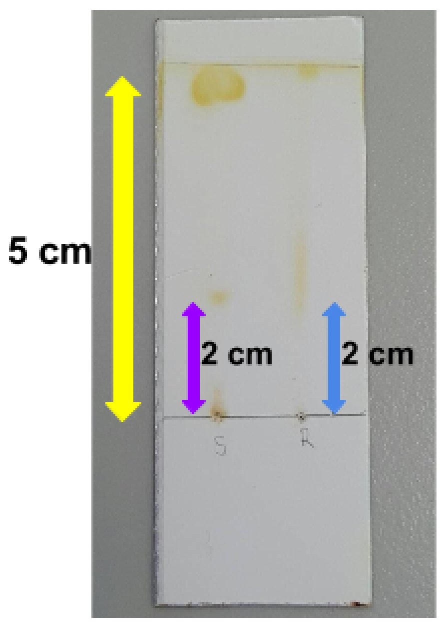
Discussion

Extracting caffeine with the use of dichloromethane from the coffee and tea leaves ,which consist not only caffeine but also tannins which is slightly soluble in dichloromethane, sodium bicarbonate is added to react with tannins and turn it to its salt or phenolic anions. This form enables tannins to totally dissolve in water instead of dichloromethane. Because caffeine with dichloromethane and tannins with water do not form homogenous mixture, due to their differences in polarity and density, two layers present in the separatory funnel.The tannins solution remains above the dichloromethane solution, so by turning the stopcock we will get the dichloromethane solution easier.As a result of emulsion forming between the two layer, anhydrous sodium sulfate was added to absorb possibly leaked water and to break the emulsion caused by turning tannins to surfactants.After that, the caffeine was separated from the dichloromethane by using rotary evaporator. Dichloromethane would be evaporated first since its boiling point is in the range of 30 and 40 degree celsius while caffeine's boiling point is 173 degree celsius-a lot higher than that of dichloromethane- so the caffeine solution remained in the container.

In this experiment, we extracted substances in coffee and tea solution to test whether if there was caffeine in it. From the results, it was indicated that it was caffeine since the similarity of result from thin-layer chromatography could be seen; however, there were unexpected errors in the experiment. The errors could be separated into two stages: errors of TLC stage and of melting point measurement. First, to test using TLC, the caffeine sample was overloaded on the plate, seen as a smear. Also, because the plate was taken out too long, the color from iodine staining began to disappear because the sample was invisible under UV light. Therefore, it might not be seen so clearly. Moreover, we could not do the calculation of TLC because we had used iodine to stain the result, in which iodine was radioactive so it was dangerous. Second, in measuring its melting point, we were to compare melting point of the caffeine reference and the sample extracted, but we could not. The sample was not dry enough to check for its melting point since we needed it in powder form to be loaded to the bottom of a capillary tube. When it was still wet, it got stuck and was not able to go to the bottom. Therefore, we could not check the melting point of the sample to compare to the one of the reference.

However, the errors presented in this experiment were good lessons for us to improve to further experiments in the future. We learned from this experiment that we should drop less sample on TLC in order to get a good result and minimize errors. Moreover, other experiments we can do in the future based on the results from this experiment is to determine whether the extracted caffeine is pure or not. Also, we may conduct the experiment further by testing the purity of extracted caffeine from different brands of coffee bag selling in market.

Calculations



In this experiment we used iodine to help us see the result on TLC, however the iodine was a harmful chemical so actually we couldn't measure the real distance traveled by the solvent and the distance traveled by the compound. Therefore, we assumed the approximate values in order to show an example of how to calculate the retention factor from TLC. According to the result of Thin Layer Chromatography, the distance traveled by the compound of the sample is around 2 cm while the distance traveled by the solvent front is 5 cm so from the retention factor formula; we divide the distance traveled by the compound by the distance traveled by the solvent front which is equal to 0.4 cm. For the reference, we also got the same number of distance traveled by the compound which is around 2 so the retention factor of the reference is also equal to 0.4 cm.

Conclusion

Based on the result, substance that was extracted from coffee and tea solution was caffeine due to its ability of being absorbed using TLC, which was similar to the reference.

References

1. http://pubchem.ncbi.nlm.nih.gov/compound/caffeine#section=Top
2. http://www.webmd.com/sleep-disorders/guide/insomnia-symptoms-and-causes
3. http://www.rsc.org/learn-chemistry/resources/chemistry-in-your-cupboard/finish/6
4. The laboratory instruction: Experiment #6 - Isolation Of Caffeine From Tea Leaves