CAFFEINE EXTRACTION FROM COFFEE

[1] INDRALI SARPOTDAR, [2]AROCKIA DIXON, [3]DEVESH SANE, [4]ASHWINI THOKAL

[1-3] Undergraduate Students at Bharati Vidyapeeth College of Engineering,
[4]Assistant Professor at Bharati Vidyapeeth College of Engineering,
[1]Department of Chemical Engineering,
[1]Bharati Vidyapeeth College of Engineering, Navi Mumbai, India.

Abstract: Caffeine extracted and characterized from coffee powder. Isolation was done by liquid-liquid extraction using dichloromethane as an extracting agent. This extraction was done in four steps: liquid-liquid extraction, drying, freezing, and vacuum filtration. The drying was done using anhydrous sodium sulphate. High-performance liquid chromatography and Melting point were the techniques used to analyze the purity of the obtained product. First, the analysis was done using melting point analysis. The melting point of caffeine extracted from the coffee powder was found to be 238°C. The absorption bands were compared with that available in the literature and were found to be similar. Further, the purity check was done using the High-performance liquid chromatography method. Effective characterization of caffeine was achieved by determining the Infrared spectrum and employing a melting point apparatus, The purity showed that the results that the extracted coffee was 90% pure. Further improvements in extraction efficiency will increase the yield and minimize wastage.

Key Words - Caffeine, Dichloromethane, High-performance liquid chromatography, Melting point analysis.

I. INTRODUCTION

CAFFEINE

Caffeine is an alkaloid compound that is found especially in tea and coffee plants and is a stimulant of the central nervous system belonging to the methylxanthine class. It is a class of naturally occurring compounds containing nitrogen and having the properties of an organic amine base. Hence, caffeine is an intensely bitter and white powder in its pure form. It is odorless and is composed of long hexagonal prisms. The formula for caffeine is $C_8H_{10}N_4O_2$

ALKALOID

Alkaloid is a class of nitrogenous organic compounds that originate from plants and has been shown to have physiological actions on humans.
Alkaloids show the nature of being colorless, crystalline, non-volatile, solid. There are a few liquid alkaloids such as coniine and nicotine, and a few colored alkaloids such as yellow berberine.

PROPERTIES OF ALKALOID

• The free alkaloids dissolve in ether, chloroform, and the salts in other non-polar solvents are insoluble.
• The alkaloids usually taste bitter.
• Alkaloids are usually insoluble or sparingly soluble in water but their salts with acids freely dissolve in water.

HOW DOES THE CAFFEINE WORK?

Caffeine invigorates the central nervous system (CNS), heart, muscles, and the centers that control blood pressure.
Caffeine can cause a rise in blood pressure sometimes but might not affect people who consume caffeine daily.
Caffeine can also act like a "water pill" that causes an increase in urine flow. Also, drinking caffeine during moderate exercise does not cause dehydration.

GENERAL PROPERTIES AND SOLUBILITY OF CAFFEINE

- Pure anhydrous caffeine is a bitter-tasting white
- odorless powder
- Melting Point – 235 to 240ºC
- Partly soluble in water at room temperature.
- Moderately soluble in ethanol.
- Density – 1.23g/cc, solid

GENERAL INFORMATION

CAFFEINE is chemically known as 3,7-Dihydro-1,3,7-Trimethyl-1H-purine-2,6-dione ; 1,3,7-trimethylxanthine ; 1,3,7-trimethyl-2,6-dioxopurine ; caffeine ; thein ; guaranine ; methyl theobromine . The molecular formula of CAFFEINE is $C_8H_{10}N_4O_2$. The molecular weight of caffeine is 194.19 wherein carbon is 49.48%, hydrogen is 5.19%, nitrogen is 28.85%, oxygen is 16.48%.

FUNCTIONAL GROUP ANALYSIS

Let's look first at the 5-membered ring. The two N atoms are part of amine groups. The C=C and C=N bonds look like functional groups, but they are neither alkene nor imine groups. They are both parts of the 5-membered system called an imidazole ring. An imidazole ring is aromatic, like benzene. Now let's look at the 6-membered ring. The C=O and N groups look like ketone and amine groups, but they are not. A C=O next to an N is an amide group. There are two amide groups in the ring. So caffeine contains two amine and two amide functional groups.

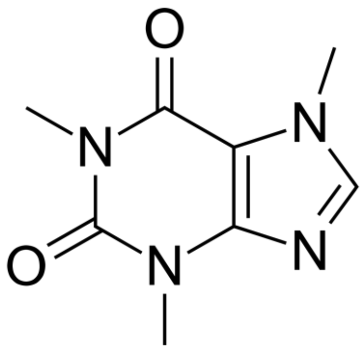

Fig No. 1 Structure of Caffeine

The caffeine content of a single serving can vary greatly depending upon the type of coffee and the method of preparation. It mainly depends on the brewing method and the coffee type. According to the National Nutrient Database, a 240-milliliter (8 US fl oz) cup of "coffee brewed from grounds" contains 95 mg caffeine, whereas an espresso (25 ml) contains 53 mg.

According to an article in the *Journal of the American Dietician Association*, coffee has the following caffeine content, depending on how it is prepared

Table1. Caffeine Content in different types of coffees according to their serving sizes

	Serving size	Caffeine content
Brewed	200 mL (7 US fl oz)	80–135 mg
Drip	200 mL (7 US fl oz)	115–175 mg
Espresso	45–60 mL (1 ½–2 US fl oz)	100 mg

As the roast level increases the fraction of caffeine content in coffee seeds themselves diminishes, whereas the opposite is true for coffee brewed from different grinds and brewing methods using the same proportion of coffee to water volume. The coffee sack (similar to the French press and other steeping methods) extracts more caffeine from dark roasted seeds; the percolator and espresso methods extract more caffeine from lightly roasted seeds.

Table2. The caffeine content in coffees according to their roast level

	Light roast	Medium roast	Dark roast
Coffee sack – coarse grind	0.046	0.045	0.054
Percolator – coarse grind	0.068	0.065	0.060
Espresso – fine grind	0.069	0.062	0.061

II. MATERIALS AND METHODS

EXTRACTING CAFFEINE FROM COFFEE

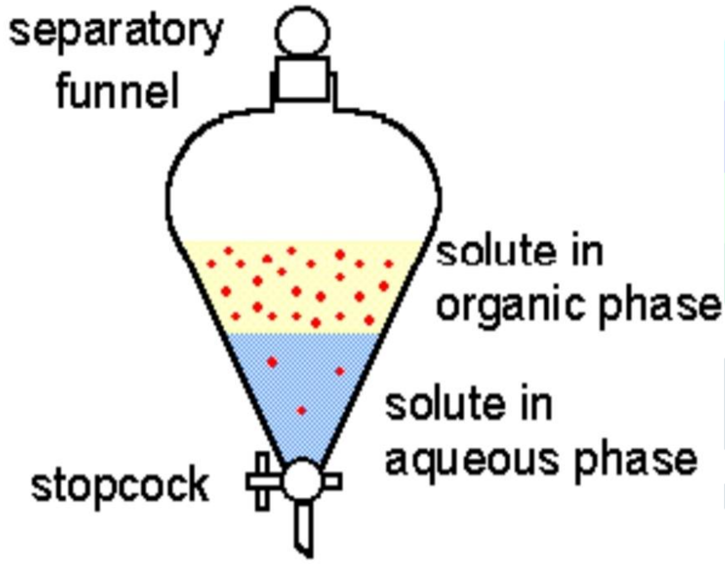

Fig.2: Experimental Diagram

The above diagram shows the process of liquid-liquid extraction of coffee (mother liquor) and the caffeine dissolved in DCM. In the process of extraction of caffeine from coffee, a separatory funnel is used for liquid-liquid extraction. Coffee is poured into the separatory funnel and to it, DCM is added. As a result, two layers are obtained – the upper layer of coffee and the lower layer of caffeine which is dissolved in the DCM.
The separatory funnel is provided with a stopcock to prevent the solution from overflow. When the bottom layer is demanded to be removed, the stopcock is adjusted in such a way that the solution flows out. When the bottom layer has been completely extracted the stopcock is closed to prevent the mother liquor (coffee) from flowing down the separatory funnel.

The procedure along with some snapshots are depicted as follows:
To extract caffeine from coffee, several techniques are accompanied. To get the solid natural product into the liquid solvent, first solid-liquid extraction must take place. This can be done by boiling the coffee powder with the addition of sodium carbonate as a base. The solution is required to cool for filtration purposes. Here the filtration is done using coffee filters or a household strainer.

Fig3. Solid-liquid Extraction

Liquid-Liquid extraction is used to separate caffeine in the organic layer. The solvent used for solid-liquid extraction is sodium carbonate whereas solvent used for liquid-liquid extraction is Dichloromethane (CH2Cl2) (Note- Dichloromethane can irritate your skin so do not handle Dichloromethane bare hand)

Fig4. Liquid-liquid Extraction

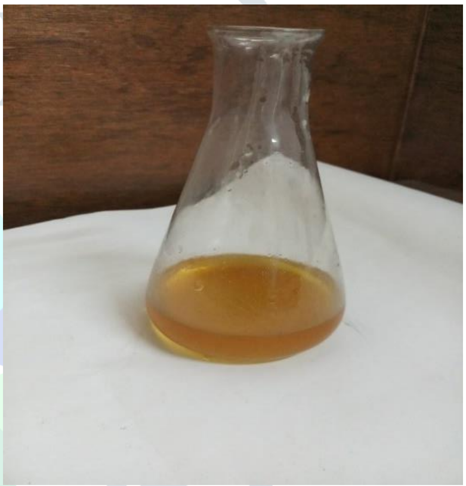

Fig5. Sodium sulphate added to the DCM layer

Once the solvent is added to the solution, two separate layers are formed. The lower layer consists of DCM in which the caffeine is dissolved. Once the lower layer is extracted from the separatory funnel, a saturated salt solution is added to the solution which absorbs all the water from the solvent. Now the next step is to add sodium sulfate to the extracted layer for the purpose of drying. The solution is then kept at rest for 20 minutes. After the layer has been dried the remaining liquid portion is then heated for it to be evaporated. The evaporation of the liquid results in the raw caffeine in a dry manner (crude caffeine). To obtain pure caffeine we need to add 95% ethanol to the obtained crude caffeine, it is then heated and frozen later. The frozen solution is then vacuum filtered and the final product obtained is raw caffeine. This is a quick and easy way to purify caffeine.
The success rate of natural product extraction is usually expressed as a percentage of the recovery rate,

$$\%\text{Recovery} = \frac{\text{Grams of caffeine Recovered}}{\text{Grams of coffee}}$$

The percentage recovery is called the purified percent recovery or crude percent recovery. The extraction with the highest percent recovery is considered the most successful extraction.

III. RESULTS AND DISCUSSION

ANALYSIS TECHNIQUES FOR CAFFEINE

Thin Layer Chromatography (TLC)
1. Different solutes have different solubility in a solvent /different solutes have different degrees of a tendency to be dissolved in the same solvent.

2. As the solution (contains the solvent with the dissolved solutes) moves along a stationary solid surface (a solid surface), different solutes adsorbed onto the solid surface to a different extent as they have different degree of adsorption characteristics (due to the different degrees of dissolve tendency)
3. The “less soluble” solute will be retained first, and the “more soluble” solutes will be retained afterward.
4. Different solutes will then be separated at different positions of the solid surface.
5. Retention Factor (RF) of each component is calculated as follows

$$Rf= \frac{\text{Grams of caffeine RecoveredDistance traveled by the component substance from the baseline}}{\text{Distance traveled by the solvent from the baseline}}$$

Pure caffeine and the extract are analyzed in the same TLC plate.

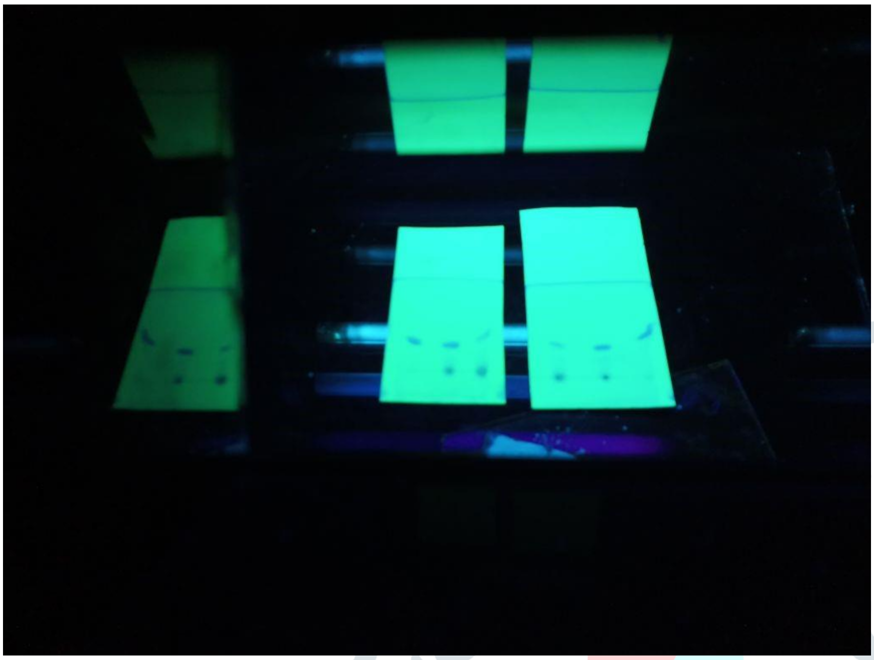

Fig6. Caffeine samples on the TLC plate

CAFFEINE CONTENT

40 mg raw caffeine was obtained from 10 gm coffee.

Fig7. Raw Caffeine

pH OF CAFFEINE

Two pH tests of crude caffeine and pure caffeine were carried out.
The pH range of pure caffeine is between 6-7.
The pH range of crude caffeine is between 7-8.

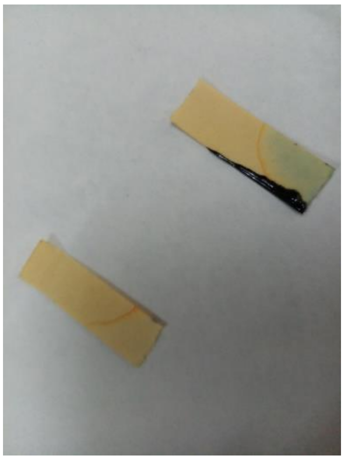

Fig8. pH Test of Caffeine Sample

DENSITY OF OBTAINED CAFFEINE

The density of the obtained caffeine is 1.23 g/cc.

COLOUR OF THE OBTAINED CAFFEINE

The color of the obtained caffeine is whitish-ochre.

ODOUR OF THE CAFFEINE PRODUCED

The obtained caffeine is odorless.

IV. CONCLUSION

Coffee is a brewed drink prepared from roasted coffee beans, which are the seeds of berries from the *Coffea* plant. Coffee is slightly acidic and has a stimulating effect on humans because of its caffeine content. In this study, coffee will be decaffeinated using dichloromethane as a solvent. This study will be carried out to check the amount of caffeine in used coffee. It is acceptable that the amount of caffeine decreased with every use. Caffeine from coffee is extracted by liquid-liquid extraction followed by recrystallization. Caffeine is the most commonly used psychoactive drug in the world. It is a pharmacologically active substance and depending on the dose, can be a mild central nervous system stimulant. Approximately 80% of the world's Population Consumes Caffeine on daily basis. The purified caffeine is then analyzed by using high-performance liquid chromatography or the Iodometric back titration method. The serious concern about the potential use of caffeine for pathogenic effects has made it one of the most broadly studied drugs. In the present study Caffeine content of different coffee, samples were studied and it is found that the caffeine content varies from 1-5%. The values generally agree well with literature quoted values of 2-5%. In the series of experiments that have been conducted, we can conclude that the caffeine content of coffee is relatively high as compared to other beverages and therefore we can also state that the caffeine is highly soluble in Dichloromethane as compared to other solvents and also the Tannins are more soluble in sodium carbonate as compared to other bases.

V. REFERENCES

1. Lisa Nichols (Butte Community College). *Organic Chemistry Laboratory Techniques* is licensed under a Creative Commons Attribution-Non Commercial-No Derivatives.
2. Batey, W., Thompson, P. J., and Thornton, J. D. (1987) Column impedance and the hydrodynamic characterization of pulsed plate-columns. Extraction â€™87. *Instn. Chem. Engrs. Symposium Series*. 103: 133.
3. Jaafar, F. and Michalowski, S. (1990) Modified BET equation for sorption/desorption isotherms, *Drying Technol.* 8(4), 811-827.
4. Keey, R. B. (1978) *Introduction to Industrial Drying Operations*, Pergamon, Oxford.
5. K. Ramalakshmi and B. Raghavan* Department of Plantation Products, Spices, and Flavour Technology, Central Food Technological Research Institute, Mysore 570 013, India
6. Alenka RC, Nestler EJ, Hyman SE (2009). "Chapter 15: Reinforcement and Addictive Disorders". In Sydor A, Brown RY (eds.). *Molecular Neuropharmacology: A Foundation for Clinical Neuroscience* (2nd ed.). New York: McGraw-Hill Medical. p. 375.

7. Cano-Marquina A, Tarín JJ, Cano A. The impact of coffee on health. Maturitas 2013;75:7-21. 10.1016/j.maturitas.2013.02.002.
8. Jeszka-Skowron M, Zgoła-Grzekowiak A, Grzekowiak T. Analytical methods applied for the characterization and the determination of bioactive compounds in coffee. Eur Food Res Technol 2015;240:19-31 10.1007/s00217-014-2356-z.
9. Ludwig IA, Clifford MN, Lean MEJ, Ashihara H, Crozier A. Coffee: biochemistry and potential impact on health. Food Funct 2014;5:1695-717. 10.1039/C4FO00042K.
10. Varnam, A. H. and Sutherland, J. P., Beverages: Technology, Chemistry, and Microbiology, Chapman and Hall, London, 1994.
11. Wealth of India. Raw materials. A Dictionary of Indian raw materials of industrial products, Council of Scientific and Industrial Research, New Delhi, 1950.
12. Abraham, K. O. and Shankaranarayana, M. L., Roasting of coffee — quality changes and flavour development, J. Coffee Res., 1990.
13. Abraham, K. O., Coffee and Coffee Products, Guide on Food Products, Spelt Trade Publication Pvt. Limited, New Bombay, 1992.
14. Gilbert, R. M., The Methylxanthine Beverages and Foods: Chemistry, Consumption and Health Effects, Spiller G. A., Ed., Alan R. Liss, 1984.
15. Roger, P. J. and Richardson, N. J., Why do we like drinks that contain caffeine, Trends Food Sci. Technol., 1993.