



नॅशनल इन्स्टिट्यूट ऑफ एज्युकेशन,
सहकारभूषण एस.के.पाटील, महाविद्यालय कुरुंदवाड
ता.शिरोळ, जि.कोल्हापूर.
भूगोलशास्त्र विभाग



प्रमाणपत्र

प्रमाणित करण्यात येते की, कु/कुमारी ...साळी.साहिब.ना.सु.
हा / ही महाविद्यालयाचा नियमित विद्यार्थी असून याने/हिने शैक्षणिक वर्ष 2020-2021 मध्ये
शिवाजी विद्यापीठ कोल्हापूर यांच्या अभ्यासक्रमानुसार प्रात्यक्षिक/ शैक्षणिक सहल/ खेडे
सर्वेक्षण कार्य पूर्ण केले असून याचा / हिचा अहवाल शास्त्रीय स्वरूपात व व्यवस्थित रित्या
सादर केला आहे.

दि:/...../...../2021

Dijay
विषय शिक्षक



R. S. N.
विभागीय प्रमुख
सहकारभूषण,
कुरुंदवाड.



SHIVAJI UNIVERSITY KOLHAPUR



NATIONAL INSTITUTE OF EDUCATION

SAHAKARBHUSHAN S K PATIL COLLEGE KURUNDWAD TAL

SHIROL DIST KOLHAPUR

A PROJECT ON

“Extraction of Caffeine from Tea Powder”

SUBMITTED BY

Student Name :

PRN No.

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4.MISS. GIRIJA DILIP KILLEDAR

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Under The Guidance of

Miss Mohite D.S

To

Department Of Chemistry

2020-21

NATIONAL INSTITUTION OF EDUCATION
SAHAKARBHUSHAN S.K.PATIL COLLEGE KURUNDWAD TAL SHIROL DIST
KOLHAPUR

DEPARTMENT OF CHEMISTRY

CERTIFICATE

This is to certify that,

1. MISS. AKSHATA BABURAO GHODAKE

2. MISS. ANUSHKA BALVANT JADHAV

3. MISS. MAYURI AJIT BAIRAGI

4. MISS. GIRIJA DILIP KILLEDAR

Students of the class **Ty.B.Sc** Satisfactorily completed the project work on "**Extraction of Caffeine from Tea Powder**" the of year 2020-21. As a partial fulfillment of the practical course for the award of the **B.Sc** Degree in Chemistry by Shivaji University Kolhapur.

Place:- Kurundwad

Date:- 07/08/2021

Project Guide

Examiner

Miss Mohite D.S
Head of Department
S.K Patil Mahavidyalya,
Kurundwad.

the different degrees of dissolve tendency) 3. The “less soluble” solute will be retained first, and the “more soluble” solutes will be retained afterwards. (Note: No two substances have the same solubility and adsorption characteristics. 4. Different solutes will then be separated on the different positions of the solid surface. 5. Retention Factor (RF) of each component is calculated as follow $Rf = \frac{\text{Grams of caffeine Recovered}}{\text{Distance travelled by the component substance from the baseline}}$ Distance travelled by the solvent from the baseline Pure caffeine and the extract are analyzed in the same TLC plate and compare any differences of their Rf.

- **Spike Test :**

By adding known amount of standard caffeine in distilled water and raw coffee solution, then carry out solvent extraction. By comparing the extraction results, we can analyze the recovery percentage of the spiked caffeine and efficiency of solvent extraction.

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Iodometric Back Titration Caffeine reacts with excess accurately known amount of iodine in acidic environment, forming insoluble precipitate. Then the insoluble precipitate is removed by filtration. Using titration by a standard sodium thio-sulphate solution with starch solution as indicator, we can determine the amount of remaining iodine, and thus the amount of caffeine can be found. Here are the chemical equations: $C_8H_{10}N_4O_2 + 2 I_2 + KI + H_2SO_4 \rightarrow C_8H_{10}N_4O_2 \cdot HI \cdot I_4 + KHSO_4$ $I_2 + 2 Na_2S_2O_3 \rightarrow 2NaI + Na_2S_4O_6$ The Analysis Technique used here is iodometric back titration. The procedure is as follows: Caffeine solution was prepared using sulphuric acid as an acidic medium. Iodine was added to it and then the solution was titrated against sodium thiosulphate till the solution becomes pale brown in color. Then starch was added as indicator after the solution becomes pale brown. Again, the solution is titrated against sodium thiosulphate and the endpoint of the reaction is dark blue to colorless solutio

ACKNOWLEDGEMENT

The Project On “Extraction of Caffeine from Tea Powder” is obviously not only a result of our work done but also many personalities have been directly or indirectly involved in this project. Report completion would not have been possible.

It gives us a great pleasure to record our gratitude to our principal Dr. Y.M.Chavan and HOD Mrs. D.S. Mohite Project guide under Miss Nampalli P. A. and Miss S.S.Gaikwad for expert guidance and continuous inspiration throughout the course of project work.

I am also thankful to the Sahakarbhushan S K Patil College Kurundwad providing us chemistry laboratory for experimental work and needed information for this project.

Place: Kurundwad

Date:07/08/2021

Students Name

PRN No.

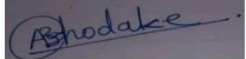
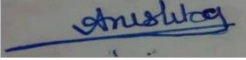
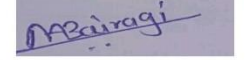
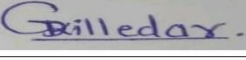
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DECLARATION

We underground student of B.sc III Chemistry department hereby declare that the project work we are submitting is our original work the topic of the project selected by us has not been previously formed the basis for the for the award of B.sc degree in chemistry.

Place: Kurundwad

Date : 07/08/2021

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INTRODUCTION

Caffeine is a naturally occurring chemical stimulant found in the leaves, seeds and fruits of a numerous plant species of a group of compounds called trimethylxanthine. Its chemical formula is $C_8H_{10}N_4O_2$. Caffeine is most commonly used to improve mental alertness, but it has many other uses. Caffeine is used by mouth or rectally in combination with painkillers (such as aspirin and acetaminophen) and a chemical called ergotamine for treating migraine headaches. It is also used with painkillers for simple headaches and preventing and treating headaches after epidural anesthesia.

1.1. Properties of Caffeine

- Systematic name: 1,3,7-trimethyl-1H-purine- 2,6(3H,7H)- Dio
- Other name: 1,3,7-trimethylxanthine & 1,3,7-trimethyl-2,6- dioxopurine
- Molecular formula: $C_8H_{10}N_4O_2$
- Molecular mass: 194.19 g/mole
- Melting point: 238°C
- Solubility in water: slightly soluble

1.2. Health Benefits of Caffeine

- Research indicates that caffeine may help protect human brain cells, which lowers the risk of developing some diseases, such as Parkinson's.
- Regular cups of coffee may stimulate the gallbladder and reduce the risk of gallstones.
- Caffeine causes the blood vessels to constrict, which may help relieve some headache pain.

- Coffee reduces inflammation and may help prevent certain heart related illnesses.
- Treats Migraine.
- Relieves Asthma Attack
- Increases the potency of analgesics.
- Caffeine is also used for weight loss and type 2 diabetes.
- Very high doses are used, often in combination with ephedrine, as an alternative to illegal stimulants
- Caffeine creams are applied to the skin to reduce redness and itching in dermatitis

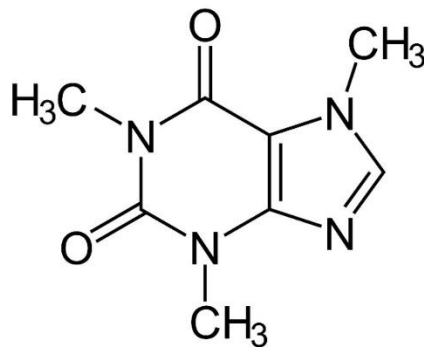
1.3. Adverse Effects of Caffeine

- There is a significant association between drinking caffeinated coffee and the decrease of bone mineral density, which leads to osteoporosis.
 - The daily consumption of caffeinated drinks can increase blood sugar levels and cause problems for people with diabetes.
 - Caffeine is a diuretic and can cause dehydration.
 - Caffeine can prevent some from falling asleep and interferes with deep sleep, which can lead to fatigue during the day.

The level of caffeine can vary depending on what is consumed. A piece of chocolate may have as little as five milligrams while energy drinks contain as much as 160 milligrams. Make sure to read the labels of pain medications and diet pills as products can have levels of caffeine as high as 200 milligrams. Michigan State University Extension recommends moderate doses of caffeine, 200 to 300 milligrams per day, which is equivalent to two to four cups of brewed coffee and is considered safe for most adults. If you are consuming more than 500 to 600 milligrams of caffeine per day, which equals four to seven cups of coffee, you may be prone to health problems including insomnia, nervousness, nausea or gastrointestinal problems, elevated heartbeat, headaches, etc. If you are experiencing unusual side-effects associated with the consumption of foods with caffeine, you should consult your physicia

INFORMATION

Caffeine works by stimulating the central nervous system (CNS), heart, muscles, and the centers that control blood pressure. Caffeine can raise blood pressure, but might not have this effect in people who use it all the time. Caffeine can also act like a “water pill” that increases urine flow. But again, it may not have this effect in people who use caffeine regularly. Also, drinking caffeine during moderate exercise is not likely to cause dehydration.



Structure of Caffeine

Table1. Caffeine Content of Common Food and Drugs

Espresso	120 mg per 2 Oz
Coffee, Regular, Brewed	103 mg per cup
Instant Coffee	57mg per cup
Coffee, Decaffeinated	2 to 4 mg per cup

Tea	30-75 mg per cup
Cocoa	5-40mg per cup
Milk Chocolate	6mg per Oz
Baking Chocolate	35mg per Oz
Coca-Cola Classis	46mg per 12 Oz
Jolt Cola	72mg per 12 Oz
Anacin Bromo Seltzer Midol	32mg per pill
Excedrin Extra Strength	65mg per pill
DexatrimDietacVivarin	200mg per pill
Dristan	16mg per pill
No-Doz	100mg per pill

In table 1 the mentioned beverages and drugs are frequently used and it reveals that among them, Espresso contains the maximum amount of caffeine as compared to other beverages and drugs. In its pure form, caffeine is a white crystalline powder that tastes very bitter. It is medically useful to stimulate the heart and also serves as increasing the rate of urine excretion. It is one of the most studied ingredients in the food supply. The most commonly known sources of caffeine are coffee and cocoa beans, guarana, and tea leaves. The amount of caffeine in food and beverage products varies depending on the serving size, the type of product and preparation method. Tea which we generally drink is made from the leaves of an Asian evergreen known as *Camellia sinensis*. The presence of caffeine in plants helps to prevent them from insects and other herbivores with the compound's bitter taste and stimulating qualities. The caffeine content of tea leaves depends on the variety and where they were grown; most tea has 3-5% by weight. The optical transition properties of caffeine were measured in different solvents (dichloromethane, water, chloroform and ethyl acetate). Caffeine has highest optical transitions in dichloromethane than the other solvents. Caffeine can be extracted more at the boiling temperature than at 30°C. Caffeine had been widely used in the food and pharma industry. The cost of extraction of caffeine from natural source is more. Research has been taken to extract it from natural source more economically.

2. MATERIALS AND METHODS

Liquid–liquid extraction (LLE) is a method to separate compounds or metal complexes, based on their relative solubilities in two different immiscible liquids, usually water (polar) and an organic solvent (non-polar). There is a net transfer of one or more species from one liquid into another liquid phase, generally from aqueous to organic. The transfer is driven by chemical potential, i.e. once the transfer is complete, the overall system of protons and electrons that make up the solutes and the solvents are in a more stable configuration (lower free energy). The solvent that is enriched in solute(s) is called extract. The feed solution that is depleted in solute(s) is called the raffinate. LLE is a basic technique in chemical laboratories, where it is performed using a variety of apparatus, from separatory funnels to countercurrent distribution equipment called as mixer settlers. This type of process is commonly performed after a chemical reaction as part of the work-up, often including an acidic work-up. Extraction is a method used for the separation of organic compound from a mixture of compound. This technique selectively dissolves one or more compounds into an appropriate solvent.

The solution of these dissolved compounds is referred to as the extract. In the case of Caffeine extraction from tea powder, the solubility of caffeine in water is 22mg/ml at 25°C, 180mg/ml at 80°C, and 670mg/ml at 100°C. Here the organic solvent Dichloromethane is used to extract caffeine from aqueous extract of tea powder because caffeine is more soluble in dichloromethane (140mg/ml) than it is in water (22mg/ml). The dichloromethane - caffeine mixture can then be separated on the basis of the different densities of dichloromethane and water because dichloromethane is much denser than water and insoluble in it. Residual water is separated from dichloromethane by drain out the dichloromethane through separating funnel, thus dichloromethane passed through the funnel while polar solvents such as water is still remaining in the funnel. In the first phase of experimentation screening was carried out in order to determine the maximum content of caffeine among black tea, green tea and coffee. The procedure was as follows: 10gm of tea, green tea and coffee sample was taken and boiled for 15 minutes along with the addition of 6gm of sodium carbonate which acts like a base which reacts with tannins to form sodium salts of tannins.

The final step of the experimentation is to determine what happens to the quantity and quality of caffeine when we change the base used during solid-liquid Extraction. For this again 10 gm of coffee is used and boiled along with the addition of sodium hydroxide as base instead of sodium carbonate. Then again vacuum filtration is carried out in order to separate the particles of coffee beans present. Then liquid-liquid extraction is used to separate caffeine in organic layer. Then organic layer obtained is kept for evaporation and the product obtained is compared. The procedure along with some snapshots are depicted as follows: In order to extract caffeine from tea, several techniques are accompanied. First, a solid-liquid extraction must take place in order to get the solid natural product into the liquid solvent. This can be done by boiling tea leaves with the addition of sodium carbonate as a base. Further to separate the tannins vacuum filtration is used.



After

Vacuum Filtration, Liquid-Liquid extraction is used to separate caffeine in organic layer. 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 handedly).

Now the next step is to keep the organic layer for evaporation of solvent which is dichloromethane.

After evaporation of solvent the left product is raw crude caffeine which is further sent to analysis

Note: The sodium carbonate acts as a base - you could use sodium hydroxide instead. When you boil tea leaves tannins dissolve in the water as well as the caffeine. If you do not use a base the tannins will also be extracted into the solvent (i.e. methylene chloride) used in the subsequent extraction. The base converts the tannins into their sodium salts - being ionic these salts are not soluble in solvents like methylene chloride so remain in the aqueous layer during extraction. This allows purer caffeine to be extracted. After carrying out repeated extractions and using vacuum filtration we get crude white crystalline caffeine as a product. Getting pure form of crystalline caffeine from crude caffeine, we need to carry out recrystallisation. Recrystallisation is a fast and easy way to purify the caffeine. The Success Of extraction involving a natural product is often expressed as percentage recovery, $\% \text{Recovery} = \frac{\text{Grams of caffeine Recovered}}{\text{Grams of tea leaves}}$ 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

2.1. Analysis Techniques for Caffeine

• Thin Layer Chromatography (TLC) :

There are different types of chromatographic methods such as paper chromatography, thin-layer chromatography, column chromatography, gas chromatography, etc. They have the same principle: Extraction of Caffeine Page | 6 1. Different solutes have different solubility in a solvent /different solutes have different degrees of 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 in 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 afterwards. (Note: No two substances have the same solubility and adsorption characteristics. 4. Different solutes will then be separated on the different positions of the solid surface. 5. Retention Factor (RF) of each component is calculated as follow $Rf = \frac{\text{Grams of caffeine Recovered}}{\text{Distance travelled by the component substance from the baseline}}$ Distance travelled by the solvent from the baseline Pure caffeine and the extract are analyzed in the same TLC plate and compare any differences of their Rf.

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Uses of Caffeine

1. In Medicine it is used to stimulate, central nervous system and to increase flow of urine.
2. Because of its stimulating effects, caffeine has been used to relieve fatigue. But it is dangerous and one may collapse if not consumes it under certain limit.
3. Caffeine is also used in analgesic tablets, as it is believed to be a pain reliever. It is also beneficial in migraines.

Effects of Caffeine

1. It is psycho-stimulant.
2. It improves physical and mental ability.
3. Its effect in learning is doubtful but intellectual performance may improve where it has been used to reduce fatigue or boredom.
4. When administered continually, it stimulates heart and nervous system and also acts as diuretic on the contrary their excessive use is harmful to digestion and their long use lead to mental retardation

RESULT.

Here Solvent used was Dichloromethane and base used was sodium Carbonate for extracting caffeine from above types of tea and coffee. S. Parvathy ,Adlet Luiz and Jaya T. Vakrey in 2014 carried out the same analysis and they extracted more amount of caffeine from black tea as compared to our experiment because they used acidified water during solid-liquid extraction. Amber Nawab, Quratulam Waseem, Javeria Asif, Fatima Ahmed in 2016 extracted caffeine from black tea and they got 3% caffeine in the sample. Muthanna J.Mohammed,Firas A. Al-Bayati in 2008 also extracted caffeine from tea and they also got 3% caffeine as the result. The variations in the caffeine content of the coffee and tea is because of using different techniques during extraction of caffeine. Now for further experimentation coffee was used with different solvents and bases, as the caffeine content of coffee is much higher as compared to other types of tea and coffee.

CONCLUSION

Tea is very rich in antioxidants. It is the most widely used beverage all over the world. It also has medicinal properties. In this study teas will be decaffeinated using dichloromethane as a solvent. This study will be carried out to check the amount of caffeine in used tea leaves. It is acceptable that the amount of caffeine decreased with every use. Caffeine from tea is extracted by liquid-liquid extraction followed by recrystallization. Caffeine is the most commonly used psychoactive drug in the world. It is a pharmacological 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 Iodometric back titration method. The serious concern about 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 tea and 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%

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