

# **“EXTRACTION OF CAFFEINE FROM TEA LEAVES”**

## **Inhouse Project Report**

Submitted for the partial fulfillment of the degree of

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Submitted by-

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**UNDER THE SUPERVISION AND GUIDANCE OF**

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## ABSTRACT

Coffee, tea, cola, guarana, mate, and other goods all contain the chemical caffeine. Among the stimulants that athletes take the most frequently is caffeine. In other natural plant materials, caffeine is present in a small amount. The main ingredient in tea is cellulose, which is not soluble in water. When tea leaves are boiled, some tannins and gallic acid are produced; however, caffeine dissolves in water. The latter two components can be converted into salts that are insoluble in water. Afterward, virtually pure caffeine may be extracted from the water using chloroform. Trimethylxanthine, a class of compounds that includes caffeine, is a naturally occurring stimulant found in the fruits, seeds, and leaves of a wide variety of plant species. Its chemical formula is  $C_8H_{10}N_4O_2$ . One of the most common stimulants used by athletes is caffeine. The National Collegiate Athletic Association (NCAA) permits the use of caffeine within reasonable bounds.

Tea (black) leaves were used to extract and characterize caffeine. Using chloroform as a solvent, isolation was accomplished via liquid-liquid extraction. The process of this extraction was as follows: 1. Boiling 2. Filtration 3. Liquid-liquid extraction 4. Recrystallization. Sodium Carbonate was used for the recrystallization process. Using high-performance liquid chromatography, the purity check was carried out. Caffeine was effectively characterized by measuring its infrared spectrum, using a differential scanning calorimeter and a melting point device. This study aims to establish the caffeine content of well-known tea varieties.

**Keywords:** Tea Leaves, Chloroform, Crystallization, Caffeine.



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## ACRONYMS

%	Percentage
mg	Miligram
ml	Mililiter
°C	Degree Celcius
oz	Ounce
gm	Gram
Approx.	Approximately
UV	Ultra Violet
nm	Nano meter
DES	Deep Eutectic Solvent
ILs	Ionic Liquids
HBD	Hydrogen Bond Doner
HBA	Hydrogent Bond Acceptor
K	Kelvin
-OH	Hydroxide group
Fig	Figure
SFE	Supercritical Fluid Extraction
MAE	Microwave Assisted extraction

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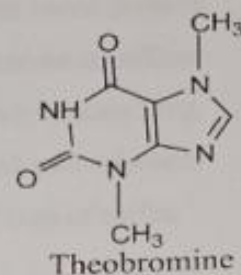
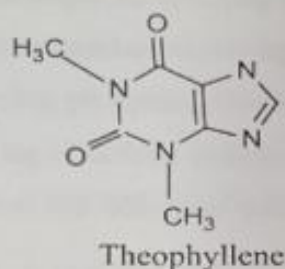
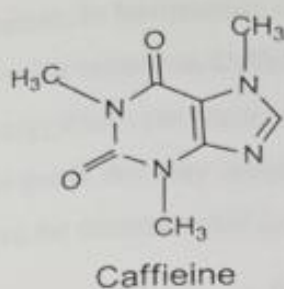
## CHAPTER 1: INTRODUCTION

Caffeine is a naturally occurring chemical stimulant that is present in the leaves, seeds, and fruits of many different plants. Although caffeine is the most often used psychoactive substance in the world, it is legal and unrestricted in almost every country.

It reduces fatigue and drowsiness by stimulating the central nervous system. It can improve one's physical strength, power, and athletic ability. The chemical compound known as caffeine, also known as 1, 3, 7 - trimethyl - 1, 7 - dihydro - 1H - purine - 2, 6 - dione or 1, 3, 7 - trimethylxanthine, with the formula  $C_8H_{10}N_4O_2$ , was discovered in 1827 and is a member of the alkaloid family, which includes compounds with heterocyclic rings that contain nitrogen.

China was the only country that produced coffee until the British Empire colonized India and established massive plantations there. Coffee consumption is a significant ritual in Japan and England, as seen by the daily coffee times held in homes and offices throughout Brazil and the choreographed Japanese coffee ceremony. At the moment, Brazil is the nation that drinks the most coffee globally. Caffeine is present in the leaves, seeds, and fruits of more than 63 distinct plant species worldwide. Coffee, cocoa beans, cola nuts, coffee leaves, yerba mate, guarana berries, and yaupon holly are the most popular sources of caffeine.

Although the amount of caffeine in coffee varies by brand, popular brands usually contain between 30 and 40 mg of caffeine per coffee bag on average. Since caffeine is not directly generated from amino acids, it is a purine-based pseudo alkaloids. In terms of pharmacology, caffeine stimulates the central nervous system, is a mild diuretic, and functions as a natural insecticide by raising heart rate, blood pressure, and gastrointestinal motility as well as acting as an algicide and bactericide. Initially extracted from coffee in 1819, caffeine was discovered by German scientist Friedlieb Ferdinand Runge. Its pure state is a white, odorless solid with a melting point between 235 and 238 degrees Celsius. Since caffeine's rings include two or more nitrogen atoms, it is categorized as a heterocyclic purine base. Due to its poor basicity, caffeine dissolves very little in water at ambient temperature (2g/100ml), but its solubility increases significantly in boiling water (66g/100ml).



**Fig-1 Chemical structure of methylxanthines [1]**

Caffeine, a stimulant, is the primary methylxanthine in tea. Two chemically related substances, theobromine and theophylline, which are also present in tea, are additional methylxanthines. The long-term attractiveness of non-alcoholic foods and drinks, such as coffee, tea, cocoa, chocolate, and a variety of soft drinks, is largely dependent on these chemicals.

For the treatment of migraine headaches, it is mixed with acetaminophen or aspirin as well as a substance known as ergotamine. Although caffeine has numerous other uses, its most common usage is to increase mental alertness. The producers of cosmetics and diet pills are the largest consumers of caffeine. It can also be used to make face scrub, body wash, soap, lip balm, and other products like lipstick with caffeine. Caffeine is present in many pharmaceutical preparations together with other ingredients such as acetylsalicylic acid, ascorbic acid, codeine, paracetamol, and other analgesics and antipyretics. The type of product, portion size, and method of preparation all influence how much.[14]

caffeine is present in food and beverage goods. The leaves of the Asian evergreen *Camelia Sinesis* are used to make tea, which is what most people consume. Tea leaves vary in their caffeine level depending on the cultivar and the growing region; most tea has 3–5% of its weight in caffeine. Different solvents (dichloromethane, water, chloroform, and ethyl acetate) were used to measure the optical transition features of caffeine.

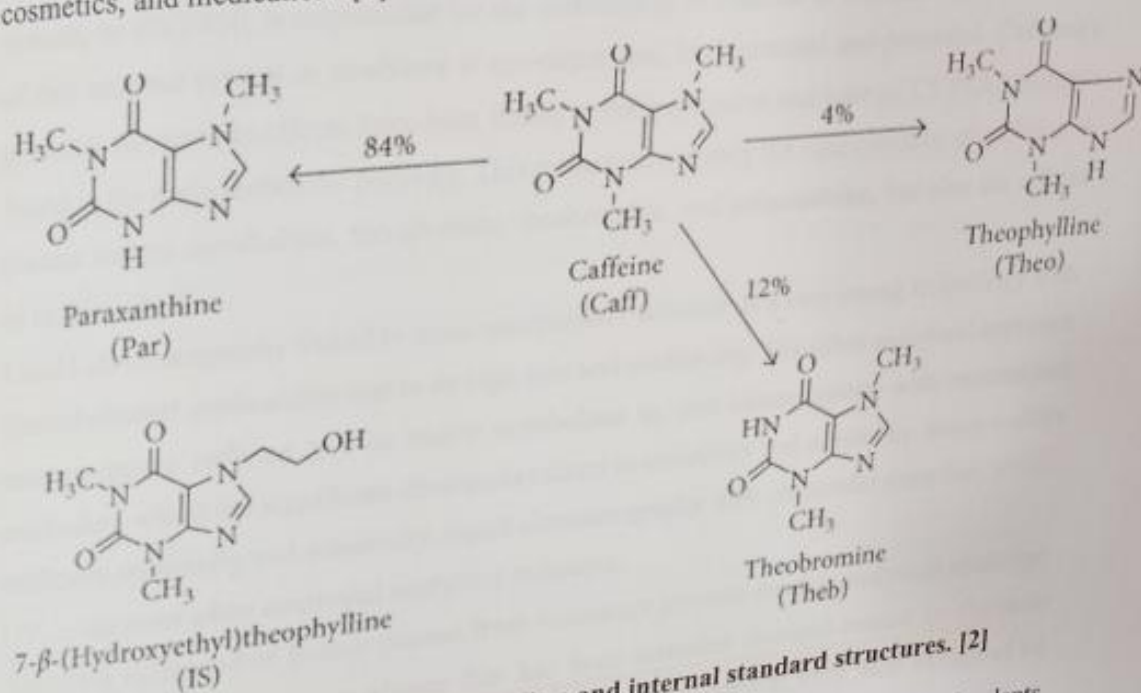
Comparing caffeine to the other solvents, dichloromethane exhibits the largest optical transitions. Compared to 30° C, more caffeine may be extracted at the boiling point. There are several ways to extract caffeine from cocoa, including liquid extraction, organic solvent extraction, and supercritical carbon dioxide extraction. Caffeine is typically extracted using solvents including ethanol, acetone, methyl chloride, chloroform, and ethyl acetate. For this extraction, a number of techniques are available, including heat reflux, ultrasonic, and Soxhlet



extraction. In laboratories, one popular technique for removing caffeine from cocoa seeds is heat reflux extraction. Caffeine content varies based on what is ingested. The amount of caffeine in energy drinks can reach 160 mg per serving, yet a piece of chocolate may only contain 5 mg. Other goods that may contain up to 200 mg of caffeine include diet pills and pain relievers. exceed the recommended daily allowance of 500–600 mg of caffeine, or 4–7 cups of coffee;

The majority of adults are thought to be safe with 200–300 mg of caffeine per day, or two to four cups of brewed coffee, according to Michigan State University Extension. health issues, such as headaches, increased heartbeat, anxiety, nausea or gastrointestinal issues, insomnia, etc.

The most popular psychoactive drug, caffeine, can have minor stimulant effects on the central nervous system. People swallow a variety of items that contain caffeine. It has physical addiction. The caffeine that is extracted can then be used to make everyday products like drinks, cosmetics, and medications.[1]



**Fig-2 Caffeine and its major metabolites and internal standard structures. [2]**

Caffeine (1,3,7-trimethylpurine-2,6-dione) is one of the most often used legal psychostimulants in the world today and is present in a large range of foods and beverages. This drug has a psychoactive ingredient. Its nonselective antagonist action targets adenosine receptors, making it a stimulating and moderately dissociative drug. Given its safer clinical



profile in comparison to theophylline, an older drug, and aminophylline, an oral version of the drug, caffeine is recognised as the first-line treatment for preterm apnea. For this indication, a standard caffeine dosage of 10 mg/kg for the load and 2.5 mg/kg for the maintenance has been defined globally. Effective doses range from 5 to 20  $\mu\text{g/mL}$ . Occasionally, increasing plasma caffeine levels to 30 or 35  $\mu\text{g/mL}$  or more is necessary to provide a therapeutic effect.

According to some, the great therapeutic value of coffee eliminates the need to check concentrations, margin that it has; nonetheless, some recommend that blood concentrations be checked on a regular basis because the postnatal period is a time of fast changes in caffeine clearance and half-life, which makes a procedure like the one suggested here particularly helpful in certain situations.

The species, strain, age, tissue, and sex differences, as well as the impact of inducers, nutritional state, and human drug metabolism, could all be explained by the existence of different cytochrome P-450 isoforms (CYP450). The liver microsomal drug-metabolizing enzyme system, or CYP450, is responsible for the metabolism of caffeine in humans. The expression of this enzyme system in newborns is age-dependent, both prenatal and postnatal. Caffeine's potential therapeutic effects have been linked to the expression and type of CYP1A2 isoform found in the main metabolic pathway. This influences not only the concentration of caffeine in plasma and its metabolites, theophylline, theobromine, and paraxanthine, but also the effects of caffeine itself.

Liquid chromatography linked to mass spectrometry detector is a very strong technology with limited clinical applicability due to its high cost and availability. The other analytical approach used to assess caffeine and its major metabolites is, and immunoassay with monoclonal antibodies, which has significant drawbacks related to selectivity and sensitivity. Since it offers sufficient sensitivity and selectivity, liquid chromatography with ultraviolet detection, or LC-UV, is the most often employed analytical technique.

Acquiring caffeine-free human plasma from volunteers presents another analytical challenge. Alternatives, such as human plasma that has been activated charcoal-treated to eliminate caffeine or synthetic plasma that has been created without caffeine, have been attempted by certain writers. Since cow whole serum is more representative than synthetic or human plasma, or serum that has been processed with activated charcoal to remove certain chemicals, we assess the viability of employing it. Comparatively speaking, we believe that serum from cows is easier to get and naturally caffeine-free than serum from human donors.[2]

## 1.1 Sources Of Caffeine

Caffeine can be found in nature in guarana, cacao, and coffee plants' fruits, leaves, and beans. Drinks and dietary supplements frequently include it. Overindulging in carbonated drinks carries a risk because they are easily absorbed quickly when eaten in large quantities, like soda and energy drinks.[13]

**Coffee:** A cup of brewed coffee, or eight ounces, contains around 95 milligrammes of caffeine. An equivalent quantity of instant coffee has about 60 mg of caffeine. Decaffeinated coffee has approximately 4 mg of caffeine.

**Espresso.** One shot, or 1.5 ounces, contains about 65 milligrammes of caffeine.

**Tea** A single cup of black tea contains about 47 milligrammes of caffeine. About 28 milligrammes are found in green tea. Decaf tea comprises two milligrammes of caffeine, while herbal tea contains none.

**Soda.** About 40 milligrammes of caffeine can be found in a 12-ounce can of regular or diet dark cola. Amounts of Mountain Dew containing the same amount of caffeine are 55 mg.

**Chocolate (cacao).** One ounce of dark chocolate has about twenty-four milligrammes of caffeine; an ounce of milk chocolate contains only a fifth of that amount.

**Guarana.** This seed is extracted and utilised in food, beverages, and supplements. It comes from a plant in South America. Coffee beans have approximately four times the amount of caffeine found in guarana seeds. The extracts from these seeds can contain up to 125 mg of caffeine per serving in drinks.

**Energy drinks.** An energy drink that contains eight ounces, or one cup, has about 85 mg of caffeine. However, the standard serving size for energy drinks is 16 ounces, which doubles the amount of caffeine to 170 milligrammes. Energy shots are significantly more concentrated than liquids; 200 mg of caffeine is found in only a 2-ounce shot.

**Supplements.** The amount of caffeine in two cups of brewed coffee, or roughly 200 mg, is found in each pill of a supplement.[3]

**Amount of Caffeine in common foods and beverages**

**Table-1 Amount of caffeine found in reputed coffee items**

Coffee	Amount (oz)	Caffeine (mg)
Coffee, generic brewed	8	95-200
Coffee, Starbucks brewed	16	330
Coffee, Dunkin Donuts brewed	16	211
Coffee, latte, misto	16	150
Espresso, Starbucks	1	75
Espresso, generic	1	64
Coffee generic instant	1 tbp granules	31
Coffee generic decaffeinated	8	2

**Table-2 Amount of caffeine found in reputed tea items**

Tea	Amount (oz)	Caffeine (mg)
Black tea, brewed	8	47
Green tea, brewed	8	25
Black tea , decaffeinated	8	2
Starbucks Tazo Chai tea latte	16	95



**Table-3 Amount of caffeine found in reputed soft drink**

Soft Drinks	Amount (oz)	Caffeine (mg)
Coke	12	35
Diet Coke	12	47
Pepsi	12	38
Diet Pepsi	12	36
Jolt Cola	12	72
Mountain Dew	12	52
7-Up	12	0
Sierra Mist	12	0
Sprite	12	0

**Table-4 Amount of caffeine found in reputed energy drinks**

Energy Drinks	Amount (oz)	Caffeine (mg)
Red Bull	8.3	77
SoBe Essential Energy, berry or orange	8	48
5-hour Energy	2	138

**Table-5 Amount of caffeine found in dessert items**

Desserts	Amount (oz)	Caffeine (mg)
Dark chocolate (70-85% cacao solids)	1	23
Milk chocolate	1.55	9
Coffee ice cream or frozen yogurt	8	2
Hot cocoa	8	8-12
Chocolate chips, semisweet	4	53
chocolate milk	8	5-8



## 1.2 Benefits Of Using Caffeine

- **Loss of weight**

1. Decreasing the need to eat momentarily and curbing the appetite.
2. Promoting thermogenesis to increase the amount of heat and energy the body produces during food digestion.

- **Brain activity**

Adenosine receptors in the brain are impacted by caffeine. Antioxidants called polyphenols are also present in coffee, and they work through a variety of mechanisms.

- **Colon and liver**

1. By promoting bile excretion through the colon wall, caffeine enemas have been proposed to assist prepare the colon for an endoscopy or colonoscopy.
2. Advocates assert that a caffeine enema supports the liver's natural detoxification processes by raising glutathione levels, an antioxidant.

- **Stroke**

Data from 34,670 women in Sweden without a history of cardiovascular disease showed that the risk of stroke was 22–25 % lower for those who drank more than one cup of coffee per day than for those who drank less.[11]

## 1.3 Risks

- **Depression**

Overindulgence in coffee consumption might worsen symptoms of anxiety and depression.

- **Blood sugar levels**

People with type 2 diabetes report greater blood glucose levels when they consume caffeine. Research suggests that coffee may counteract the actions of insulin, which could result in a tiny but noticeable increase in blood sugar, particularly after meals.

- **Lack of continence**

In a study including 1,356 women, the risk of bladder issues was shown to be 70% higher in those who used 329 mg of caffeine daily, or three cups of coffee or more.[12]

## 1.4 Green Solvents

Green solvents, sometimes referred to as biosolvents, are environmentally acceptable solvents made from the processing of agricultural crops. Petrochemical solvents are used in the majority of chemical reactions, which has detrimental effects on the environment. Green solvents were developed as a more environmentally friendly alternative to petrochemical solvents. Ethyl lactate is one green solvent that is produced during the digestion of maize. Ethyl lactate is the name of the lactic acid ester. Because lactate ester solvents are 100% biodegradable, easily recyclable, noncorrosive, noncarcinogenic, and non-ozone depleting, the paint and coatings industry employs them extensively. Ethyl lactate is a particularly attractive solvent for the coatings industry due to its high boiling point, low vapour pressure, low surface tension, and strong solvency power. Not only is it an excellent paint remover and graffiti remover, but it's also a desirable coating for polystyrene, metal, and wood. Ethyl lactate has replaced solvents such as xylene, acetone, and toluene, greatly improving working safety. There are more applications for ethyl lactate, including cleaning the polyurethane industry. Many polyurethane resins can be dissolved by ethyl lactate because of its potent solvent strength. Grease can be successfully cleaned from a variety of metal surfaces thanks to ethyl lactate's superior cleaning properties.[4]

### 1.4.1 Deep Eutectic Solvent

Newly developed green electrolyte compounds are called deep eutectic solvents (DESs). Abbott and associates presented the initial description of DESs in 2003. Compounds with a melting point substantially lower than the initial components are combined to form a DES. Combining an appropriate hydrogen bond donor (HBD) and acceptor (HBA) results in the creation of DESs. Not all combinations of HBAs and HBDs will result in a DESs because the definition of DESs calls for a melting point that is somewhat near to ambient temperature.

The limitations of ionic liquids (ILs) have been thought to be mitigated by deep eutectic solvents (DESs), which are composed of donors and acceptors of hydrogen bonds at a particular molar ratio. The DES has more advantages in terms of safety, biodegradability, and cost when compared to ILs. Similar to ILs, DESs can also be programmed solvents, meaning that component ratio adjustments can control their physicochemical characteristics. DESs have been applied to the pretreatment, delignification, and extraction of biomass. Furthermore, a lot of research has been done on cellulose dissolving in DESs. But the solubility lagged well



behind that of ILs. The ability of cellulose and DESs to interact was lowered because of the strong hydrogen bonding between the acceptor and donor. The mass transfer resistance in the dissolving systems, which will be the bottleneck for industrial applications, is also caused by its viscosity, which is similar to that of ILs.

The low melting point of DESs is caused by low lattice energy, high intermolecular hydrogen bonding interaction, and entropy change. DESs fall into four general categories: type I is metal chloride combined with quaternary ammonium salts; type II is metal chloride hydrate combined with a hydrogen bond donor; type III is metal chloride hydrate combined with a hydrogen bond donor; and type IV is metal chloride hydrate combined with a hydrogen bond donor.

The synthesis of DESs is more easier than that of ILs; all that is required is the simple mixing of HBD and HBA, without the requirement for a solvent, a purification procedure, or the creation of byproducts (Anastas & Eghbali, 2010).[5]

One of the ingredients in coffee pulp that has been found to be accountable for the bitterness and stimulating qualities of coffee brew is caffeine. Caffeine has become more popular since it can be a key component of many functional beverages, cosmetics, and pharmaceuticals. Research has been done to see if it is possible to extract caffeine from CP in an economical and straightforward manner.

The field of chemistry has recently embraced the concepts of green chemistry, which encourage the creation of new solvent systems, the use of non-hazardous media, and the use of ecologically friendly methods. In this specific context, deep eutectic solvents (DES) have been developed as an alternative to harmful organic solvents. Deep eutectic solvents are mixtures of two or more compounds whose freezing points are below the melting points of any of their component parts. These are mixtures of hydrogen bond acceptors (HBAs) like choline chloride and hydrogen bond donors (HBDs) such organic acids, sugars, amides, and alcohols. They are an effective method of eliminating phytochemicals from a range of plants due to their great chemical/thermal stability, low toxicity, and recyclability. In order to extract caffeine and chlorogenic acid from green coffee beans, several DES have been studied recently. Betaine-sorbitol, choline chloride-sorbitol, and their mixture with urea are a few of them.

Additionally, studies on the extraction of leftover coffee grounds and coffee pulp using 1,6-hexanediol-choline chloride and lactic acid-choline chloride, respectively, have been conducted. Glycerol is a polar, nontoxic, biodegradable, recyclable, very inert, and stable solvent that is mostly obtained from renewable resources. It has been utilised in several processes in place of organic solvents. Published research studies have examined the effectiveness of eutectic solvents, such as glycerol and choline chloride, as well as the use of glycerol as a solvent; however, the combination of glycerol and choline chloride has not been studied for the extraction of coffee pulp.

Additionally, a number of studies have discussed the benefits of using mathematical models to improve our comprehension of how to regulate an extraction process's parameters and to offer helpful data for equipment scalability. Fick's law of diffusion, empirical models, film theory, and unsteady diffusion can all be used to explain the kinetics of an extraction process. Nevertheless, there is no data available for the kinetic modeling of the extraction of coffee pulp.

Thus, the objective of this research is to develop a food-grade DES solution-based non-toxic, eco-friendly, and effective "green" technique of extracting caffeine from coffee pulp. The suggested extraction procedure's effectiveness is compared to the outcomes of employing conventional solvents, and the operating parameters are modified. Additionally, a model is developed to describe the kinetic mechanisms of extraction, and an appropriate model is employed to specify the relationship between the model constants and the extraction variables.[6]

### 1.4.2 DES Synthesis

In a molecular ratio of 1:3, glycerol (HBD) and choline chloride (HBA) comprised the solvent. The two materials were mixed at 600 rpm and 70 °C for an hour, or until a transparent mixture was obtained in a bottle with a bottom. Vessel viscosity was measured at 25 °C using a Brookfield rotational viscometer (LVDV-II+, Brookfield Engineering Laboratories, Inc., Stoughton, Massachusetts). Every measurement involved the use of three duplicate samples.

ATR-FT-IR spectra were acquired using the Spectra Manager software (Jasco, Essex, UK), a high-throughput Single Reflection ATR that made use of a diamond crystal, and a 6700 IR spectrometer that was equipped with a DLaTGS detector. For every spectrum in the absorbance mode, 32 scans with a resolution of 4 cm<sup>-1</sup> were obtained, covering a range of 4000 to 400



cm<sup>-1</sup>. The spectrum was collected and captured against a background using a dry, clean cell. The relevant spectrum for each sample was produced by averaging six recorded spectra prior to doing additional pre-processing.[6]

### 1.4.3 Ethyl Lactate (Ethyl 2-Hydroxy-Propanoate).

Ethyl lactate, sometimes referred to as ethyl 2-hydroxy-propanoate, is an agrochemical that is entirely biodegradable, non-corrosive, non-carcinogenic, and does not deplete the ozone layer. It can be utilised as a financially advantageous alternative to traditional liquid solvents. Its low toxicity led to its declaration as GRAS (generally regarded as safe), and the US FDA approved it for use as an ingredient in food and medicine. These characteristics have increased awareness of ethyl lactate's use as a green solvent in the food industry. Many potential applications have been reported, such as the extraction of carotenoids from diverse plant matrices, the separation of edible oil components (squalene and tocopherol), and the extraction of  $\gamma$ -linolenic acid from spirulina (Golmakani et al., 2012).

The scientists have also observed that caffeine dissolves in ethyl lactate. The solubility at 303 K was reported to be 3.2% by mass, which is quite similar to the values reported for the solubility of caffeine in water. These solubility data, which demonstrate the possible use of ethyl lactate as a green solvent for caffeine extraction from natural sources, served as the impetus for the current work. To the best of our knowledge, this is the first time that green coffee bean and green tea leaf extraction using ethyl lactate has been shown.[7]

### 1.4.4 Ethyl lactate as a Green Solvent

It is crucial to investigate new, environmentally friendly, effective, and safe solvents for human consumption since there are worries about the safety of food and the effects that solvent extraction technologies may have on the environment. A substance known as a "green solvent" poses no threat to the environment or public health. Supercritical fluids, ionic liquids, and water are the most often investigated green solvents. The lactic acid ester ethyl lactate belongs to a recently developed class of green solvents. [8]

### 1.4.5 Ethyl lactate offers an eco-friendly

This is the first evaluation of the bio-renewable agrochemical solvent for this use, it was created lately through fermentation from feedstock generated from corn. The material, also known as ethyl 2-hydroxy-propanoate, was put to the test by chemical engineers to see if it could extract caffeine from green tea and coffee leaves. In an accelerated solvent extraction system (ASE 350) from US company Dionex, static extraction tests were performed at 100, 150, and 200 degrees Celsius. [9]

### 1.4.6 High recovery levels

When compared to other solvents, ethyl lactate showed significant caffeine recovery levels of up to 60%, suggesting that it provided a competitive option. According to analysis, it was a more effective method of extracting caffeine than ethanol or ethyl acetate. The solvents of choice for extracting caffeine from coffee beans historically included dichloromethane, benzene, chloroform, and trichloroethylene. Caffeine was extracted from green tea using acetone, methanol, ethanol, acetonitrile, and ethyl acetate. But, they also co-extract catechins throughout the process, which makes green tea less useful as a functional health beverage. [9]



## CHAPTER 2: LITERATURE SURVEY

Pourbafrani, M., et al. (2019). "Extraction of caffeine from tea leaves using various solvents"

This study investigates the extraction of caffeine from tea leaves using various solvents, including water and organic solvents like dichloromethane and ethyl acetate. It talks about the effects these solvents have on the environment and human health as well as how well they dissolve caffeine. The study comes to the conclusion that although organic solvents are effective, there are serious safety and environmental concerns due to their toxicity and volatility[21].

Marták, J., et al. (2018) "Ionic liquids as green solvents for caffeine extraction.

The study looks on using ionic liquids as more environmentally friendly ways to extract caffeine. Comparing these solvents to conventional organic solvents, they are more environmentally beneficial due to their low volatility, high efficiency, and potential for recycling. Ionic liquids may successfully extract caffeine from tea leaves, as the study shows, but more optimization is required to lower prices and improve their usefulness[22].

M. Balakrishnan et al. (2021) "Supercritical fluid extraction of caffeine: process optimisation and comparison with conventional methods" .

This study investigates the process of removing caffeine from tea leaves using supercritical CO<sub>2</sub>. The study highlights how supercritical fluid extraction (SFE), which leaves no solvent residue in the finished product, has great selectivity and efficiency. It talks about how SFE is environmentally friendly and how its operating parameters can be changed. The study contrasts SFE with conventional techniques, emphasising the former's lower initial equipment investment and the latter's improved results[23].

Zhang, Y., et al. (2020) "Efficient and Mechanism-Based Microwave-Assisted Caffeine Extraction from Tea Leaves," .

The goal of the study is to improve the process of extracting caffeine from tea leaves by employing microwave energy. It describes how the oscillation of water molecules in the leaves caused by microwaves results in internal pressure and heat that breaks down cell walls and effectively releases caffeine. The study emphasises the advantages of microwave-assisted

extraction (MAE) over conventional techniques, including shorter extraction periods, less solvent use, and increased energy efficiency[24].

**Park, J. et al. (2019) "A comparative study of extraction methods for caffeine from tea leaves".**

This comparative analysis assesses the effectiveness, potential effects on the environment, and usefulness of three different techniques for extracting caffeine: solvent extraction, supercritical fluid extraction (SFE), and microwave-assisted extraction (MAE). The study comes to the conclusion that MAE offers an excellent balance of efficiency, speed, and environmental sustainability, while SFE delivers the best purity and selectivity. Although it is still possible, traditional solvent extraction presents serious environmental problems[25].

**Gupta, S., et al. (2020) "Optimisation techniques in caffeine extraction processes".**

The authors talk about the latest developments in artificial intelligence and computational techniques for improving caffeine extraction procedures. The goal of the study is to reduce expenses and environmental effect while increasing yield and efficiency. It draws attention to how process optimisation strategies can enhance current procedures, making them more effective and long-lasting. The study highlights the significance of creating novel approaches to maximise extraction conditions and parameters[26].

**Kumar, A., et al. (2021) "Hybrid extraction techniques for caffeine from tea leaves,".**

In order to increase overall efficiency and sustainability in the extraction of caffeine from tea leaves, this study investigates the possibility of combining various extraction techniques, such as microwave-assisted extraction (MAE) and supercritical fluid extraction (SFE). The study's encouraging findings demonstrate how hybrid approaches might take advantage of each method's advantages to improve extraction results. According to the research, these combinations may provide a more effective and sustainable method of extracting caffeine.[27]



## CHAPTER 3: PROBLEM FORMULATION

Every year, India generates a sizable amount of discarded tea. The nation produced over 1,400 million kg of tea trash in 2022, which included fibers, stalks, sweepings, fluff, and other non-conforming tea materials. The processing of tea leaves and other industrial processes in tea plants are the main causes of this waste. In an attempt to reduce the waste from this tea and increase economic efficiency, efforts are being made to transform it into value-added items including medicinal materials, antioxidants, and preservatives[15].

Caffeine is a widely consumed stimulant found naturally in tea leaves. The extraction of caffeine is a crucial process for various industries, including food, beverages, and pharmaceuticals. Traditional methods of extraction often involve organic solvents, which, while effective, pose significant environmental and health risks. Emerging methods such as supercritical fluid extraction (SFE) and microwave-assisted extraction (MAE) offer more sustainable and efficient alternatives but come with their own set of challenges and limitations. The optimization and development of extraction methods are essential for enhancing efficiency, reducing costs, and minimizing environmental impact[16].

### Problem Statement

Caffeine extraction from tea leaves using both traditional and modern methods comes with a number of challenges regarding cost, environmental impact, and efficiency. While traditional solvent extraction methods are effective, they are also environmentally harmful. While newer techniques like SFE and MAE offer better environmental profiles, they also come with challenges like high initial costs and process optimization. To improve the overall sustainability and efficiency of caffeine extraction, comprehensive research is needed to optimise these methods, develop greener solvents, and investigate hybrid techniques[17].

### Objective

1. Evaluate Current Techniques: Determine the effectiveness, influence on the environment, and viability of classic solvent extraction, SFE, and MAE for the extraction of caffeine from tea leaves.
2. Optimise Extraction Processes: To improve yield and efficiency, optimise the parameters and circumstances of current extraction procedures using computational approaches and artificial intelligence.
3. Develop Green Solvents: Research the possibility of ionic liquids and other biodegradable

solvents as eco-friendly substitutes for solvents used in the extraction of coffee.

4. Investigate Hybrid Techniques: To capitalise on their advantages and produce superior extraction results, combine several extraction techniques (such as SFE and MAE).
5. Assess Economic Viability: Examine each extraction method's cost-effectiveness, taking into account the upfront costs, ongoing expenses, and long-term advantages[18].

### Significance

The study will offer insightful information about enhancing the methods used to extract caffeine, thereby promoting more environmentally friendly procedures in the food, beverage, and pharmaceutical sectors. This project intends to improve the effectiveness and environmental sustainability of caffeine extraction from tea leaves by refining processes, creating greener solvents, and investigating hybrid methodologies[19].



## CHAPTER 4: MATERIALS AND METHADODOLOGY

### 4.1 Apparatus And Material Required

1. Separating Funnel- used for the phase separation of organic and aqueous layer.



Fig 3. Separating Funnel

2. Beaker- used for the collection of stained liquid after boiling.

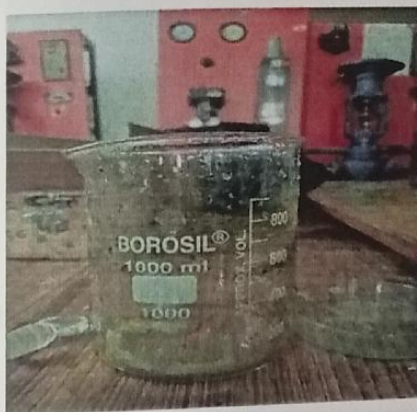


Fig 4 Beaker

3. Heating mantel- used for the purpose of heating the liquids.



Fig 5. Heating mantel



4. Funnel- prove liquids to flow through small opening.



Fig 6. Funnel

5. Filter paper- for separation of solids.



Fig 7. Filter Paper

6. Chloroform- used as a solvent to extract the caffeine.



Fig 8. Chloroform

7. Distill water- mixed with tea leaves for boiling purpose.



Fig 9. Distill water

8. Tea leaves- Raw material from which caffeine gets extracted.



Fig 10. Tea Leaves

9. Sodium carbonate- 7gms of Sodium Carbonate was used as a base.



Fig 11. Sodium Carbonate

10. Petri disk- used for the collection of organic liquid from separating funnel



Fig 12. Petri disc

## 4.2 Methadology

At starting we will take tea leaves as shown in Fig 13. and the we will take a flask and add 45gm of tea leaves and measure it, as shown in Fig 14.



Fig 13. Tea leaves



Fig 14. Measured Tea leaves

### 4.2.1 Boiling

Measure 300 ml of distilled water and put it into the pan along with sodium carbonate and measured tea leaves and boil it for 10 minutes as shown in Fig 15. The primary function of  $\text{Na}_2\text{CO}_3$  is to increase caffeine's solubility, which facilitates its extraction. All things considered when caffeine is extracted from tea leaves,  $\text{Na}_2\text{CO}_3$  is not meant to remove cellulose, collagen, or tannins.





Fig 15. Boiling of tea leaves

#### 4.2.2 Staining and Filtration

Stain the boiled liquid into a beaker put the solid material back into the pan and repeat the same process with distilled water by not adding the sodium carbonate then filter the liquid with the help of a funnel and filter paper as shown in Fig 16.



Fig 16. Filtration process

#### 4.2.3 Liquid-Liquid Extraction

Put the filtered liquid in a separating funnel, add 10 ml of chloroform in it, and shake it gently. Put it in a stand and wait for 10 min so that the liquids get separated, repeat this process 3-4 times and collect the liquid in Petri disc as shown in Fig 17.



Fig 17. Extraction of caffeine by adding chloroform

#### 4.2.4 Crystallization

The liquid is collected in a petri disc, put it on a heating mantle and let the liquid boil as shown in Fig 17. When all the liquid gets evaporated then some needle-like white compound will be shown in the petri disc as shown in Fig 18.



Fig18. Evaporation



Fig 19. Caffeine

## CHAPTER 5: RESULT AND DISCUSSION

In order to extract caffeine from the aforementioned tea varieties, sodium carbonate was utilised as the base and Chloroform as the solvent.

Here, chloroform ( $\text{CHCl}_3$ ) is used as a solvent. With the help of  $\text{NaCO}_3$ , chloroform is a substance that can dissolve caffeine. Chloroform ensures phase separation of the two liquids and assists in removing the aqueous phase from the organic phase with as little cross-contamination as possible due to its high density ( $1.47 \text{ g/cm}^3$ ) and capacity to force a sharper separation of the organic and aqueous phases. Caffeine is 94.53% soluble in chloroform during the extraction procedure.

Types of Beverages	Amount of Sample taken (gm)	Extracted raw crude caffeine (gm)
Black Tea	45	Approx. 1

45 gm of tea leaves can produce approximately 1 gm of caffeine.

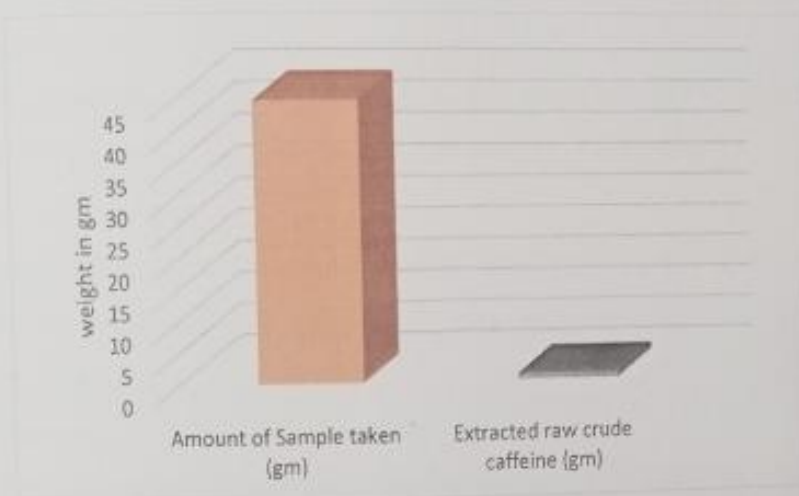


Fig 20. Sample taken to caffeine graph



## CHAPTER 6: CONCLUSION

Tea contains a lot of antioxidants. It is the beverage that is most frequently drank all across the world. It also has healing properties. The project will employ chloroform as a solvent to decaffeinate teas. The goal of this study is to determine the caffeine content of the tea leaves that are used. Caffeine is extracted from tea using a liquid-liquid extraction method, followed by recrystallization. Because of its greater solubility in chloroform, caffeine is extracted from a water-based tea leaf extract using this method, which uses chloroform as an organic solvent. However, the tannins that have low solubility in chloroform can be removed by transforming them into salts (phenolic anions through the addition of sodium carbonate) tannins are large acidic phenolic compounds that can be turned into salts by removing the -OH group which will stay in the water. Due to the existence of Caffeine, tea more popular as a stimulating substance with addictive properties.

## CHAPTER 7: ACHIEVED OUTCOMES AND SOCIAL RELEVANCE

Indian researchers have made great progress in turning tea waste into items with additional value. Among these results are:

1. Antioxidant Supplements: Green tea waste is used to make inexpensive, highly antioxidant supplements.
2. Fruit juices can now be kept fresh for up to a year thanks to the development of organic preservatives made from green tea residue.
3. Pharmaceutical Uses: Wasted tea leaves are turned into powdered catechin and pharmaceutical-grade super activated carbon for therapeutic purposes.
4. Environmental Impact: Promoting sustainable practices in the tea sector and reducing waste. [20]

### Characterization

Different physical methods were employed to characterize the crystalline caffeine.

#### 1. Melting point:

Using a digital melting point device, the melting points of many extracted pure samples were measured. 235 degree Celcius was the samples' average melting point.

#### 2. Thin Layer Chromatography:

Using chloroform as the mobile phase, a plate containing the refined product (crystalline caffeine) was created and seen under a UV lamp. After measurement, the Rf value was discovered to be 0.63.

#### 3. IR Spectroscopy:

An FT-IR spectroscopy was used to analyse the extracted, pure caffeine's IR spectra.

#### 4. UV Spectrometry:

The UV Absorption Spectrum of the Extracted Purified Crystalline Caffeine was prepared using a UV Absorption Spectrophotometer with various absorbances at various wavelengths. The maximum absorbance was 275 nm.[10]

## **Societal Relevance**

### **Impact on the Environment**

Through the reduction of waste produced in the tea business, caffeine can be extracted and repurposed from waste tea, thereby addressing major environmental concerns. The carbon footprint of conventional extraction procedures can be reduced and hazardous consequences can be minimised with the development of green solvents and efficient extraction processes.

### **Financial Gains**

Making use of tea trash to make value-added goods like medicinal materials and antioxidants can increase economic efficiency and open up new markets for tea growers. This supports sustainable agriculture methods while also enhancing the tea industry.

### **Innovation and Health**

The creative use of natural resources demonstrated by turning tea trash into health supplements and organic preservatives gives consumers environmentally and health-conscious products. This is in keeping with the rising demand from customers for environmentally and health-conscious products.



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## INTERNATIONAL JOURNAL OF RESEARCH AND ANALYTICAL REVIEWS (IJRAR) | IJRAR.ORG

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### Extraction of Caffeine from Tea Leaves

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#### Abstract:

A stimulating substance, caffeine is a white, crystalline, bitter xanthine alkaloid. It can be found in different amounts in the fruit, leaves, and seeds of several plants. It functions as a natural pesticide by paralyzing and killing some insects that feed on the plants and by improving pollinators' reward memory. Caffeine was extracted and studied from tea (black) leaves. Liquid-liquid extraction was used to achieve isolation using chloroform as the solvent. This extraction was done in the following manner: 1. Steaming 2. Screening 3. Extraction from liquid to liquid 4. Crystallization again. The recrystallization procedure made use of sodium carbonate. The objective of this study is to determine the caffeine levels of popular tea varieties. **Keywords:** Tea Leaves, Chloroform, Crystallization, Caffeine.

#### Introduction:

The psychoactive drug caffeine is a central nervous system stimulant that was discovered in 1819 by Friedrich Ferdinand Runge, a German chemist. Caffeine is a naturally occurring chemical stimulant that is included in many plant foods, including leaves, seeds, and fruits [1]. Despite being illegal and widely used in almost every country, caffeine is the most commonly used psychoactive substance in the world. It stimulates the central nervous system, which lessens weariness and drowsiness. It can enhance a person's physical prowess, strength, and agility [2].

In addition, it is used in conjunction with painkillers to alleviate and prevent headaches brought on by epidural procedures. Riches of caffeine 1, 3, 7-trimethylxanthine ( $C_8H_{10}N_4O_2$ ), originally discovered in 1827, is a member of the alkaloid family that has nitrogen in its heterocyclic ring structure. It is also referred to as 3, 7-dihydro, 1, 3, 7-trimethyl, 1H-purine, 2, 6 -dione, and 3,7 trimethylxanthine [3]. Up until the British Empire invaded India and set up extensive plantations there, China was the only nation that produced coffee. Brazil has daily coffee times in homes and companies, while the Japanese coffee ceremony is a choreographed ritual that demonstrates how important coffee consumption is in Japan and England. At the moment, Brazil leads the world in coffee consumption [4].

When dichloromethane is compared to the other solvents, caffeine shows the biggest optical transitions. At the boiling point, more caffeine may be extracted than at 30° C. Caffeine can be extracted from cocoa in a few different methods, such as liquid extraction, organic solvent extraction, and supercritical carbon dioxide extraction. Solvents such as ethanol, acetone, methyl chloride, chloroform, and ethyl acetate are commonly used to extract caffeine. There are several methods available for this extraction, such as Soxhlet, heat reflux, and ultrasonic. Heat reflux extraction is a common method used in labs to extract caffeine from cocoa seeds [5].

#### Materials Required:

Separating Funnel: used for separating aqueous and organic layer.

Beaker : for collecting filtered solution.

Heating mantle: for crystallization process.

Funnel and Filter paper: used for the purpose of filtering solid materials.

Choloroform : used as a solvent.

Distill water : to boil tea leaves.

Sodium carbonate: to increase caffeine solubility.

Petri disc: used to collect organic layer from the separating funnel.

### Experiment:

- 1) Measure 45 gm of tea leaves.
- 2) Measure 300 ml of distilled water and put it in the pan.
- 3) Add tea leaves and 7 gm of sodium carbonate
- 4) Boil the tea leaves for a few minutes.
- 5) Strain the tea leaves.
- 6) Repeat steps 2,4,5 for 3 to 5 times.
- 7) Collect strained liquid in a beaker
- 8) Filter the liquid with filter paper.
- 9) Put the filtered liquid in a separating funnel
- 10) Now add 10 ml of chloroform in a separating funnel and shake it gently.
- 11) Put it in stand and wait for 10 min so that the liquids layers get separated.
- 12) Collect the clear liquid in the Petri disc.
- 13) Repeat step 10,11 for 3-4 times.
- 14) Now the liquid which is collected in a petri disc, put it on heating mantle and let the liquid boil.
- 15) When all the liquids get evaporated then we will see a needle-like structure in the petri disc.

### Results And Discussion:

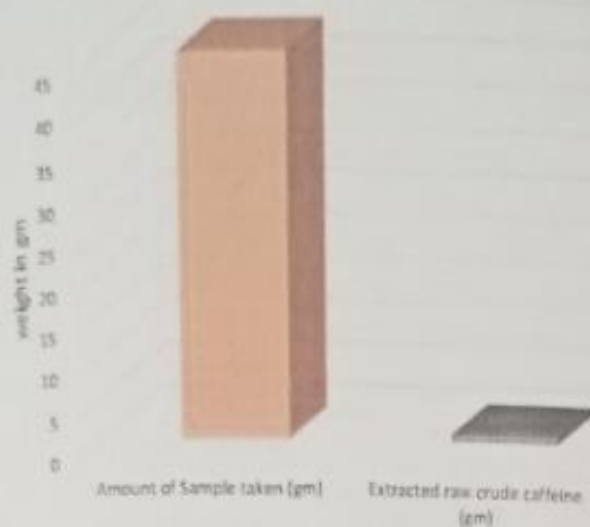





45 gm of tea leaves can produce approximately 1gm of caffeine.

Sodium carbonate served as the base and chloroform as the solvent in the caffeine extraction process for the aforementioned tea types.



The solvent employed here is chloroform ( $\text{CHCl}_3$ ). Caffeine can be dissolved with the use of chloroform and  $\text{CaCO}_3$  (lime). With a high density of  $1.47 \text{ g/cm}^3$  and the ability to compel a sharper separation of the organic and aqueous phases, chloroform assures phase separation of the two liquids and helps remove the aqueous phase from the organic phase with as little cross-contamination as possible. The extraction process of caffeine yields a 94.53% soluble in chloroform.

Types of Beverages	Amount of Sample taken (gm)	Extracted raw crude caffeine (gm)
Black Tea	45	Approx. 1

Figures And Table:

S.No.	Figures	Process
1		Measured 45 gm of tea leaves
2		Boiling
3		Filtration



4		Liquid-liquid separation
5		Evaporation

**Conclusion:**

Antioxidants are abundant in tea. It is the beverage that people drink most of all around the world. It also possesses medicinal properties. In this experiment, teas will be decaffeinated using chloroform as a solvent. This study aims to regulate the caffeine content of the used tea leaves. Caffeine from tea is extracted using liquid-liquid extraction, followed by recrystallization. Because caffeine is more soluble in chloroform than in water, it is used as an organic solvent in this procedure to extract caffeine from a water-based tea leaf extract. While tannins are big, acidic phenolic compounds that can be converted into salts by removing the -OH group that will remain in the water, tannins with low solubility in chloroform can be eliminated by converting them into salts (phenolic anions) with the addition of sodium carbonate. Because it contains caffeine, tea has become more well-known as an invigorating and addicting beverage.

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