

Review of Caffeine Extraction from Tea Leaves

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Abstract. Caffeine is a highly studied common alkaloid of tea and contributes a large portion of the alkaloid consumer market as a safe and widely accepted psychoactive substance. As a result, caffeine extraction becomes an important process with high demand in both the laboratory and industrial manufacture around the globe. This publication provides an overview of caffeine extraction methods from classical approaches to modern methods. Among these is the oldest and most established method: solvent extraction. Many designs with modern caffeine extraction are focused on the enhancement of profitability through the reduction of waste and pollution. This publication assesses the various techniques and methods of caffeine extraction along with the fundamental principles, mechanisms, and potential advantages/gains along with limitations and disadvantages of each. This publication also seeks to determine the extraction process most suitable for the simple, cost-effective, and large number of caffeine production facilities. This extraction process is explained in depth.

Keywords: Caffeine, Solvent Extraction, Supercritical Fluid Extraction, green technology

1. Introduction

Caffeine is an organic compound known as 1,3,7-trimethylxanthine and is consumed reliably by millions. Caffeine is naturally found in a variety of foods like tea and cocoa [1]. Because of the numerous foods containing caffeine that have been consumed for thousands of years, the stimulating disparity led scientists to extract and study these substances. In 1819, the German chemist, Friedlieb Ferdinand Runge, was the first scientist to isolate caffeine in its relative purity. From that point, the market implemented caffeine as an additive in a variety of products. To the surprise of many, these products that were legally stimulating and contained caffeine were highly coveted. Because of this, the market boomed from the immense sale of caffeine foods and beverages. As it began to sell, it became an enormous problem in the supply and demand of caffeine: efficiently producing it. The traditional methods for caffeine extraction had very low product yields and high raw material consumption. Many of these high costs represented enormous potential that was left unclaimed. Caffeine became the focus for a multitude of researchers. Chemically speaking, 1,3,7-trimethylxanthine is a highly stimulating purine that functions as a central nervous system alkaloid.

The primary chemical function of caffeine involves the blocking of adenosine receptors. Since adenosine causes the brain to feel fatigued, blocking receptors leads to a feeling of mental alertness.

This is why the brain feels more alert after coffee. In addition to the commonly known use of coffee as a beverage, caffeine also has other uses in medicine. It is used to treat headaches for example.

While caffeine is a natural chemical, the natural sources that contain caffeine do not contain caffeine by itself. In natural sources, caffeine is only a very small percentage of its entire structure. Actually, in most natural sources that contain caffeine, caffeine is a very small percentage of the entire structure and for most natural sources that contain caffeine, caffeine is a material that is present in a large quantity in a complex structure that contains other chemicals that are considered to be of lower value in most caffeine extraction processes. This means that additional chemicals that are of lower value are the major components in most caffeine extraction procedures. This is where most of the work in caffeine extraction is being done to an adequate caffeine extraction system.

With that being said, especially in a highly competitive market, the places where caffeine extraction is most heavily focused, be it in the academic community or industry. This is what the following paper aims to do. It aims to present all of the major caffeine extraction procedures and present all of the major decaffeination procedures in the order that they were developed. This is what paper is going to do. It's going to aim to present all the major decaffeination procedures in the order that they were developed.

2. Chemical properties of caffeine

2.1. Molecular structure and classification

Caffeine is classified as a xanthine alkaloid. It is formulated as $C_8H_{10}N_4O_2$ and is built on the purine framework. A purine has 3 methyl groups on its nitrogen atoms. Theobromine and theophylline lack the methyl groups.

2.2. Physical and chemical properties

The physical and chemical properties influence extraction of caffeine. Caffeine is a solid white crystal at room temperature and has a melting range of between $235^{\circ}C$ and $238^{\circ}C$. The crystallization of caffeine in the extraction is often done using ammonia and other organic solvents.

Caffeine's structure containing a nitrogen and oxygen affinity makes it a moderately polar organic compound. Caffeine is a weak base so it is not ionized in the water which makes water an unsuitable solvent for its destruction.

2.3. Relevance to the extraction process

Often caffeine is extracted from tea leaves in Laboratory settings. It relies on the unique acid base characteristics of caffeine and its degree of dual solubility during extraction. Furthermore, careful acidity and alkalinity balance are important for results reliability. Caffeine is a weak base and tea leaves contain tannins, which are acidic. Therefore, some base must be added to neutralize tannins and achieve a lower quantity of impurities in the organic phase that result from the extraction.

Regarding the other parameters, it is equally important to control the temperature and the time of extraction, as they influence the extraction efficiency and the solubility and the choice of the appropriate solvent facilitates the extraction.

3. Traditional extraction methods

3.1. General description of traditional methods

Traditional extraction methods are easily accessible because the equipment and environmental requirements for these methods are minimal. Traditional methods are highly employed due to the minimal requirements, as the equipment and cost in the student laboratories are minimal. Solvent extraction is the most common laboratory method and is combined with a crystallization step to enhance the purity of the extracted caffeine.

3.2. Liquid - liquid extraction method

To begin with, tea water solution is prepared and then an alkaline substance, such as sodium carbonate, is added to neutralize the acidic components of tea. Since caffeine is an alkaline substance, devoid of acid increases the likelihood of caffeine entering the organic phase which improves the yield and purity of caffeine [2].

In the following phase, ethyl acetate, an organic solvent, is introduced and the two phases are gently mixed. The organic phase is where the caffeine ends up, due to its affinity and the highest solubility of organic solvents. Thus, the first separation of caffeine occurs via phase separation.

3.3. Phase separation and repeated extraction

Once adequate time is allotted for separation, the distinct separation of the two phases occurs. The upper organic phase contains the caffeine due to its basic behavior. The organic phase is separately drawn up by a suction pipette. The purification and extraction can be repeated to achieve greater yield.

Partial emulsification may occur at the boundary of the two phases. Gentle stirring of the layers with a thin stick, followed by a settling period is necessary. The stick is withdrawn every few seconds, to allow the emulsification layer to gradually disappear.

3.4. Drying of organic extracts

Once the organic phase is separated, to increase the separation factor, the organic phase is dried. The protection from further patches of dew is a result of sodium, which upon dissolving absorbs water. A greater delay allowed is necessary, to allow the sodium to continue to withdraw from the reactant.

3.5. Evaporation and crude product formation

Once the residual desiccant is removed, the product is placed in the rotary evaporator and evaporated at reduced pressure and constant temperature. A dry precipitate can be observed as the caffeine solid around the collection flask. A rough calculation of caffeine yield can be conducted at this point, but the caffeine collected is actually crude caffeine and may be mixed with some pigments, some other unreacted organic substance and other impurities that would require further purification.

3.6. Recrystallization for purification

Recrystallization is also involved caffeine purification in this technique. Following this, the crude product obtained in step 4.5 is dissolved in isopropanol. As that dissolved product is allowed to attain room temperature, caffeine crystals can be observed as they precipitate from the solution. During this, vibration should be kept to a minimum to avoid disrupting the crystallization. For improved crystallization, a low crystal yield can also be supplemented with an ice bath.

3.7. Isolation and yield calculation

The purified caffeine crystals were subsequently isolated by the process of vacuum filtration. Following the removal of residual impurities, the washing agent was allowed to dry, and the data was then subjected to statistical treatment. The yield was determined by a mass difference that was recorded in the filtration process.

4. Modern extraction methods – supercritical fluid extraction (SFE)

4.1. Supercritical fluid extraction

Caffeine extraction technology is gaining interest with the extraction of caffeine from the coffee beans and the production of decaffeinated coffee. The extraction of caffeine from other products, like tea, is of great importance. In modern technology, there is a wider scope of innovative methods of extraction beyond the traditional methods of extraction [3, 4]. Among the many modern techniques, I would like to focus on the supercritical fluid extraction method. Once the advantages and disadvantages of this method and the different areas of application are explained, the method will be appreciated fully for its value and its potential.

4.2. Introduction of SFE

Supercritical Fluid Extraction (SFE) is a growing extraction method which has gradually replaced several other extraction methods, including traditional extraction methods for the past 10 years. For this purpose, it is used as a sustainable green technology [5, 6]. Even as a new method, there are difficulties like adjustments for each run which require raw materials and/or are different in terms of purities. However, proper experimentation and data processing and parameter tuning and using appropriate methods of maximization make it a viable method and an obtainable approach.

In the SFE method, the fluid is kept at a temperature and pressure above the critical point, allowing the supercritical substance to be in the state of a liquid and a gas, which in turn, increases its solubility [7].

4.3. Properties of supercritical CO₂

Supercritical carbon dioxide fluid is the most ubiquitous to researchers since its critical temperature and pressure aren't that high [8]. Among the supercritical fluids, collection of the gas has some advantages and is a desirable extraction solvent because of its high solubility, ease of diffusion, low viscosity, and low toxicity [9]. As there are no global warming potential and no environmental pollution, it is an eco-friendly material. In its supercritical state, carbon dioxide has the diffusivity of a gas and the density of a condensed liquid. It easily permeates solid substances [10].

4.4. Mechanism of caffeine extraction by using SFE

In the SFE process, supercritical carbon dioxide dissolves within the tea material, diffuses, and carries the caffeine solute with it out of the fluid. The SFE method provides the flexibility of adjusting pressure or temperature that enhances the caffeine solubility in carbon dioxide and controls the caffeine solubility in the carbon dioxide and the quantity of impurities that are dissolved. Caffeine polar solubility is further enhanced in carbon dioxide through the addition of ethanol, since the ethanol and carbon dioxide molecular polarity increases caffeine solubility in the supercritical fluid.

4.5. Advantages of SFE

In recent times, environmental capture has transformed and has undoubtedly become a significant area of concern for many. One of SFE's advantages over other extraction methods is that it is considered to be the most environmentally friendly of the other techniques. It carries out the whole extraction process without the use of organic, toxic solvents that can potentially harm the environment. A gas like carbon dioxide, which is abundant and considered to be safe, can be easily removed by simply reducing the pressure and increasing the temperature. In addition, there is no carbon dioxide left in the final product.

SFE places considerable pre-extraction work which results in a parameter related to pre-extraction work at its best. However, tailor-fitting extraction work to the physical controls of the system is relatively more rewarding as the power of adjustment at the system's disposal achieves work of considerable value, offers greater speed of extraction work, and greater capacity than SFE. These advantages account for the potentially large industrial caffeine output.

4.6. Limitations of SFE

SFE, with its relatively few drawbacks, is still a method that is not widely used. The equipment is costly and remains out-of-budget for many students and for many small and medium-scale laboratories. Furthermore, it is safe to say that regular maintenance has a high evolutionary cost given the complexity of the equipment, the high cost, and the necessity of the regular maintenance. Depending on work, prescriptive temperature and pressure changes may need to be made, but relatively few students need the determination and guidance.

4.7. Comparison with conventional methods

Supercritical fluid extraction (SFE) is much more environmentally friendly than conventional extraction methods. This SFE is achieved by avoiding the use of harmful organic solvents. However, students and laboratories that need to achieve extractions of lower volume and of lower grade would clearly find conventional extraction methods more convenient. Hence, SFE is more appropriate for larger scale laboratories (and industrial laboratories).

5. Conclusion

As a whole, the extraction of caffeine brings substantial research and commercial value in both the industrial sector and in the scientific community. Due to the fact that the extraction methods used in student laboratories are simple and economical, they have a wide range of applicability to help users master extraction methods, they are methods that are environmentally unfriendly, polluting and

wasting a large number of steps, which can hardly be regarded as efficient. On the contrary, SFE is the first method of extraction that is said to have been industrialized. It is in a class of extraction methods that is comparatively more efficient, more environment friendly and leaves less residue in the product obtained. Unfortunately, the first industrial methods of extraction have a very high price per unit operation and are very unfriendly as they do to small and medium sized laboratories and students of these larger laboratories.

In conclusion, almost all of extraction methods that have been developed, whether they are the more conventional methods of extraction that have been around for sometime or the more recent SFE commonly applied extraction technologies, have their merits and their applications differ. It is quite possible that the new methods of extraction would be more environmentally friendly, cheaper to operate and simple to apply while maintaining the yield and purity to industrial standards.

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