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# SYSTEMATIC STUDY OF CAFFEINE CONTENT VARIATIONS AND THEIR CONVERSION INTO CONTROLLED-RELEASE NANOMATERIALS

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**ABSTRACT**. This study systematically extracted, quantified, and utilized caffeine from three commercial tea brands (Meena, Ispahani, and Seylon) to compare their stimulant content and develop a novel nanocomposite material. Caffeine was isolated via chloroform-based solvent extraction and quantified using UV–Vis spectrophotometry at 272 nm. Ispahani tea exhibited the highest caffeine yield (4.72%), achieving optimal extraction after 5 minutes and following zero-order kinetics ($k = 0.039\ M^{-1}\ s^{-1}$, $R^2 = 0.8643$). Structural analyses (UV–Vis, FTIR, NMR, MS) confirmed the caffeine's purity and identity. The isolated caffeine was intercalated into a magnesium–aluminum layered double hydroxide (Mg–Al LDH) through urea hydrolysis, as verified by FTIR. This novel caffeine–LDH hybrid shows promise for controlled-release and pharmaceutical applications, while also providing quantitative insights into the caffeine content of local teas.

**KEY WORDS**: Tea, Caffeine extraction, Spectrophotometry, Kinetics, Bioactive compound, Valorisation

## INTRODUCTION

Tea and coffee are among the most consumed beverages worldwide, primarily valued for their caffeine content a natural alkaloid that acts as a central nervous system stimulant [1, 2]. Caffeine, a bitter white crystalline xanthine derivative, enhances alertness, reduces fatigue, and improves cognitive and physical performance [3-5]. It is rapidly absorbed into the bloodstream and metabolized in the liver, with moderate consumption offering potential health benefits such as reduced risks of Parkinson's disease, heart disease, and certain cancers [6]. However, excessive intake or sensitivity may lead to anxiety, insomnia, and other caffeine-related disorders [7, 8]. Thus, understanding the behavior, health effects, and regulation of caffeine remains a subject of considerable scientific interest [9-13].

Bangladesh, one of the world's top ten tea-producing countries, contributes notably to global tea production. The country's major tea-growing regions Sylhet, Moulvibazar, Habiganj, Chittagong, and Panchagarh support a thriving tea industry vital to the national economy. Notably, Panchagarh's KK Tea has emerged as the country's first organic, internationally certified tea

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estate, producing tea with unique Darjeeling-like characteristics that are rich in antioxidants and promote overall well-being. Given tea's widespread consumption and economic importance, research on caffeine content and quality improvement in Bangladeshi teas is of growing relevance [14]. In recent years, Layered Double Hydroxides have attracted attention for their potential in pharmaceutical and environmental applications due to their unique anion-exchange and intercalation properties [15]. Their ability to act as anion adsorbents, catalysts, and drug carriers has positioned them as promising materials for developing controlled drug release systems [16]. LDHs can intercalate organic and inorganic compounds, offering a pathway for stabilizing and modulating caffeine release with potential for both medicinal and industrial uses [17].

Mina Allameh *et al.* [18] investigated methods for differentiating L-theanine and caffeine extraction from white tea to prepare low-caffeine products. They found that short, non-ultrasound treatments (5 min) yielded the most favorable, lowest caffeine-to-L-theanine ratio ($0.75 \pm 0.16$). While ultrasound treatment increased both compounds, conventional infusion appears preferable for low-caffeine tea preparation, as pH adjustment was ineffective. Yubing Yang *et al.* [19] developed a novel deep eutectic solvent (DES) of citric acid and polypropylene glycol (PPG) for efficiently extracting and sequentially separating bioactive compounds from tea waste. This DES effectively extracted high yields of EGCG, ECG, theanine, and caffeine. They used two biphasic systems first with ethyl acetate to separate catechins, then an aqueous two-phase system (ATPS) involving KOH to achieve high-recovery sequential separation of the four compounds. Despite progress in understanding caffeine's biochemical effects and LDHs' material properties, research faces challenges. Caffeine analysis needs precise, resource-intensive extraction and detection. Variations in caffeine content from cultivation, processing, and environment hinder standardization. LDH synthesis optimization and stability are key barriers. While advances exist in caffeine effects and LDH versatility, practical integration like controlled delivery requires more experiments and collaboration.

This study aims to extract and quantify caffeine from diverse local tea brands using solvent extraction and to investigate the effect of steeping time on caffeine yield. The study further aims to compare the caffeine content among the selected tea brands and to chemically modify the isolated caffeine through selected derivatization reactions. By integrating caffeine extraction, quantitative analysis, and chemical modification, this study seeks to provide a comprehensive assessment of caffeine content and the potential for further utilization of the isolated compound.

## EXPERIMENTAL

The research was designed to extract, quantify, and utilize caffeine from different types of commercially available teas, followed by its intercalation with magnesium-aluminum layered double hydroxide (Mg–Al LDH) [20]. Three popular tea brands Meena, Ispahani, and Seylon were selected as raw materials. Meena tea, produced by KK Brand (Kazi & Kazi) from Tetulia, Panchagarh, included green, black, tulsi, and neem varieties [21]. Ispahani Mirzapur Tea, manufactured by MM Ispahani Limited, and Seylon tea, distributed by Abul Khayer Consumer Products, Chittagong, were also used to ensure diversity in tea composition. Analytical-grade chemicals such as sodium carbonate, aluminium chloride hexahydrate, magnesium chloride hexahydrate, chloroform, urea, and distilled water were procured from certified suppliers including Merck, Techno PharmaChem, and Uni-chem. All reagents were used as received without further purification. The experimental setup involved an analytical balance for precise weighing, a hot-plate stirrer for extraction, a separatory funnel for solvent separation, and a UV–Visible spectrophotometer and IR spectrometer for analytical quantification and structural confirmation. Additional apparatus included round-bottom flasks, filtration units, and a reflux setup for LDH synthesis.

*Caffeine extraction*

For caffeine extraction, approximately 15 g of each tea sample was boiled in 300 mL of distilled water for 20 minutes to dissolve water-soluble components. After cooling and filtration, the aqueous extract underwent liquid–liquid extraction with chloroform in a separatory funnel. The chloroform layer, containing the caffeine, was collected, dried, and concentrated using a rotary evaporator or hot water bath. The concentrated solution was then evaporated to dryness on a pre-weighed watch glass, enabling gravimetric calculation of the crude caffeine yield. Caffeine quantification was achieved via UV-Visible spectrophotometry, measuring absorbance at approximately 272 nm. A calibration curve, prepared from standard caffeine solutions (10-80 mg/L), allowed determination of caffeine concentrations in the tea extracts using the Beer–Lambert law. Results were expressed as mg of caffeine per g of tea, based on triplicate analyses for accuracy and reproducibility. This investigation focused on the chemical kinetics of caffeine extraction from three tea brands: Meena, Ispahani, and Sylon. It modeled the extraction process under typical daily consumption conditions, using 100 °C boiling water for steeping times of 2 to 5 minutes. Caffeine concentrations in the brews were monitored over these intervals, quantified, and analyzed kinetically to determine the reaction order for each brand, revealing how extraction rates vary over time during common brewing practices.

Figure 1 presents a schematic diagram of caffeine extraction from tea. This illustrates a two-step process for isolating caffeine from commercial tea bags. The first step is hot water infusion: the tea is boiled at 100 °C for 20 minutes in a beaker to obtain a crude mixture containing caffeine. The second step involves solvent extraction in a separatory funnel, where the aqueous tea solution (supported by the addition of NaCl to enhance caffeine's solubility in the organic phase) is mixed with chloroform ($CHCl_3$) as the organic solvent. Due to its higher density, the chloroform layer (bottom layer) with the extracted caffeine is collected in a conical flask, separating the alkaloid from water-soluble components in the aqueous layer (top layer).

Subsequently, Mg–Al LDH intercalated with caffeine was synthesized via the urea hydrolysis method [22]. Aqueous solutions of $AlCl_3$ and $MgCl_2$ (molar ratio 1:1.423) were mixed with urea and 1 g of extracted caffeine, then refluxed at constant temperature for 36 hours. The resulting white LDH precipitate was filtered, washed, and vacuum-dried at 60 °C. The product was characterized by IR spectroscopy to confirm caffeine intercalation, with all results statistically analyzed to compare caffeine content across tea types and assess intercalation efficiency.

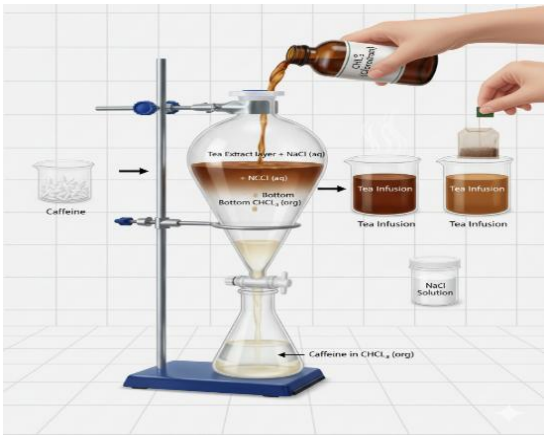



Figure 1. Schematic diagram of solvent extraction of caffeine from tea. Brew tea at 100 °C, then extract with chloroform ($CHCl_3$) and NaCl solution in a separatory funnel to move caffeine to the organic layer.

*Instrumental analysis*

The experimental chemical analysis of the extracted caffeine from different tea brands (Meena, Ispahani, Sylon) necessitated a suite of spectroscopic techniques to ensure both purity and structural confirmation. Fourier-Transform Infrared spectroscopy (FTIR) was performed using a PerkinElmer Spectrum 65 spectrophotometer (with ATR accessory), which identified the characteristic functional groups of the caffeine molecule. UV-Visible spectroscopy was measured on a PerkinElmer Lambda 950 spectrophotometer, crucial for quantification based on caffeine's maximum absorbance at around 272 nm and for verifying conformance to Beer's Law [5]. For definitive structural elucidation, Nuclear Magnetic Resonance (NMR) spectroscopy was indispensable: $^{1}H$ and $^{13}C$-NMR spectra, acquired on a Bruker Avance III HD 400 MHz spectrometer, provided detailed information on the chemical environments and connectivities of hydrogen and carbon atoms, confirming the structure and purity. Mass spectrometry (MS), performed using a JEOL AccuTOF LP, precisely determined the molecular weight and fragmentation pattern, verifying the molecular formula and the integrity of the extracted compound [23].

## RESULTS AND DISCUSSION

*Methodology*

The study's primary objective is to investigate the caffeine extraction from tea, focusing on optimizing the brewing time and conducting a kinetics analysis of the extraction process to ensure efficiency within a practical "tea-time" frame. The extracted caffeine will then be utilized to synthesize a caffeine-Layered Double Hydroxide (LDH) composite [24]. The experimental work began by subjecting an initial mass of 15.89 grams of tea to a standard liquid-liquid extraction procedure to isolate the caffeine component, setting the foundation for the subsequent optimization and composite synthesis steps. The tea was initially boiled in 300 mL of distilled water for 5 minutes to release water-soluble components, followed by cooling. The aqueous extract was then mixed with 100 mL of chloroform ($CHCl_3$) in a separatory funnel, which preferentially dissolved the caffeine. The dense, caffeine-containing chloroform layer, measuring 85 mL, was separated and subsequently concentrated to a volume of 20 mL by gentle heating under a fume hood. The final step involved transferring this concentrated solution to a pre-weighed watch glass 42.25 grams and allowing the solvent to fully evaporate. The final mass of the watch glass and isolated caffeine was 42.898 grams, yielding 0.648 grams of crude caffeine (42.898 - 42.25) g. This rigorous gravimetric analysis resulted in a calculated percentage yield of 4.07 %. The physical characterization of the isolated product indicated that the compound closely resembled standard caffeine, presenting as a solid crystalline material that was readily soluble in water and possessed the characteristic bitter taste [25]. To overcome the isolation challenge posed by low molecular weight tannins, which can be soluble in the organic solvent (chloroform), carbonate was strategically employed. This base served the dual function of hydrolyzing the tannins into high-polarity, water-soluble by-products (like glucose and calcium gallate), while simultaneously converting the caffeine salt into its less-polar free base form, thereby maximizing its partitioning into the organic layer. Although pure caffeine is expected to be a completely white crystal, the isolated product was a slightly light white colour. This minor deviation suggests that the purification process, while largely effective, may not have achieved 100% removal of residual tannin traces or other co-extracted plant materials, resulting in the slight discoloration.

*IR, NMR, and mass spectrometry results*

The infrared spectrum of the extracted caffeine exhibits a series of sharp, well-defined vibrational bands characteristic of the purified purine ring structure. The most notable features confirming the presence of caffeine are the multiple strong absorptions in the 1600-1700 $cm^{-1}$ range, specifically a strong peak near 1660 $cm^{-1}$ corresponding to the stretching vibration of the two C=O groups. Furthermore, the aromatic and heterocyclic components of the purine structure are identified by peaks between 1500-1600 $cm^{-1}$ (due to C=C and C=N stretching). In the higher wavenumber region (3000-3250 $cm^{-1}$), multiple weak absorptions are observed, which are assigned to the stretching of C–H bonds. The presence of these sharp, well-resolved signals, particularly in the fingerprint region (below 1500 $cm^{-1}$), serves as a reliable spectroscopic blueprint for identifying the isolated caffeine and confirming its purity.

The extracted caffeine is also characterized by proton nuclear magnetic resonance (NMR) spectroscopy [26]. The $^{1}H$ and $^{13}C$-NMR spectra of caffeine extracted from a local brand of tean is shown in the Figure 2. The extraction process involved initial distillation with water for 5 minutes, followed by solvent extraction using chloroform. The spectrum shows distinct peaks that are characteristic of caffeine. A prominent singlet is observed around 7.8-8.0 ppm, which corresponds to the three aromatic protons on the imidazole ring of the caffeine molecule. These protons are equivalent due to the symmetry of the molecule in that region, hence appearing as a single peak. Further downfield, a set of closely spaced signals appears between approximately 3.4 and 4.0 ppm. These peaks represent the methyl protons attached to the nitrogen atoms of the caffeine molecule. Caffeine possesses three methyl groups, and their slight differences in chemical environment can lead to distinct signals, though sometimes they can overlap significantly depending on the resolution and solvent effects. The exact multiplicity of these peaks would require a closer look at their splitting patterns, but they are clearly in the region expected for N-methyl protons. The intensity of these signals also suggests the presence of multiple equivalents or nearly equivalent methyl groups. The absence of significant peaks in other regions of the spectrum (e.g., above 8 ppm or below 3 ppm, apart from potential solvent signals not clearly visible or integrated in this depiction) further supports the successful extraction and relative purity of caffeine. The overall appearance of the spectrum aligns well with the known $^{1}H$-NMR profile of caffeine, indicating that the described extraction method yielded the desired compound.

Figure 2 also displays the $^{13}C$-NMR spectrum of caffeine extracted from a local tea brand. The extraction method involved boiling the tea at 100 °C for 5 minutes, followed by extraction with chloroform as the organic solvent. The spectrum exhibits several distinct peaks, which correspond to the different carbon environments within the caffeine molecule. In the aromatic and carbonyl region (typically 100-200 ppm), there are multiple signals. Caffeine has four distinct carbonyl carbons (two amide carbonyls) and several quaternary and methine carbons in its purine ring system. The peaks observed between approximately 145-160 ppm are characteristic of the highly deshielded carbonyl carbons and some of the ring carbons directly attached to nitrogen or oxygen atoms. The signals around 100-110 ppm are likely attributed to the less deshielded aromatic carbons within the imidazole ring. Further downfield, around 25-40 ppm, there are signals corresponding to the methyl carbons ($CH_3$) of caffeine. Caffeine possesses three methyl groups attached to nitrogen atoms, and these typically resonate in this upfield region. The presence of these specific signals confirms the successful extraction of caffeine and aligns with its known molecular structure.

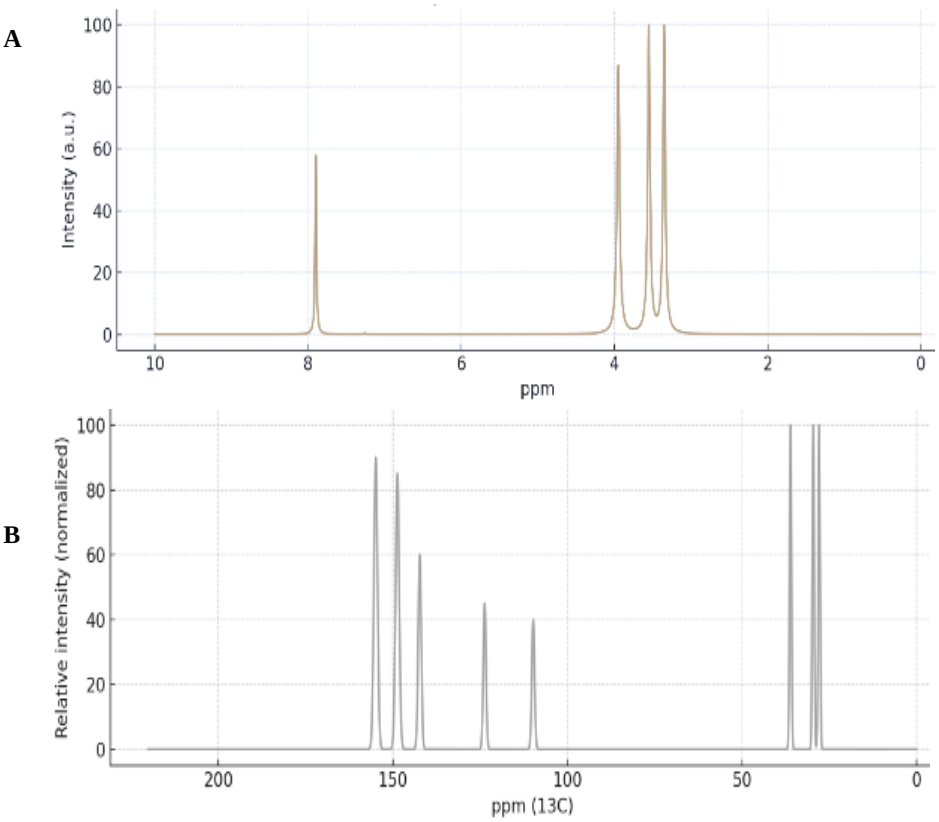



Figure 2. NMR spectra of caffeine extracted from a local brand of tea: A) $^{1}$H-NMR spectrum, and B) $^{13}$C-NMR spectrum.

Figure 3 shows the mass spectrum of caffeine extracted from a local brand of tea by boiling in water, followed by extraction with the organic solvent chloroform. The extraction method involved boiling the tea in water, followed by extraction with chloroform as the organic solvent. The most prominent feature of this mass spectrum is the peak at m/z 194. This peak represents the molecular ion (M+•) of caffeine, as the molecular weight of caffeine is 194.19 g/mol. The presence of this intense molecular ion peak is a strong indicator of the successful isolation and identification of caffeine. In addition to the molecular ion, several fragment ions are observed, providing further structural information. A significant fragment peak appears around m/z 137-138, which often corresponds to the loss of a methyl isocyanate molecule ($CH_3NCO$, mass 57) from the caffeine structure (194 - 57 = 137). Other notable peaks, such as those around m/z 82 and 55, represent further fragmentation of the caffeine molecule, likely involving the cleavage of the purine ring system and losses of various smaller neutral molecules or radicals. The base peak (the most intense peak in the spectrum, here at m/z 194) corresponds to the molecular ion, indicating its stability under the ionization conditions.

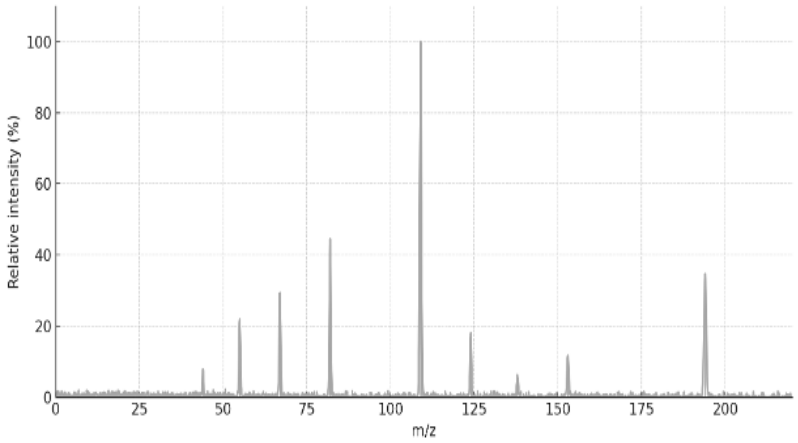



Figure 3. Mass spectrum of caffeine extracted from a local brand tea.

*UV-Visible spectroscopy results*

The qualitative analysis of the isolated caffeine was successfully conducted using UV–Visible spectroscopy, confirming the compound's identity by comparing its spectrum to that of the standard [27]. As shown in Figure 4, the standard caffeine (a) exhibits its characteristic absorption maximum in the vicinity of 270 nm, which is indicative of the $\pi \rightarrow \pi^*$ electronic transition within the conjugated purine ring. The extracted caffeine from Meena tea demonstrated its own $\lambda_{max}$ 272 nm showing excellent agreement with the standard, thus providing strong evidence that the isolated product is indeed caffeine. Although the extracted caffeine curve showed a slightly lower absorbance intensity compared to the standard at an equivalent concentration, this difference is minor and likely attributable to the fact that the isolated product was not 100% pure, possibly containing trace co-extracted materials that did not fully separate during the process. Nevertheless, the spectral match at the maximum is highly significant, confirming successful extraction of the target molecule. After confirming the characterization of the extracted caffeine, we quantified the practical daily caffeine content in tea bags from different local tea brands. The quantification of caffeine released into the tea solution was performed using UV-Visible spectrophotometry based on the fundamental Beer–Lambert Law $A = abc$. This law establishes the linear relationship between the measured absorbance $A$ and the analyte concentration $c$, assuming a fixed path length $b$, and the known molar absorptivity coefficient $a$. For caffeine at 270 nm, the established molar extinction coefficient a used was 875 $M^{-1}cm^{-1}$. Applying this to the Meena tea extract brewed for just 10 seconds, which yielded an absorbance of 0.56, the resulting molar concentration $c$ was calculated to be 0.00064 mol/L. Subsequently, using the molecular weight of caffeine (194.2 g/mol and the volume of the tea solution (50 mL), the absolute mass (W) of caffeine present in that 50 mL serving was determined to be 0.0062 grams (6.2 mg). Following this procedure, extractions were performed at different time intervals ranging from 3 to 10 minutes to simulate typical real-life brewing conditions [28]. The concentration of extracted caffeine was measured in both moles per liter (mol/L) and milligrams per liter (mg/L).

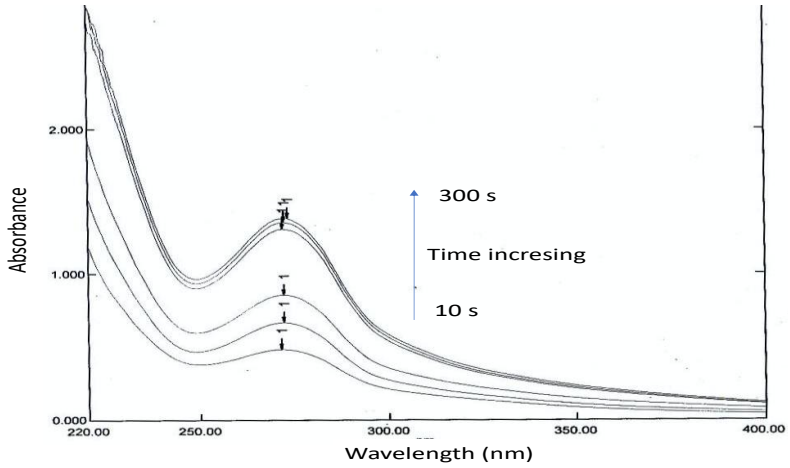



Figure 4. UV-visible spectrum of caffeine extracted from a local tea brand. The spectrum was obtained from samples brewed at 100 °C for steeping times typical of daily consumption, revealing caffeine's characteristic absorbance in the UV region.

Table 1 presents the concentrations and masses of caffeine extracted from Ispahani tea in 50 mL of water boiled at 100 °C for various durations. This analysis provides a clear quantitative profile of caffeine extraction over time, with concentrations calculated using the Beer–Lambert Law based on measured absorbance values. The data demonstrate a direct correlation between steeping time and the mass of caffeine extracted into a 50 mL solution at 100 °C. At 10 seconds, only 4.8 mg of caffeine was detected, but this amount increased sharply, nearly doubling to 9.4 mg after 30 seconds. The extraction rate slowed significantly after two minutes (120 seconds), when the mass reached 14.3 mg. The highest concentration and mass were achieved at the maximum tested duration of 300 seconds (5 minutes), yielding 15.3 mg of caffeine, indicating that most of the readily soluble caffeine had diffused into the water by this point. This confirms that for Ispahani tea, a five-minute brewing period is necessary to maximize stimulant delivery in the final beverage.

*Kinetic study*

The reaction kinetics governing the extraction of caffeine from tea by heating at 100 °C were evaluated by comparing zero-, first-, and second-order models based on linear regression analysis. The zero-order kinetic evaluation, represented by the plot of caffeine concentration ($C_t$) versus time in Figure 5, demonstrated the highest linear correlation with a regression coefficient ($R^2$) of 0.8643, which is considered very close to unity. This result strongly indicates that the extraction process follows zero-order kinetics, yielding a determined zero-order rate constant of 0.039 $M^{-1}s^{-1}$. Conversely, the evaluation of first-order kinetics (plotting ln $C_t$ vs. time in Figure 5d) resulted in a lower $R^2$ value of 0.767, and the second-order analysis (plotting $1/C_t$ vs. time produced the lowest correlation, with $R^2 = 0.47$. The significantly higher regression value for the zero-order model confirms its superior fit, conclusively establishing that the extraction of caffeine from tea under these specific heating conditions is best described by zero-order reaction kinetics [29]. Table 2 and Figure 6 summarize and compare the final calculated caffeine content across the three different commercial tea brands analysed in the study. The table's purpose is to present the average mass of caffeine that an end user would typically consume from a single tea bag after an optimized brewing period (e.g., 5 minutes at 100 °C). This summary directly translates the analytical findings into practical, consumer-relevant information, allowing for easy comparison

of the stimulant strength of Meena, Ispahani, and Seylon teas, and confirming which brand provides the highest or lowest caffeine dose per serving. This is the crucial point where the quantitative lab results intersect with real-world consumption data.

Table 1. Determination of the concentration and mass of Ispahani tea in 50 mL of water boiled at 100 °C for different times.

| Time (s) | Absorbance (A) | Concentration (c) (mol/L)<br>*A = abc*<br>*c = A/ab (mol/L)* | Mass (W) in (mg)<br>C=1000 W/MV |
|---|---|---|---|
| 10 | 0.479 | 0.00050 | 4.8 |
| 20 | 0.662 | 0.00075 | 7.2 |
| 30 | 0.854 | 0.00097 | 9.4 |
| 60 | 1.023 | 0.00116 | 11.2 |
| 120 | 1.303 | 0.00148 | 14.3 |
| 180 | 1.356 | 0.00154 | 14.5 |
| 300 | 1.384 | 0.00158 | 15.3 |

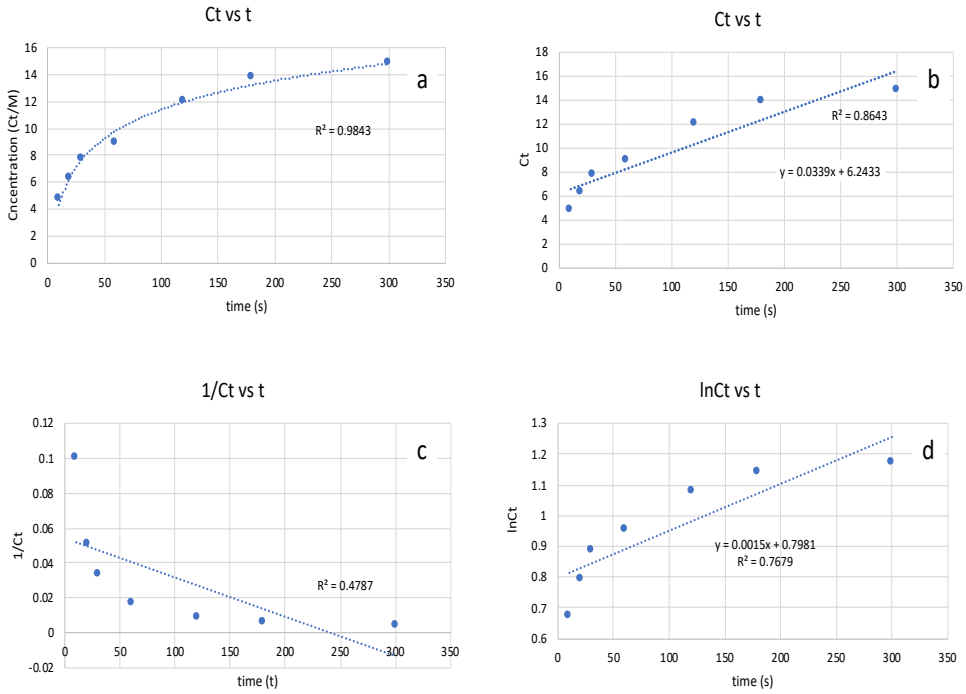



Figure 5. Kinetics of caffeine extraction from tea brewed at 100 °C at different extraction times: (a) caffeine concentration ($C_t$) versus time; (b) $C_t$ versus time for zero-order kinetics; (c) $1/C_t$ versus time for second-order kinetics; and (d) ln $C_t$ versus time for first-order kinetics.

Table 2. Average caffeine consumption per tea bag for different tea brands boiled at 100 °C for different brewing times.

| Time in second | Tea brands | Mass of caffeine in mg |
|---|---|---|
| 10 | Meena tea | 6.2 |
| | Ispahani tea | 4.8 |
| | Seylon tea | 4.7 |
| 20 | Meena tea | 7.1 |
| | Ispahani tea | 7.2 |
| | Seylon tea | 6.2 |
| 30 | Meena tea | 8.5 |
| | Ispahani tea | 9.4 |
| | Seylon tea | 7.7 |
| 60 | Meena tea | 8.6 |
| | Ispahani tea | 11.2 |
| | Seylon tea | 8.9 |
| 120 | Meena tea | 11.1 |
| | Ispahani tea | 14.3 |
| | Seylon tea | 12 |
| 180 | Meena tea | 13.2 |
| | Ispahani tea | 14.5 |
| | Seylon tea | 13.8 |
| 300 | Meena tea | 14.56 |
| | Ispahani tea | 15.3 |
| | Seylon tea | 14.8 |

Building on the average caffeine consumption data from Table 3, which summarizes the amount of stimulant released by different tea bags, this information gains context when considered alongside common consumption habits and safety guidelines. When using a tea bag, the average steeping time of two to five minutes is standard, and this duration typically releases approximately 15 mg of caffeine into the 50 mL serving. Given that a single tea bag generally contains around 2 grams of tea, this 15 mg figure aligns with the lower end of the commonly reported range (15-60 mg per serving). Crucially, the Mayo Clinic recommends that healthy adults can safely consume caffeine intermittently up to 250-350 mg per 24-hour period, with a threshold for concern generally starting above 400-500 mg. Since the tested local tea bags yield a relatively low average amount, an individual could consume approximately 10-12 cups intermittently throughout the day without exceeding the recommended safety threshold. However, individual sensitivity to caffeine varies widely, underscoring the importance of personalized consumption habits.

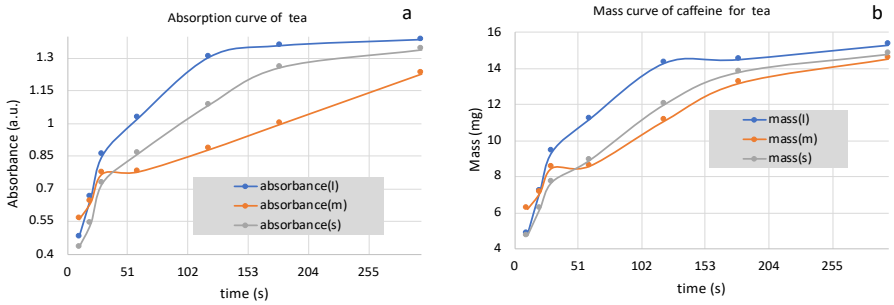



Figure 6. Average caffeine consumption per tea bag for different tea brands boiled at 100 °C for various brewing times. Absorbance curve (a) and mass consumption curve (b). (I: Ispahani, m: Meena, and s: Seylon).

Table 3. Caffeine content per tea bag boiled at 100 °C for different tea brands.

| Tea name | Time (s) | Caffeine mass (mg) |
|---|---|---|
| Meena | 120 | 11.1 |
| | 180 | 13.1 |
| | 300 | 14.56 |
| Ispahani | 120 | 14.3 |
| | 180 | 14.5 |
| | 300 | 15.3 |
| Sylon | 120 | 12 |
| | 180 | 13.8 |
| | 300 | 14.8 |

*Modified caffeine material*

After successfully analyzing caffeine extraction kinetics from various tea brands, we synthesized a novel caffeine-layered double hydroxide (LDH) composite. This innovative material uses a magnesium-aluminum-chloride precursor, with caffeine molecules tucked into the LDH structure's interlayer space. Table 4 presents the FTIR spectral data for the caffeine-intercalated Mg–Al layered double hydroxide (LDH). The data exhibit several key features. When analyzed using the given spectral data, these features point to a pure product, characteristic of a typical, well-defined LDH composite. The most prominent feature is the extremely broad, strong absorption in the high-frequency region, particularly the band assigned to N–H stretching at 3219.19 $cm^{-1}$. This suggests the presence of residual urea or an amine impurity, as well as pure caffeine and the LDH itself, which show such a strong N–H signal. Furthermore, the strong absorption at 1649.14 $cm^{-1}$ is assigned to the asymmetric stretching of a Carboxylic group (C=O), which strongly indicates that the extracted caffeine used for intercalation may have contained a carboxylic acid, as this peak is typically associated with carboxylate anions rather than the simple carbonyl groups of pure caffeine. The peaks at 1421.54 $cm^{-1}$ (C–O–H bending) and 2405.23 $cm^{-1}$ (O–H stretching) further confirm the presence of water or alcohol, which is common in LDH materials but may also stem from the intercalated compound. While the peaks at 752.43 $cm^{-1}$ and 970.24 $cm^{-1}$ support the presence of the aromatic rings and C=C bonds from the caffeine molecule, the dominant spurious signals suggest that the intercalation may have occurred with a form of caffeine.

This study successfully extracted, quantified, and characterized caffeine from three commercially available Bangladeshi tea brands, Meena, Ispahani, and Seylon and further demonstrated the kinetics of caffeine extraction time in our practical daily life uses of tea-time and conditions. In addition, intercalation into a Mg–Al layered double hydroxide (LDH) matrix was formulated by extracting caffeine from the local brand tea [30]. Among the tested samples, Ispahani tea showed the highest caffeine yield (4.72%), followed by Meena (4.07%) and Seylon (3.15%), corresponding to caffeine contents of approximately 94 mg, 80 mg, and 63 mg per tea bag, respectively. The physical characteristics of the isolated caffeine light white colour, crystalline texture, water solubility, and bitter taste were consistent with those of standard caffeine. FT-IR spectroscopy confirmed the presence of key functional groups (C=O, C–N, and C=N), closely matching the reference spectra, indicating successful extraction and high purity of the compound. UV–Visible spectroscopic analysis revealed an absorption maximum at 272 nm, characteristic of the $\pi \rightarrow \pi^*$ transition in caffeine's conjugated purine ring, confirming its molecular identity [31]. Quantitative analysis based on Beer–Lambert's law showed that caffeine concentration increased with steeping time in all samples, reaching maximum levels after 5 minutes of brewing 15.3 mg per 50 mL for Ispahani, 14.6 mg for Meena, and 14.8 mg for Seylon. These results fall within the typical caffeine content range reported for black and green teas (15-60 mg per serving).

Table 4. FT-IR spectral data for the layered double hydroxide (aluminum-magnesium chloride).

| Serial | Position ($cm^{-1}$) | Assignment |
|---|---|---|
| 1 | 752.43 | Aromatic C-H bending |
| 2 | 970.24 | C=C stretching |
| 3 | 1421.54 | C-O-H bending |
| 4 | 1649.14 | Asymmetric stretching of carboxylic group |
| 5 | 2405.23 | O-H stretching |
| 6 | 3219.19 | N-H stretching |

## CONCLUSIONS

This study extracted and measured caffeine from three commercial tea brands, Meena, Ispahani, and Seylon using solvent extraction and UV–visible spectrophotometry. Ispahani had the highest caffeine yield (4.72%), followed by Meena (4.07%). The best brewing time was 5 minutes. At 100 °C, caffeine extraction followed zero-order kinetics, with a reported rate constant of 0.039 $M^{-1}$ $s^{-1}$. FTIR, NMR ($^{1}H$ and $^{13}C$), and mass spectrometry confirmed the isolated caffeine's identity and purity. The study also synthesized caffeine-intercalated magnesium–aluminum layered double hydroxide (Mg–Al LDH) using urea hydrolysis. FTIR results confirmed intercalation, suggesting possible uses in controlled-release materials and drug delivery. The work shows how compounds from everyday natural sources can be used to create functional materials. Future research could improve caffeine intercalation and examine release under physiological conditions. Researchers could also test other plant-derived compounds, scale up the process using greener solvents or microwave-assisted extraction, and explore applications in pharmaceuticals, cosmetics, and smart packaging. Overall, this study connects natural product chemistry with materials science to support the development of sustainable functional nanomaterials.

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