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OPTIMIZATION OF CAFFEINE EXTRACTION FROM DRIED COFFEE BEANS: EVALUATING THE EFFECTS OF SOLVENT RATIO AND TIME IN A CONTINUOUS SOLID-LIQUID EXTRACTION PROCESS FOR PHARMACEUTICAL APPLICATIONS

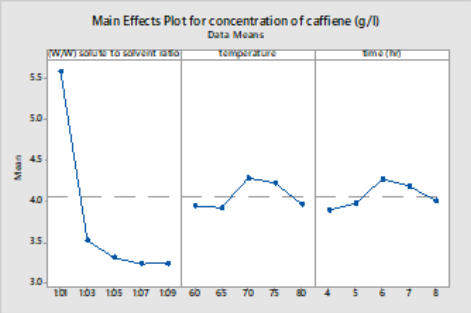
Wid Numan Mohammed*, Salih Rushdi

Department of Chemical Engineering, University of Al-Qadisiyah, Al-Qadisiya, Iraq

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*Corresponding author
wid.numan.mohammed@qu.edu.iq

Graphical abstract



Abstract

The efficient extraction of caffeine from coffee beans is critical for pharmaceutical and food industries, yet the optimization of process parameters remains a challenge. This study investigates the use of Soxhlet extraction combined with Taguchi statistical methodology to optimize caffeine extraction from dried coffee beans. Twenty-five experimental runs were designed to evaluate the effects of solute-to-solvent ratio, extraction temperature, and extraction time on caffeine concentration and recovery efficiency. The results revealed that the solute-to-solvent ratio is the most influential factor, with higher ratios (1:7–1:9) achieving over 90% extraction efficiency, while lower ratios (1:1) led to higher caffeine concentration but incomplete extraction due to rapid solvent saturation. The optimal extraction conditions were determined to be 70°C and 6 hours, with temperature and time showing minor effects. The statistical model was validated by residual analysis. Compared to previous studies, this work demonstrates improved extraction efficiency and provides practical recommendations for large-scale caffeine extraction. These findings offer valuable insights for enhancing industrial processes in pharmaceutical and food applications.

Keywords: Caffeine extraction, Soxhlet, Taguchi method, Optimization, Extraction efficiency

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1.0 INTRODUCTION

Caffeine is one of the most widely consumed psychoactive substances globally, present in coffee, tea, soft drinks, and various pharmaceuticals. Its physiological and pharmacological effects, especially as a central nervous system stimulant, have made caffeine an essential compound in both the food and pharmaceutical industries [1, 2]. Extraction of caffeine from plant materials, particularly coffee beans, remains a subject of ongoing research due to the need for efficient, cost-effective, and environmentally friendly processes [3, 4]. Various extraction techniques have been developed and applied to optimize caffeine recovery, including conventional methods such as heat reflux and infusion, as well as advanced techniques like microwave-assisted extraction (MAE), supercritical fluid extraction (SFE), and Soxhlet extraction [5-7]. Among these, the Soxhlet method is

recognized for its efficiency in solid-liquid extraction, though its performance is highly dependent on process parameters such as temperature, extraction time, and the solute-to-solvent ratio [8]. More recently, deep eutectic solvents (DES) have been introduced as promising alternatives for caffeine extraction due to their low toxicity and high selectivity; however, these methods often require complex solvent preparation and have not yet been widely adopted in large-scale applications [9, 10].

To address these challenges, this study employs the Taguchi statistical method to systematically optimize the main factors influencing caffeine extraction from dried coffee beans using the Soxhlet technique. This approach enables comprehensive evaluation of the effects and interactions of temperature, extraction time, and solute-to-solvent ratio, aiming to establish practical recommendations for industrial-scale extraction processes.

While previous studies on caffeine extraction from coffee beans have investigated the influence of process parameters, most have focused on single-factor experiments or limited parameter ranges, often without considering statistical optimization approaches. This work provides a novel contribution by applying the Taguchi statistical method with an L25 orthogonal array to simultaneously evaluate and optimize three key parameters—solute-to-solvent ratio, temperature, and extraction time—in Soxhlet extraction using ethanol. The study identifies the solute-to-solvent ratio as the dominant factor for both caffeine concentration and recovery, establishes optimal operating conditions (70 °C, 6 h, and 1:7–1:9 ratio), and compares the outcomes with literature data for deep eutectic solvents (DES), highlighting the practical advantages and limitations of each approach. These findings offer actionable guidance for both laboratory and industrial-scale caffeine extraction processes in pharmaceutical and food applications.

The organization of this paper is as follows: Section 2 describes the materials and methodology, including experimental design and statistical analysis. Section 3 presents and discusses the results. Section 4 concludes the main findings and suggests future directions.

2.0 METHODOLOGY

The Taguchi method is an efficient statistical approach used to optimize process parameters with minimal experimental runs, making it particularly suitable for multifactorial experiments like extraction optimization [11]. In this study, the Taguchi method was employed to systematically evaluate the effects of three main factors—solute-to-solvent ratio, extraction temperature, and extraction time—on caffeine extraction from dried coffee beans using the Soxhlet apparatus.

Three process variables were selected as control factors:

- Solute-to-solvent ratio (five levels: 1:1, 1:3, 1:5, 1:7, 1:9 w/w).
- Extraction temperature (five levels: 60°C, 65°C, 70°C, 75°C, 80°C).
- Extraction time (five levels: 4, 5, 6, 7, and 8 hours).

Resulting in 25 experimental runs. This design reduces the number of experiments required compared to a full factorial approach, while still enabling the estimation of main effects and selected interactions with statistical validity [12].

For each experimental condition, caffeine concentration and extraction efficiency were measured as response variables. The collected data were analyzed using Minitab 18 software to compute the signal-to-noise (S/N) ratios and to identify the optimal levels for each factor. The statistical significance of each parameter was further assessed by Analysis of Variance (ANOVA), ensuring the robustness of the findings.

This Taguchi-based approach facilitated the identification of the most influential parameter and provided reliable recommendations for maximizing caffeine extraction under practical laboratory and industrial conditions. Table 1 displays the process variables.

Table 1 Process variables with their levels

Variables	Minimum	maximum
Sample-to-solvent ratio(w/w)	1:1	1:9
Temperature (°C)	60	80
Time (hr)	4	8

According to the Taguchi array, twenty-five experiments were conducted to extract caffeine from coffee using a Soxhlet apparatus with ethanol as the solvent. Figure 1 shows the experimental apparatus for this process, The chemical process in this study is carried out according to the following steps:

The procedure includes the preparation of a specified volume of solvent (ethanol) and a solid material dried coffee beans (DCB). The sample is placed in the sample holder. Next, the Soxhlet apparatus is assembled over a round-bottom flask, and the condenser is connected to the apparatus. One of the basic requirements is that the sample holder extends over the solvent drain tube. Lastly, a recirculating water chiller keeps the condenser cool while heating the solvent to the required temperature. The T80 dual-beam absorption spectrophotometer, which has a spectral resolution of 2 nanometers and a large LCD screen, Standard samples with known concentrations were prepared and examined in the device to obtain absorbance.

Subsequently, the unknown concentration of the resulting extract samples was analyzed in the device, and the concentration was calculated using the equation (1)

[y=0.0981x-0.123] (1)



Figure 1 Soxhlet extractor

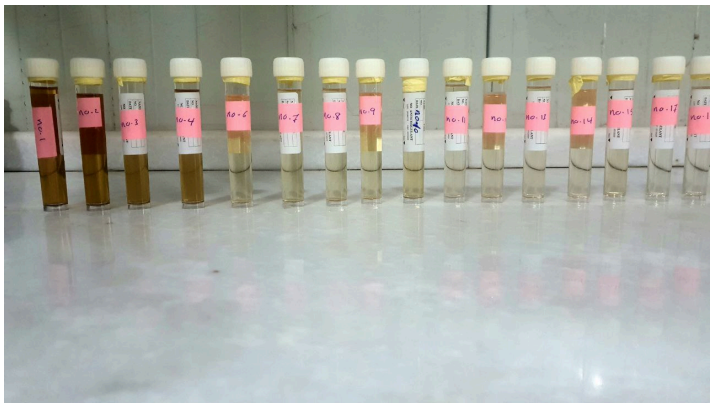


Figure 2 Samples of the extract resulting from the extraction process

3.0 RESULTS AND DISCUSSION

The graph in Figure 3 represents the Main Effects Plot for the extracted caffeine concentration (grams/liter), showing the effect of three main factors on the caffeine concentration.

3.1 Effect of the ratio of (DCB) to the solvent

The main-effects plot in Figure 3 confirms that the solute-to-solvent ratio is the dominant driver of the measured extract concentration. At 1:1, the extract concentration is maximal (~5.6 g/L) because dilution is minimal; however, the overall recovery is limited by rapid solvent saturation and constrained mass transfer within the solid matrix. Increasing the ratio to 1:3–1:9 progressively lowers the measured concentration (g/L) due to dilution, yet improves overall recovery by sustaining a higher concentration gradient (ΔC), enhancing diffusion, and delaying saturation—consistent with solid–liquid extraction studies on coffee and spent coffee grounds [9, 13-15].

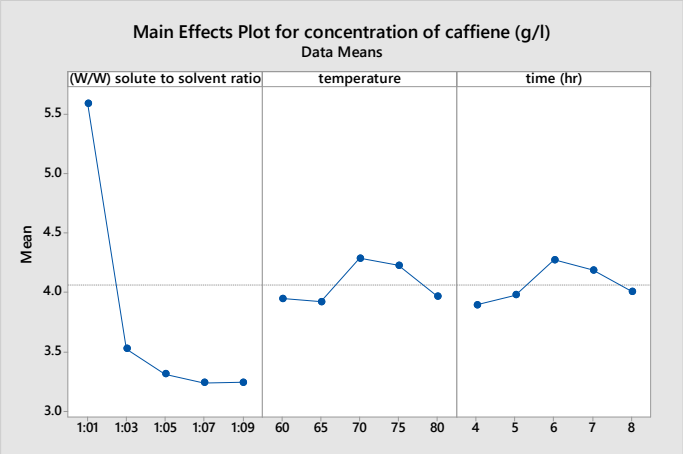


Figure 3 main effects plot for concentration of caffeine

In comparison with Deep Eutectic Solvents (DES) often show higher selectivity toward caffeine, but their higher viscosity introduces mass-transfer resistance; practical protocols typically compensate with larger solvent-to-solid ratios, co-solvent dilution, vigorous agitation, or ultrasound assistance [16-18]. In contrast, ethanol–Soxhlet benefits from low viscosity and high diffusivity, allowing efficient mass transfer even at lower ratios.

3.2 Effect of Temperature

In Figure 3, we observe that within the range of 60-80 degrees Celsius, the effect of temperature is slight, with a shallow maximum around ~70 degrees Celsius followed by a slight decrease. Under Soxhlet conditions with ethanol (operating near the solvent's boiling point), caffeine solubility is already near-saturated across much of this range; further heating yields diminishing returns. Similar narrow thermal windows—where gains in solubility/diffusivity are balanced by co-extraction or thermal effects—have been reported for coffee matrices and related bioactives [10, 19-21].

When comparsion soxhlet with DES, Many DES/NADES systems can achieve efficient extraction at lower temperatures (≈40–60 °C) due to specific hydrogen-bonding/ionic interactions that enhance solvation, potentially reducing energy demand. However, operating colder increases viscosity, so

mass-transfer enhancement (agitation/ultrasound) becomes critical to match Soxhlet rates [17, 18, 22].

3.3 Effect of Extraction Time

In Figure 3, we observe that the extraction time shows a nonlinear pattern: the concentration increases from 4 hours to about 6 hours (with a maximum of approximately 4.2 grams/liter), then decreases slightly at 7-8 hours. The early stages reflect rapid washing and the spread of large pores; while the later stages are limited in the spread of fine fields/within the granules, approaching equilibrium around 6-7 hours. Extended reflux may also promote co-extraction of non-targets or minor thermal changes, explaining the small decline in measured concentration—consistent with reports that >85–90% of caffeine is extracted in the first half of the total time for coffee systems [10, 21, 23].

In comparison Soxhlet with DES, DES-based protocols can reach high recoveries in shorter times when ultrasound or intense stirring is applied to overcome viscosity-limited transport; without intensification, DES may require longer contact than Soxhlet to attain comparable yields, especially for dense matrices or coarser grinds [16, 18, 24].

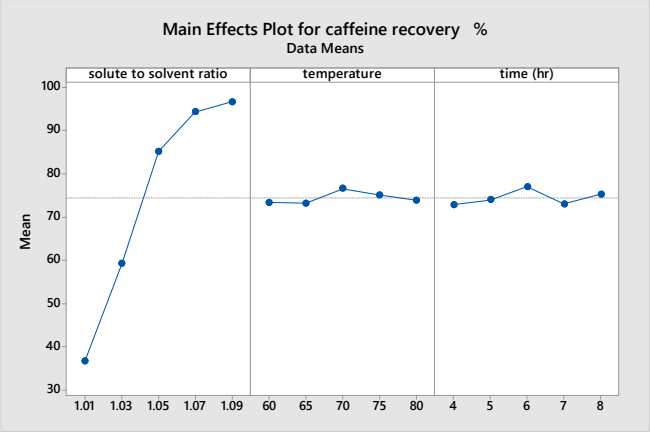


Figure 4 main effects plot for recovery of caffeine

Figure 4 The Main Effects Plot for Caffeine Recovery % shows how three factors—solute to solvent ratio (DCB), temperature, and extraction time—affect caffeine recovery efficiency.

3.4 Effect of the ratio of (DCB) to the solvent on caffeine recovery

Figure 4 show a strong positive correlation was observed between the increase in solvent volume and the caffeine recovery rate. At a very low ratio of dry matter to solvent (1:1), the caffeine recovery was remarkably poor, achieving only about 35%, likely due to rapid solvent saturation and insufficient solubilization of all the available caffeine content. As the solvent ratio increased to 1:7 and 1:9, the recovery rate improved significantly, reaching values of over 90%, indicating a much more efficient extraction. These results suggest that higher solvent-to-solid ratios greatly enhance the mass transfer process and facilitate more complete caffeine recovery from the plant matrix. This trend aligns with established findings in solid–liquid extraction processes, where larger solvent volumes increase the concentration gradient, reduce solvent saturation, and enhance compound diffusion and desorption [25, 26].

3.5 Effect of Temperature on caffeine recovery

From Figure 4 temperature shows minimal impact on caffeine recovery within the tested range. Recovery values remain relatively constant (~72–76%), with only a small peak near 70 °C. In Soxhlet extraction with ethanol, operating near the solvent's boiling point ensures high caffeine solubility throughout the range, making temperature less critical for recovery compared to solvent ratio.

Similar weak thermal effects have been reported in studies on coffee extraction, where recovery was more strongly influenced by solvent volume and particle size than by temperature, once above a certain threshold [10, 19, 21, 20].

3.6 Effect of Extraction Time on caffeine recovery:

In Figure 4, time shows a slight and limited effect on retrieval efficiency. It increases slightly at the sixth hour to reach about 75%, then stabilizes thereafter. This means there is a relatively ideal time (about 6 hours) with no significant difference at other times [23].

As a final conclusion, the primary factor that clearly determines the efficiency of caffeine recovery is the ratio of solute to solvent. Temperature and time have relatively limited effects. The ideal ratio for complete caffeine recovery is high solvent ratios (1:07 or 1:09).

3.7 Analysis of Variance using (ANOVA)

ANOVA analysis showed that the ratio of solute to solvent is the most important factor that significantly affects the extracted caffeine concentration ($P = 0.000$), while the factors of temperature and time do not have a clear significant effect ($P > 0.05$). Therefore, it is better to focus on adjusting the solute-to-solvent ratio to achieve the best results, while changes in temperature and time within the tested limits do not have a significant impact.

Table 2 Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
solute to solvent ratio	4	13294.7	3323.68	233.08	0.000
temperature	4	39.8	9.96	0.70	0.608
time (hr)	4	59.9	14.96	1.05	0.422
Error	12	171.1	14.26		
Total	24	13565.5			

3.8 Interaction Effects

Figure 5 shows that the concentration of extracted caffeine is significantly affected by the solute-to-solvent ratio, with the lowest ratio (1.01) clearly yielding the highest concentration. Additionally, the optimal extraction time is approximately 6 hours, and the optimal temperature is 70°C. The change in caffeine concentration at higher solvent ratios is very limited, and there is no strong interaction between the factors at those ratios. Therefore, optimizing the solute-to-solvent ratio, time, and temperature within these limits yields the best results for caffeine extraction.

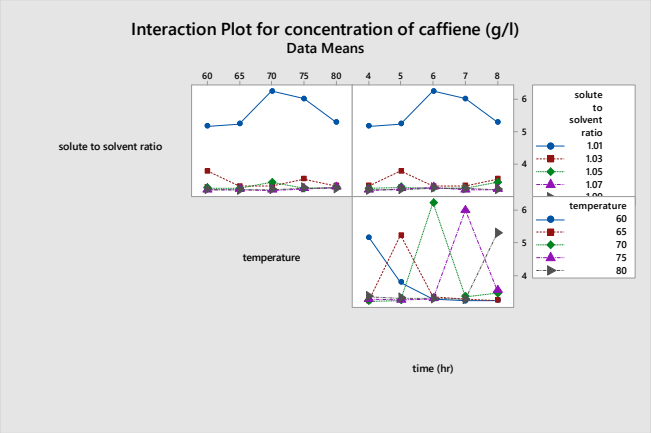


Figure 5 Interaction Effects Plot for caffeine concentration.

Figure 6 illustrates the interaction effect among the three factors (solute-to-solvent ratio, temperature, and time) on caffeine recovery efficiency (Caffeine Recovery %). The interaction plot showed that there is a strong interaction between time and temperature on caffeine extraction efficiency. Therefore, it is important to adjust these two factors together to achieve optimal extraction efficiency. While the ratio of solute to solvent is the primary factor in enhancing extraction efficiency, its effect is significantly stable and not clearly influenced by the other two factors.

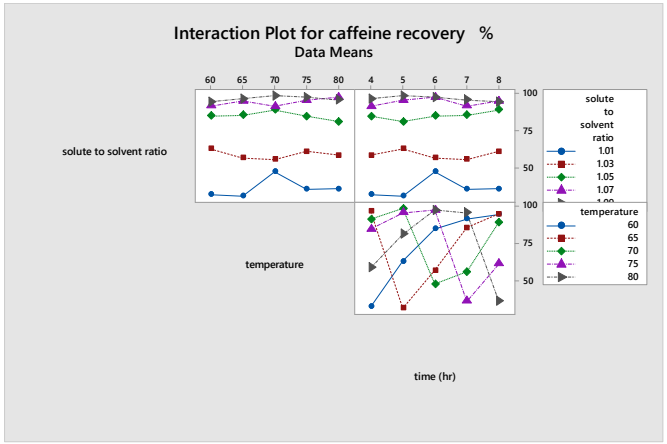
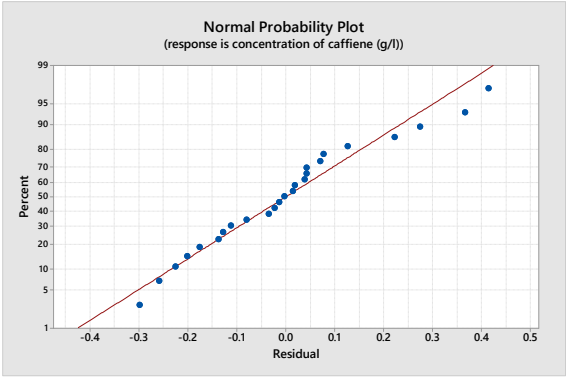


Figure 6 Interaction Effects Plot for Caffeine Recovery % .

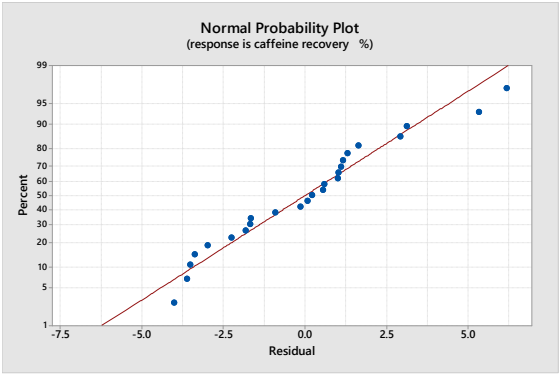
3.9 Residual Analysis

In Figure 7 through the residual analysis of both responses (caffeine extraction efficiency and caffeine concentration), it was found that the model used is correct and suitable for the data. The normal distribution of the residuals showed that the statistical assumptions were met (normal distribution and homogeneity of variance), and there were no significant deviations affecting the accuracy of the results or the validity of the model.

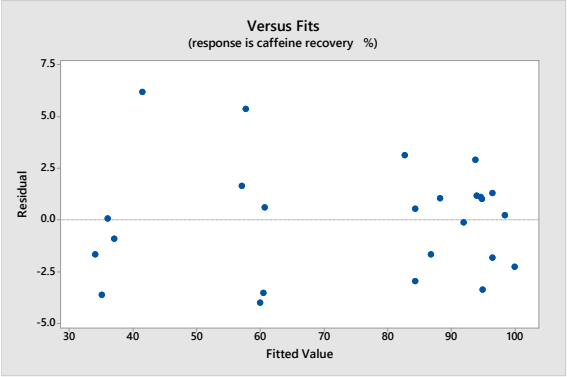
a)



b)



c)



d)

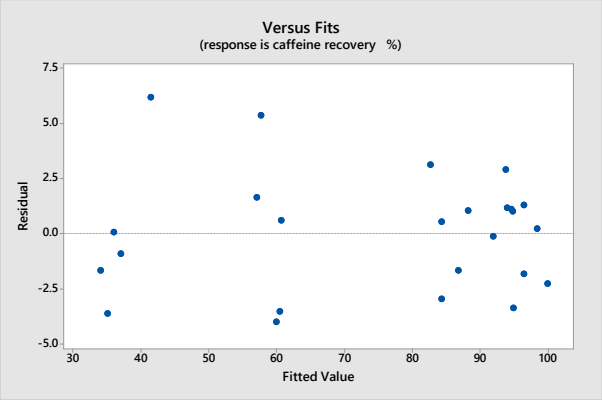


Figure 7 (a), (b), (c) and (d) Residual plots

4.0 CONCLUSION

This study optimized caffeine extraction from dried coffee beans using Soxhlet–ethanol and the Taguchi method. The solute-to-solvent ratio was the most influential factor, with higher ratios (1:7–1:9) achieving over 90% recovery, while lower ratios (1:1) yielded higher concentrations but incomplete extraction. Temperature (optimal at 70 °C) and time (optimal at 6 h) had minor effects within the tested range. Compared with reported DES-based methods, Soxhlet–ethanol provided faster mass transfer and higher efficiency without process intensification, offering practical guidance for industrial applications.

5.0 LIMITATIONS AND FUTURE WORK

This study was conducted using a single extraction technique (Soxhlet) with ethanol as the solvent, and the optimization focused on three factors—solute-to-solvent ratio, temperature, and extraction time—within a specific range of values. Other influential parameters, such as particle size, solvent type, and pretreatment methods, were not examined and may further affect extraction performance. In addition, the experiments were performed on a laboratory scale, and potential scale-up challenges, such as energy consumption, solvent recovery efficiency, and process economics, were not assessed.

Future research could explore the use of alternative green solvents, such as deep eutectic solvents (DES), under optimized mass-transfer enhancement techniques (e.g., ultrasound-assisted extraction) to compare performance with ethanol–Soxhlet. Investigating the combined effect of multiple physical and chemical parameters, applying real industrial-scale trials, and evaluating the economic and environmental impacts of the optimized process would further strengthen the applicability of the findings.

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Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

References

[1] van Dam, Rob M., Frank B. Hu, and Walter C. Willett. 2020. Coffee, Caffeine, and Health. *New England Journal of Medicine*. 383(4): 369–78. https://doi.org/10.1056/NEJMra1816604.

[2] Fredholm, Bertil B., Karl Bättig, Jan Holmén, Astrid Nehlig, and Edwin E. Zvartau. 1999. Actions of Caffeine in the Brain with Special Reference to Factors That Contribute to Its Widespread Use. *Pharmacological Reviews*. 51(1): 83–133.

[3] Derry, Sheena, Christopher J. Derry, and R. Andrew Moore. 2014. Caffeine as an Analgesic Adjuvant for Acute Pain in Adults. *Cochrane Database of Systematic Reviews*. 2014(12). https://doi.org/10.1002/14651858.CD009281.

[4] Weldegebreal, Biniyam, Mesfin Redi-Abshiro, and Bhagwan Singh Chandravanshi. 2017. Development of New Analytical Methods for the Determination of Caffeine Content in Aqueous Solution of Green Coffee Beans. *Chemistry Central Journal*. 11: 1–9. https://doi.org/10.1186/s13065-017-0356-3.

[5] Alamri, G., and S. Rushdi. 2024. Advances in the Synthesis and Manufacturing Techniques of Ibuprofen: A Comprehensive Review. In *AIP Conference Proceedings*. Melville, NY: AIP Publishing. https://doi.org/10.1063/5.0237359.

[6] Lo Coco, Francesco, Francesca Lanuzza, Giuseppe Micali, and Nunzio Cappello. 2007. Determination of Theobromine, Theophylline, and Caffeine in By-Products of Cupuacu and Cacao Seeds by High-Performance Liquid Chromatography. *Journal of Chromatographic Science*. 45(5): 273–75. https://doi.org/10.1093/chromsci/45.5.273.

[7] Wale, K., and B. Girma. 2023. An Overview of Techniques for Extracting Caffeine from Coffee for Quantification. *American Journal of Chemical and Biochemical Engineering*. 7(2): 15–19. https://doi.org/10.11648/j.ajcbe.20230702.11.

[8] Zaid, H., *et al.* 2023. Investigative Study of Olives Oil Extraction Using Economic Methods. In *AIP Conference Proceedings*. Melville, NY: AIP Publishing. https://doi.org/10.1063/5.0150143.

[9] Efthymiopoulos, Ioannis, *et al*. 2019. Effect of Solvent Extraction Parameters on the Recovery of Oil from Spent Coffee Grounds for Biofuel Production. *Waste and Biomass Valorization*. 10: 253–64. https://doi.org/10.1007/s12649-017-0061-4.

[10] Fuller, Michael, and Niny Z. Rao. 2017. The Effect of Time, Roasting Temperature, and Grind Size on Caffeine and Chlorogenic Acid Concentrations in Cold Brew Coffee. *Scientific Reports*. 7(1): 17979. https://doi.org/10.1038/s41598-017-18247-4.

[11] Roy, Ranjit K. 2010. *A Primer on the Taguchi Method*. Dearborn, MI: Society of Manufacturing Engineers.

[12] Hossain, M. S., *et al*. 2025. Sustainable Coloration and Functionalization of Silk Fabric Through Chinese Fringe Leaves (*Loropetalum chinense*) Extraction Using the Taguchi Method: An Alternative for Waste Management. *Fibers and Polymers*. 26(2): 739–54. https://doi.org/10.1007/s12221-024-00836-6.

[13] Hassan, S. R., and A. M. Al-Yaqoobi. 2024. Extraction of Caffeine from Spent Coffee Ground by Solid-Liquid Extraction. *Baghdad Science Journal*. 21(6): 2017–17. https://doi.org/10.21123/bsj.2023.8721.

[14] Soeswanto, B., *et al.* 2023. The Effect of Solvent-to-Coffee Ratio on Caffeine Content in Ethyl Acetate Extracts of Arabica Gayo Coffee Beans. *KOVALEN: Jurnal Riset Kimia*. 9(3): 224–31. https://doi.org/10.22487/kovalen.2023.v9.i3.16573.

[15] Gigliobianco, Maria Rita, *et al.* 2020. Optimization of the Extraction from Spent Coffee Grounds Using the Desirability Approach. *Antioxidants*. 9(5): 370. https://doi.org/10.3390/antiox9050370.

[16] Cai, C., *et al.* 2019. Deep Eutectic Solvents Used as the Green Media for the Efficient Extraction of Caffeine from Chinese Dark Tea. *Separation and Purification Technology*. 227: 115723. https://doi.org/10.1016/j.seppur.2019.115723.

[17] Ahmad, I., *et al.* 2021. Optimization of Betaine-Sorbitol Natural Deep Eutectic Solvent-Based Ultrasound-Assisted Extraction and Pancreatic Lipase Inhibitory Activity of Chlorogenic Acid and Caffeine Content from Robusta Green Coffee Beans. *Heliyon*. 7(8): e07702. https://doi.org/10.1016/j.heliyon.2021.e07702.

[18] Jeliński, Tomasz, and Piotr Cysewski. 2022. Quantification of Caffeine Interactions in Choline Chloride Natural Deep Eutectic Solvents: Solubility Measurements and COSMO-RS-DARE Interpretation. *International Journal of Molecular Sciences*. 23(14): 7832. https://doi.org/10.3390/ijms23147832.

[19] Raheem, S., *et al.* 2024. Caffeine Extraction from Spent Coffee Grounds by Solid-Liquid and Ultrasound-Assisted Extraction: Kinetic and Thermodynamic Study. *Iraqi Journal of Chemical and Petroleum Engineering*. 25(1): 49–57. https://doi.org/10.31699/IJCPE.2024.1.5.

[20] Olechno, E., *et al.* 2021. Influence of Various Factors on Caffeine Content in Coffee Brews. *Foods*. 10(6): 1208. https://doi.org/10.3390/foods10061208.

[21] Vandeponseele, A., *et al.* 2021. Study of Influential Parameters of the Caffeine Extraction from Spent Coffee Grounds: From Brewing Coffee Method to the Waste Treatment Conditions. *Clean Technologies*. 3(2): 335–50. https://doi.org/10.3390/cleantechnol3020019.

[22] Hikmawanti, N. P. E., *et al.* 2021. Natural Deep Eutectic Solvents (NADES): Phytochemical Extraction Performance Enhancer for Pharmaceutical and Nutraceutical Product Development. *Plants*. 10(10): 2091. https://doi.org/10.3390/plants10102091.

[23] Barroso, L. A., *et al.* 2022. Optimization of the Brewing Parameters on Coffee Extraction Using a Central Composite Rotatable Design. *JSFA Reports*. 2(3): 107–15.

[24] Loukri, A., *et al.* 2022. Green Extraction of Caffeine from Coffee Pulp Using a Deep Eutectic Solvent (DES). *Applied Food Research*. 2(2): 100176. https://doi.org/10.1016/j.afres.2022.100176.

[25] Wang, X., *et al.* 2020. Distribution Survey, Phytochemical and Transcriptome Analysis to Identify Candidate Genes Involved in Biosynthesis of Functional Components in *Zanthoxylum nitidum*. *Industrial Crops and Products*. 150: 112345. https://doi.org/10.1016/j.indcrop.2020.112345.

[26] Raithore, S., and Devin G. Peterson. 2018. Effects of Polyol Type and Particle Size on Flavor Release in Chewing Gum. *Food Chemistry*. 253: 293–99. https://doi.org/10.1016/j.foodchem.2018.01.123.