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ABSTRACT

In this study, in order to confirm the potential of coffee waste as a recyclable resource material, the caffeine content of coffee waste and the effective active ingredients of coffee waste extract were confirmed using an ultrasonic extraction method. In order to conduct a preliminary experiment to determine whether extraction of coffee waste affects caffeine reduction, ultrasonic extraction was performed after immersion at room temperature for 48 hours at ethanol content of 0%, 30%, 50%, 70%, and 100%. The coffee waste recovered after extraction were analyzed for active ingredients using GC-MS.

Except for the coffee waste after extraction with 100% ethanol, almost no components other than caffeine were detected in the coffee waste recovered after extraction by ethanol content. As a result of quantitative analysis of caffeine in coffee grounds using LC-MS, a caffeine reduction rate of 72.1% was confirmed in the coffee grounds recovered after ultrasonic extraction with 70% ethanol.

Keywords: coffee waste, recycle, caffein, ultrasonic extraction, decaffein.

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ABSTRACT

In this study, to confirm the potential of coffee waste as a recyclable resource material, the caffeine content of coffee waste and the effective active ingredients of coffee waste extract were established using an ultrasonic extraction method. To conduct a preliminary experiment to determine whether extraction of coffee waste affects caffeine reduction, ultrasonic extraction was performed after immersion at room temperature for 48 hours at ethanol content of 0%, 30%, 50%, 70%, and 100%. The coffee waste recovered after extraction was analyzed for active ingredients using GC-MS. Except for the coffee waste after extraction with 100% ethanol, almost no components other than caffeine were detected in the coffee waste recovered after extraction by ethanol content. As a result of quantitative analysis of caffeine in coffee grounds using LC-MS, a caffeine removal rate of 72.1% was confirmed in the coffee grounds recovered after ultrasonic extraction with 70% ethanol.

As a result of a preliminary experimental study, this study was conducted to confirm whether repeated re-extraction removed more than 90% of the caffeine content in the coffee waste and met the decaffeination standard. Based on the results of a preliminary experiment that confirmed the highest caffeine reduction rate in ultrasonic extracted coffee waste with 70% ethanol content, extraction was repeated 1 to 3 times using coffee waste with 70% ethanol content in the same manner as the preliminary experiment. As a result of quantitative analysis of caffeine in coffee waste using LC-MS in this experiment, a high reduction rate of 99.5% of caffeine content was confirmed to be removed from coffee waste recovered by repeated extraction with 70% ethanol three times.

Keywords: coffee waste, recycle, caffein, ultrasonic extraction, decaffein.

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I. INTRODUCTION

The development of the food industry and food processing technology, along with the improvement of the standard of living of modern people, has shaped the coffee culture [1, 2, 3]. Coffee has become an essential item in the preferences of contemporary individuals, to the extent that it is often referred to as the "second water"[4]. How to make a cup of Americano, coffee is extracted using water, roasted coffee beans, The seeds of the coffee tree's fruit[5].

During the coffee extraction process, coffee waste, which refers to the leftover residues after the coffee extraction process, is generated. When making a cup of coffee, approximately 15 grams of coffee beans are used, with 14.7 grams being discarded, and 0.3 grams, which is 2%, is consumed as a beverage [6]. Coffee waste is generated worldwide at a rate of 10 million tons annually, and the amount of coffee waste is increasing by coffee consumption. In 2019, an 149,038 tons of coffee waste were produced [7, 8]. If such coffee waste is incinerated or landfilled without recycling, it incurs an annual cost of KRW 35 billion [9. 10].

Decaffeination refers to removing caffeine from coffee beans, cocoa, tea leaves, and other

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Decaffeination refers to the process of removing caffeine from coffee beans, cocoa, tea leaves, and other substances that contain caffeine. Therefore, decaffeinated coffee typically contains 1-2% of the original caffeine component, making it suitable for pregnant women, diabetes patients, and individuals with sensitive stomachs, and it is also known as 'caffeine-free' coffee [11]. The international standard for decaffeinated coffee is that more than 97% of caffeine is removed. Therefore, even in decaffeinated coffee, a small amount of caffeine may still be contained [12].

Caffeine is a compound formed by combining 'Coffee' with '-ine,' representing an alkaloid substance that collectively refers to basic organic compounds found within plant organisms. Alkaloid substances often exhibit significant physiological and pharmacological effects, with notable examples including nicotine, morphine, and caffeine. Caffeine has a stimulating effect on the central and the peripheral nervous system, which results in mental alertness and an increase in concentration and productivity [13, 14].

Therefore, caffeine may have some benefits for adults, but its impact on the central nervous system of children has not been sufficiently researched yet. The central nervous system of children is still in development, which raises concerns about the potential side effects and health implications of caffeine [15]. Caffeine can be absorbed through the skin, so caution is necessary when using caffeine-containing substances in items for children, such as educational aids.

Research on the utilization of coffee waste includes studies on artificial cover material manufacturing for sewage sludge using coffee sludge [16], activated carbon adsorbents [17], eco-friendly composting [18], wastewater treatment [19], coffee soap, and air freshener production [20]. However, the actual usage of coffee waste remains minimal. Furthermore, recently, some clay products for children have incorporated recycled materials, including coffee waste. However, existing clay products made with recycled coffee waste have limitations for use by children due to factors such as caffeine content. Therefore, educational aids made from recycled coffee waste should minimize caffeine content, taking into account the age and sensitivity of children. Therefore, the purpose of this study is to confirm the reduction of caffeine using a repeated extraction method with coffee waste, investigate its decaffeination potential, assess the feasibility of coffee waste as a sustainable resource, and check its safety and potential as a recyclable resource material.

II. EXPERIMENT

2.1 Materials

The coffee waste used in this study were provided by Harmony Co., Ltd. in the form of coffee ground powder in February 2023 and were stored at room temperature for conducting experiments.

2.2 Extraction Condition Experiment for Coffee Waste I

For Experiment I of coffee waste extraction, distilled water and ethanol were used as solvents. The volume was set at 300 ml, with a sample weight of 30 g. Ethanol was used in varying concentrations of 0%, 30%, 50%, 70%, and 100%, and extraction was carried out at room temperature for 48 hours after immersion. The ultrasonic extraction conditions involved using an extraction device (ULTRA SONIC, BMWORKS, Daejeon, Korea) at a temperature of 20°C, operating at 20 kHz for 30 minutes. The extracted solution was subjected to decompressed filtration through a 5-8 ㎛ filter (Hyun Dai, Micro, Korea) using a vacuum pump (DOA-P704-AC, GAST Manufacturing Inc, U.S.A). After the filtration, the coffee extract was dried in a 60°C Coven for 48 hours and stored at 4°C for GC-MS analysis and caffeine analysis.

2.3 Repeated Extraction Experimental Conditions for Coffee Waste Ⅱ

For the repeated extraction of coffee waste in Experiment II, the same solvents, water and ethanol, were used as in Experiment I. For the ultrasonic extraction conditions for Experiment II, the volume was set at 300 ㎖, with a sample weight of 30 g, and 70% ethanol was used. The extraction was carried out in the same way as

Experiment 1 at room temperature for 48 hours after immersion. After filtration, the coffee waste was dried in a 60°C oven for 48 hours, and the process of obtaining the coffee waste extraction material was repeated 1-3 times.

2.4 Caffeine Quantification in Coffee Waste Using LC-MS

The caffeine content in coffee waste was analyzed using HPLC (Agilent 1290). After a 50 mg sample was measured and injected into a vial, 2 ml of methanol was added, and sonication was carried out for 60 minutes. The analysis was conducted using a 0.45 ㎛ PVDF syringe filter. An LC column, Kinetex 2.6 ㎛ EVO C18 100Å 2.1×100 mm, was used. The column temperature was maintained at 40°C, with a flow rate of 0.300 mL/min, and an injection volume of ㎕. The MS was conducted under the conditions of a gas temperature of 300°C, a drying gas flow rate of 10 L/min, and a nebulizer pressure of 45 psi. The LC-MS analysis method for this experiment was carried out using in the same way.

2.5 Effective Active Ingredients Analysis in Coffee Waste Extract Using GC-MS

For the analysis of effective active ingredients in the coffee waste extract for ExperimentI, GC-MS (Gas chromatography-mass spectrometry) was used. For the GC analysis, a column (Agilent 19091S-433) was used with HP-5ms (30 m x 250 ㎛ x 0.25 ㎛), and the analysis was conducted for 45 minutes. The injection volume was ㎕, and the carrier gas was helium maintained at a flow rate of 10 ㎖/min. The oven temperature was initially held at 40°C for 3 minutes and then increased at a rate of 5°C/min to a final temperature of 300°C, which was held for 1 minute during the analysis.

III. RESULTS AND CONSIDERATIONS

The results for the coffee waste recovered after ultrasonic extraction in this study are as follows. The coffee waste recovered after extraction in Experiment I was labeled as original materials (ORG) and A to E, depending on the ethanol content.

The coffee waste recovered after extraction with 0% ethanol ultrasonic extraction is labeled as A, 30% ethanol as B, 50% ethanol as C, 70% ethanol as D, and 100% ethanol as E, as shown in <Table 1>.

Table 1: Numbering Based on the Ethanol Content of the Extract by Ultrasonic Extractor

DW : Ethanol	Ultrasonic extractor
100 : 0	A
70 : 30	B
50 : 50	C
30 : 70	D
0 : 100	E

In Experiment II, depending on the number of extractions, the recollected materials were labeled as original materials (ORG) and G1 to G3.

The coffee waste recovered after ultrasonic extraction with 70% ethanol is labeled as G1 for the first extraction, G2 for the second extraction, and G3 for the third extraction, as shown in <Table 2>.

Table 2: Numbering Based on the Ethanol Content of the Extract by Ultrasonic Extractor

DW : Ethanol	Number of Repeated Extractions	Ultrasonic extractor
30 : 70	1 time	G1
	2 times	G2
	3 times	G3

3.1. Caffeine Content Analysis of Coffee Waste Using LC-MS

The caffeine content of coffee waste analyzed using LC-MS is as follows.

As shown in <Table 3 & Figure 1>, the 70% ethanol ultrasonic coffee waste exhibited the lowest caffeine content at 404.89 µg/g, showing a 72.1% reduction in caffeine compared to the initial caffeine content of the ORG <Table 4>.

Table 3: LC-MS Quantitative Analysis Results for Caffeine in the Coffee Waste Extract by the Ultrasonic Device Used in the Experiment

Sample	mg	g	Caffeine Content in 2 ml of Extracted Methanol (µg)	A 10-Fold Dilution factor	Final Concentration µg/g
ORG	46.9	0.0469	6.809	68.093	1451.9
A	47.4	0.0474	6.676	66.763	1408.5
B	53.4	0.0534	3.976	39.760	744.57
C	50.5	0.0505	3.841	38.411	760.61
D	50.7	0.0507	2.053	20.528	404.89
E	51.3	0.0513	6.650	66.504	1296.4

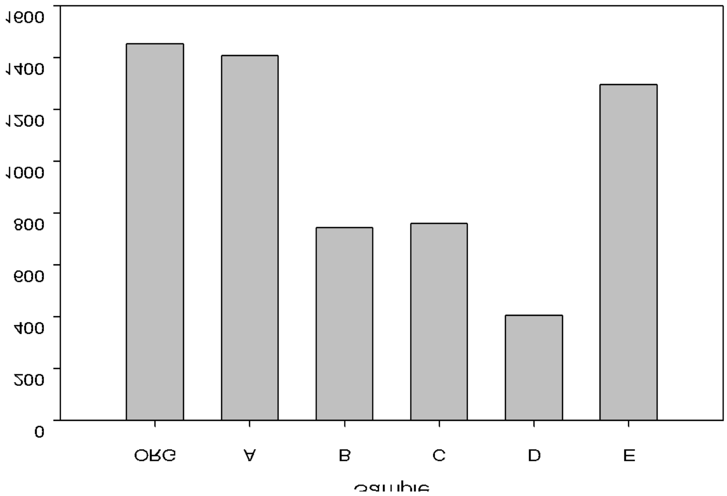


Figure 1: Comparison of LC-MS Quantitative Analysis Results of Caffeine in Coffee Waste Extract by Ultrasonic Device Used in the Experiment;

Table 4: Final Concentration Reduction Rate as a Result of LC-MS Quantitative Analysis of Caffeine in the Coffee Waste Extract by the Ultrasonic Device Used in the Experiment

Sample	Final Concentration (µg/g)	Reduction Rate (%)
ORG	1451.9	-
A	1408.5	3.00
B	744.57	48.7
C	760.61	47.6
D	404.89	72.1
E	1296.4	10.7

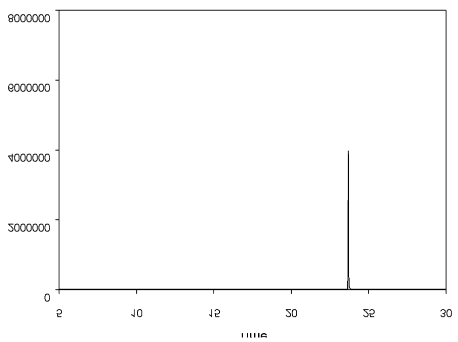
3.2 GC-MS Analysis of Effective Active Ingredients in Coffee Waste

The GC-MS analysis results of effective ingredients in the recovered coffee waste after extraction are presented in <Table 5>. No other compounds were detected in the coffee waste recovered after ultrasonic extraction except for caffeine. However, in the coffee waste recovered after extraction with 100% ethanol, compounds such as 2,4-Dimethyl-1-heptene, n-hexadecanoic acid, and Benzene were detected. The GC-MS results graph of the coffee waste recovered after extraction, based on the ethanol content, is shown in Figure 2.

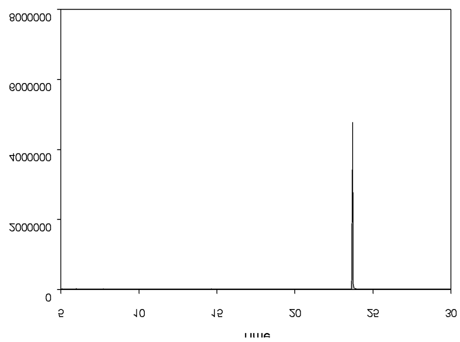
Table 5: Results of GC-MS Analysis of Coffee Waste Extract by Ultrasonic Extractor

(Unit: Area %)

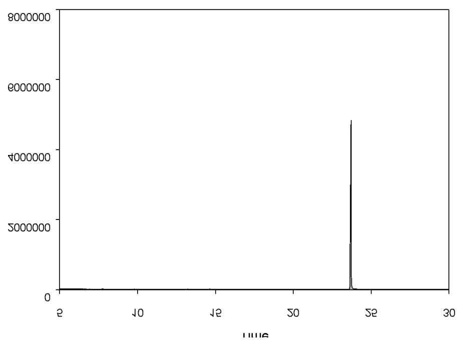
Extract Active Ingredient	Caffeine	2,4-Dimethyl-1-hEptene	n-Hexadecanoicacid (Palmitic Acid)	Benzene
A	100	-	-	-
B	100	-	-	-
C	100	-	-	-
D	17.7	-	-	-
E	19.3	13.4	1.88	10.4



(A)



(B)



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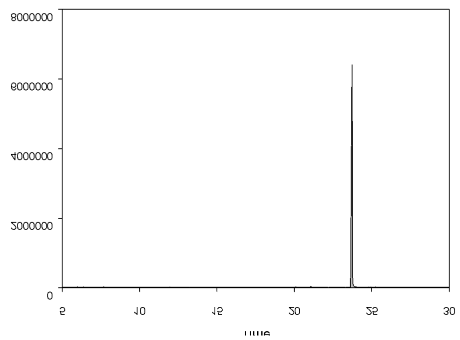
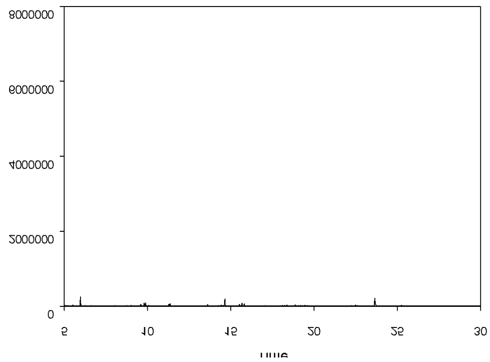


(D)



(E)

Figure 2: Comparison of GC-MS Analysis Results of the Coffee Waste Extract by the Ultrasonic Extractor

3.2 Quantitative Analysis Results of Caffeine Using LC-MS for This Experiment

The caffeine content in the coffee waste recovered after ultrasonic extraction is as follows.

As shown in <Table 7 and 8>, a significant caffeine reduction rate of 99.5% was observed in the coffee waste obtained from ultrasonic extraction using 70% ethanol after three times of repeated extraction. The Korea Food and Drug Administration has confirmed that decaffeination, with 90% removal of caffeine, can be achieved with just two repeated experiments of coffee waste extraction <Figure 3>.

Table 6: LC-MS Quantitative Analysis Results for Caffeine in the Coffee Waste Extract by the Ultrasonic Device Used in the Experiment

Sample	mg	g	Caffeine Content in 2 Ml of Extracted Methanol (μg)	A 10-Fold Dilution Factor	Final Concentration μg/g
ORG	53.7	0.0537	12.8	128.1	2386.1
G1	50.0	0.0500	3.22	32.22	644.49
G2	52.0	0.0520	3.55	3.550	68.230
G3	50.6	0.0506	0.59	0.590	11.730

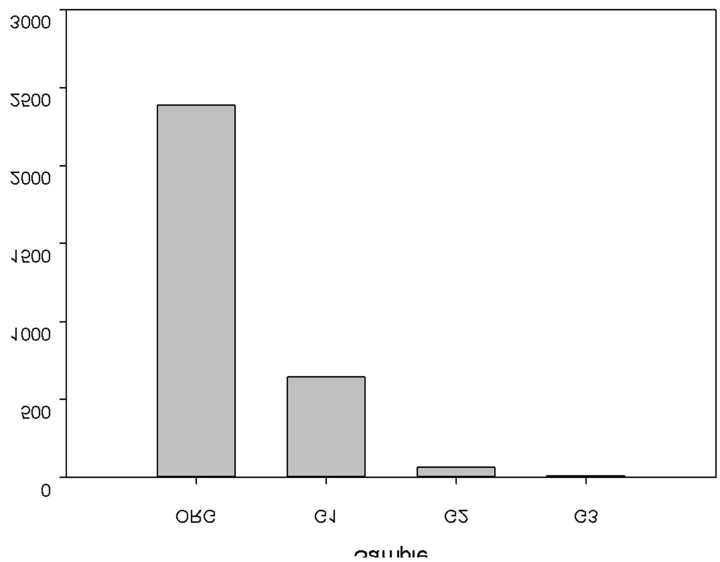


Figure 3: Comparison of LC-MS Quantitative Analysis Results of Caffeine in Coffee Waste Extract by Ultrasonic Device in the Experiment;

Table 7: Final Concentration Reduction Rate as a Result of LC-MS Quantitative Analysis of Caffeine in the Coffee Waste Extract by the Ultrasonic Device in the Experiment

Sample	Final Concentration (μg/g)	Reduction Rate (%)
ORG	2386.1	-
G1	644.49	73.0
G2	68.230	97.2
G3	11.730	99.5

VI. CONCLUSION

This study utilized ultrasonic extraction to investigate the caffeine content and effective active ingredients in coffee waste, confirming its potential as a recyclable resource The ultrasonic extraction method was used for coffee waste extraction, and preliminary experiments were conducted under conditions ranging from 0% to 100% ethanol content. The GC-MS analysis results of effective ingredients in coffee waste revealed that, except for the coffee waste recovered after 100% ethanol extraction, hardly any other components were detected in coffee waste recovered after extraction with different ethanol contents. The LC-MS analysis of caffeine content revealed a 72.1% reduction in caffeine in the coffee waste recovered after 70% ethanol ultrasonic extraction, compared to the coffee waste without ultrasound extraction.

Furthermore, additional research was conducted to confirm whether the caffeine content within coffee waste could be reduced by over 90% to meet decaffeination standards. The re-extraction was performed using 70% ethanol, which showed the highest caffeine reduction rate among the ethanol concentration ranges. In addition, repeated extraction was performed 1 to 3 times, and the extraction method was ultrasonic extraction conducted under preliminary experimental conditions. The quantitative analysis of caffeine in coffee waste using LC-MS revealed a high caffeine removal rate of 99.5% in the coffee waste recovered after three times repeated extractions with 70% ethanol. As a result of this study, the potential for decaffeination through the repeated ultrasonic extraction of coffee waste was confirmed, and this verified its potential as a recyclable resource material.

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REFERENCES

1. J. Y. Choi, A Study on Purchasing Characteristics and Consumer Sat is faction on Health Functional Beverage according to Food-related Lifestyle Hotel Resort Research, 12(1), pp.179-196. (2012).
2. Scott K, The sweet and the bitter of mammalian taste, Curr Opi Neurobiol 14: pp. 423-427, (2004).
3. Emile Peynaud, The Taste of wine, The wine appreciation guild, pp. 31-114, 1987.
4. H. U. Park, H. E. Lee, World Coffee Industry Trends, World agricul ture, No. 145, pp. 97-116, (2012).
5. Robert HR, Barone J J. Biological effects of caffeine-history and use. Food Technol, 37:32-39.(1983).
6. S. W. Kim. A Study on the Utilization ofSpentCoffeeasAnimalFeed. ThesisKonkukUniversity.p11-13. (1990).
7. I. C. O (International Coffee Organization) resaerch, rade Statistics Tables, World coffee consumption. https://www.ico.org/trade_statistics.asp?section=Statistics. (2021)
8. Y. J. Park, 5 trend changes and prospects in the coffee industry, Korea Economic Review, Vol. 848, No. 0, Hyundai Research Institute, 1p, (2019),
9. K. M. Kim, Y. S. Park. A plan to turn bio energy into fuel by establishing a coffee grounds collection system. See p.7 (2020).

10. National Assembly Research Service. (Ministry of Foreign Affairs > News, Notices > Press Release, (2021).
11. Zabot GL. Decaffeination using supercritical carbon dioxide. In: Green Sustainable Process for Chemical and Environmental Engineering and Science, Elsevier, Amsterdam, Nederlanden, p 255-278 (2020).
12. KCS (Korea Customs Service). Coffee (bean) import trends in 202 2. Available from: https://www.customs.go.kr/streamdocs/view/sd;stream%20amdocsId=72059269162294321 , Accessed May. 10, (2023).
13. Pohler, H., Caffeine intoxication and addiction, J. Nurse Pract., 6, 49-52, (2010).
14. O'Neil, M. J., The Merck Index - An encyclopedia of chemicals, drugs, and biologicals. 14th edition., White house Station, NJ, Merck and Co., Inc, (2013).
15. J. E. Lee, W. Huh, E. J. Choi, Pattern Analysis of High-caffeine Energy Drink Consumption and Adverse Effects among College Students in a University. Yakhak Hoeji. 57(2), (2013).
16. G. Y. Lee, H. H. Hyeon, T. J. Ryu, J. Y. Yoon, W. S. Lee, C. H. Lee, K. J. Park, "Re-evaluation of artificial cover for sewage sludge using Busan phosphorus gypsum and coffee sludge". Journal of the Korean Society of Waste and Resource Circulation 2011, pp.224-226, (2011).
17. H. Y. Kang, "Adsorption characteristics of activated carbon prepared from coffee grounds", PhD thesis, Chonnam National University Graduate School, (2007).
18. H. S. Kim, "Eco-friendly composting using coffee by-products", master's thesis, Kwangwoon University Graduate School of Environmental Studies, (2012).
19. C. H. Song, C. H. Lee, T. L. Huh, J. H. Ahn, H. C. Yang, Development of substrates for the production of basidiocarps of Flammulina velutipes. Korean J Mycol.21:212-216, (1993).
20. E. J. Song, J. Y. Kim, S. Y. Lee, S. Y. KIM, K. B. W. R. Kim, S. J. Kim, S. Y. Yoon, S. J. Lee, C. J. Lee, D. H. Ahn, Effect of roasted ground coffee residue extracton shelf-life and quality of salted mackerel.J Korean SocFood SciNutr.38:780-786, (2009).