Proceedings of the International MultiConference of Engineers and Computer Scientists 2009 Vol II
IMECS 2009, March 18 - 20, 2009, Hong Kong

Influence of Extraction Manufacturing Process on Caffeine Concentration

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***Abstract*—Coffee is quite popular of the leisure drink, and the ingredients of coffee include caffeine, aroma, protein, tannic acid and fat et al. The references show a small amount of caffeine can stimulate the brain and enhance memory, if caffeine is over the amount that heart, high blood pressure, kidney and coronary artery would be caused a negative impact. In this paper, we use the special ultrasound method with high-frequency, well penetrating power and the sound wave to extract the caffeine. And also, a new extraction equipment is designed to make the coffee powder and extraction liquid of water mixed efficiently. The different operating conditions for extraction experiments are executed and the obtained results are also compared. The results show that caffeine has already reached the saturated condition at 15 second of extracting time and the quantity of caffeine is increased with temperature raised. However, the influence of vibration frequency is not significant.**

***Index Terms*—Ultrasound 、 extraction 、 caffeine**

I. Introduction

The use of focused high-frequency acoustic energy, and its attendant penetrating power, promises to be an energy efficient and chemical-neutral method of extraction. When high-frequency acoustic energy is used in conjunction with a specific mechanical vibration/agitation within the medium then the extraction process is further enhanced. This is due to cavitations and the thermal properties that result from agitation. This technique has become common in contemporary food processing and storage methods [1]. It was found that the sonication-assisted extraction of ginseng saponins was about three times faster than the traditional extraction method. Ultrasound extraction was not only more efficient but also a more convenient process for the recovery and purification of the active ingredients of plant materials [2]. In addition, ultrasound extraction can enhance energy and transfer speed, and improve the speed of the solvent's absorption of the extracts [3]. This is achieved by destroying the cell walls and thereby diffusing the desired extracts throughout the medium. This in turn makes the desired extractions more accessible to the solvents in the medium. The technology of ultrasound extraction has improved upon the shortcomings of traditional solvent extraction by reducing the processing time and working without the use of harmful solvents. At the same time this method produces higher-yield rates than traditional extraction methods. Because this process operates at low temperature it reduces heat loss, and avoids losing or destroying volatile substances that have a low boiling point.

Caffeine is found in a variety of food and drinks. It is the most commonly ingested stimulant in the world. When used in moderation, caffeine can stimulate the brain and enhance memory, inspire enthusiasm, clarify thought, reduce fatigue and sleepiness. However excessive amounts of caffeine can cause anxiety, feelings of unrest, heart palpitations, headaches, diarrhea and insomnia among other negative effects. Some literature indicates that caffeine can have a detrimental effect on human health. These negative effects include exacerbating coronary artery disease, raising blood pressure, and heightening the risk of heart attack and kidney disease [4,5].

In their research regarding the effects of extraction processes on aromatic constituents, Céline Sarrazin et al compared the resultant aromatic oil extractions of five different extraction methods: including Press Oil Aroma Extraction (oil), Supercritical Fluid Extraction (SFE), Simultaneous Distillation-Extraction (SDE). Vacuum Steam-Stripping with water (VSS water) and Vacuum Steam-Stripping with methylene chloride (VSS CH2Cl2). Sarrazin's studies concluded that the VSS water process produced the best results for extracting coffee's aromatic components [6].

This work was supported in part by the National Science Council, Taiwan under Grant NSC-97-2221-E-269-024 & NSC-96-2622-E-269-035-CC3.

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This paper looks specifically at a special high-frequency ultrasound method that utilizes sound waves to extract caffeine. Our paper also considers new extraction equipment that is designed to optimize the consistency of the coffee powder and extraction liquid to allow for a highly efficient extraction process. The extraction experiments were executed under a wide spectrum of operating conditions and the obtained results compared.

II. Experiment Method

This experiment uses an ultrasound machine to extract caffeine from coffee. By controlling the temperature, wave frequency, and time duration, of each extraction we can measure these variables affects on the caffeine content of each extraction as well as a comparative analysis of the extraction liquid by High Performance Liquid Chromatography (HPLC).

A. Experiment Material and Equipment：

TABLE I
Material Table of Experimental

Type / Project	Material Name	Material Varieties
1	RO Water	- - -
2	Coffee powder of Italy	Arabica and Robusta Origin: Vietnam

TABLE II
Equipment Table of Experimental

Type / Project	Equipment Name	Equipment Model
1	Grinding Machine	YU CHI MACHINERY D3V-10
2	Filter	40mesh
3	Water Bath	HIPOINT BC-2D
4	Refrigerated Circulation Bath	YIH DER BL-720
5	Ultrasonic Cleaning Equipment	(1) DELTA DC200H (2) DELTA DC600H
6	High Performance Liquid Chromatography (HPLC)	(1) Pump： HITACHI L-2130。 (2) Auto Sampler： HITACHI L-2200 (3) Column Over： HITACHI L-2300 (4) Diode Array Detector： HITACHI L-2450 (5) RI Detector： HITACHI L-2490

B. Method of Experiment：

Table 3 illustrates the control parameters in this experiment. These include: tank temperature and solution temperature that ranged between 5℃ to 95℃. Each experiment used 300ml of RO water and 30 grams of coffee powder. The operating frequency was either 28KHz or 42KHz. The oscillation periods were either 15 seconds or 30 seconds. The crucial variable in this experiment was the solution temperature. The experiment was conducted using increments of 5℃. Each extraction was then tested for caffeine content.

TABLE III
Control Parameters of Experimental

No. / Conditions	1	2	3	4	5	6	7
Temperature of Tank (℃)	5	20	35	50	65	80	95
Temperature of Solution (℃)	5	20	35	50	65	80	95
RO Water (ml)	300	300	300	300	300	300	300
Coffee powder (g)	30	30	30	30	30	30	30
Operating Frequency (KHz)	28/42	28/42	28/42	28/42	28/42	28/42	28/42
Time of Oscillation(s)	15/30	15/30	15/30	15/30	15/30	15/30	15/30

C. Structure of Experiment：

As shown in Figure 1, this equipment was built from a Refrigerated Circulation Bath, a Water Bath, and Ultrasonic Cleaning Equipment. To ensure a consistent and accurate temperature for each experiment a circulation bath was used to either raise or lower the temperature of the coffee/RO solution. If the required operating temperature was lower than 25℃, the Refrigerated Circulation Bath would cool the liquid mixture from room temperature to the desired temperature; if a temperature of more than 25℃ was required, the Water Bath would heat the liquid mixture from room temperature to desired temperature. When the correct temperature for the specific experiment was reached the coffee powder was mixed into the RO water. This mixture was then processed through one of two separate ultrasonic cleaning equipment machines. One ran at 28 KHz for a duration of 15 seconds and the other ran at 42 KHz for a duration of 15 seconds. The results of these extractions were then collected in collection tank 1 and collection tank 2 respectively. The process was then repeated for a duration of 30 seconds. This extraction was also added to the tanks. Each collection tank then held a solution of two extractions. One tank held a solution of extracts that had been processed for 15 and 30 seconds using 28 KHz and the other tank held a solution of extracts that had been processed for 15 and 30 seconds using 42 KHz. Experiment structure shown in Figure 1：

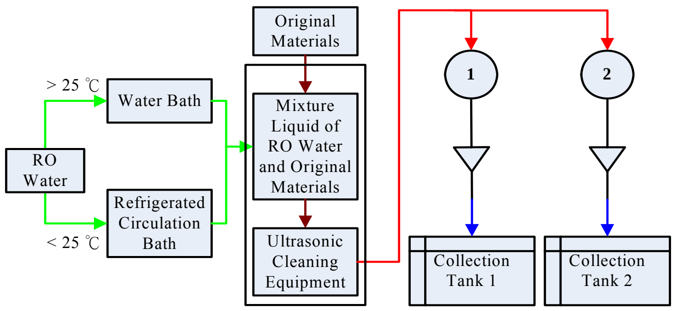

Fig. 1. Structure of Experiment

D. *Experiment Method*:

First, a grinder was used to crush coffee beans into a fine consistency and filtered through a 40 mesh filter. When the temperature reached the operating temperature, a mixture of coffee powder and RO water was placed into the ultrasound machine, and then processed under various extraction conditions, as show in Table 3. The extraction liquid was initially filtered through a 40 mesh filter and collected. In order to achieve a ratio of extraction liquid to water of 1:9 for a final dilution of 10 times, the extraction liquid was further filtered through a 0.45μm filter paper. And then, the caffeine content was used as a base for comparative analysis of the HPLC.

The integrity of the process and analysis was verified by using the known caffeine content of a standardized coffee. The results of the caffeine content of the standardized coffee were compared to the analysized caffeine content of the sample provided by the issuing laboratory. If these results were not similar the experiment was repeated with adjustment to the operating conditions.

Finally, these results were tabulated and the comparisons indicated the optimum values for time/frequency and temperature that would produce the greatest concentrations of caffeine. The experiment's process is shown in Figure 2：

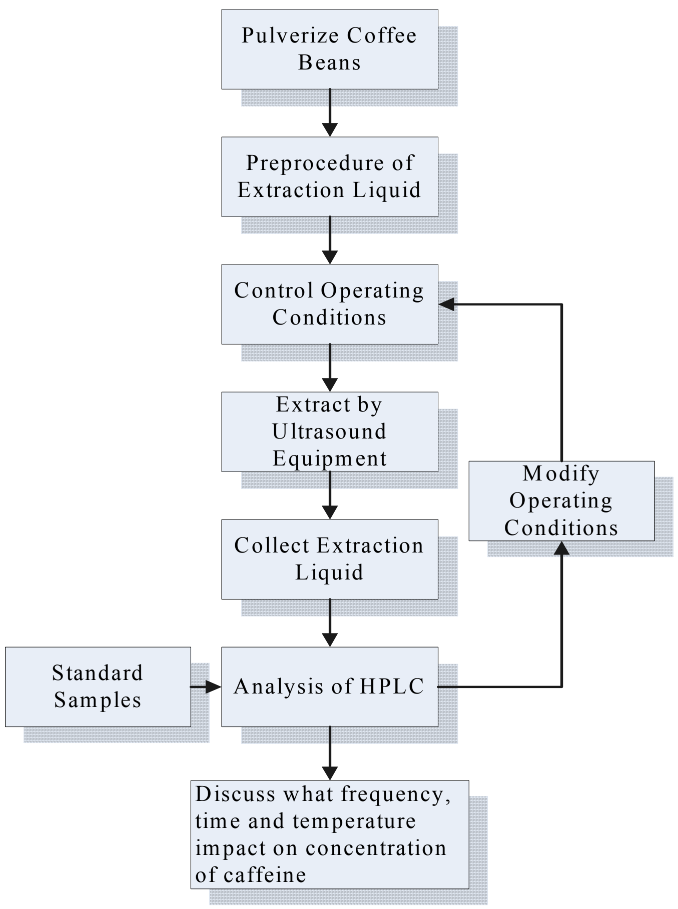

Fig. 2. Process of Experiment

III. Results and Discussion

A. *Analysis of HPLC*:

Figure 3 shown the results from an operating frequency of 28 KHz and an extraction time of 30 seconds, the temperature were 5℃, 80℃ and 95℃, which resulted in concentrations of 16ppm, 1202ppm and 1422ppm respectively, of caffeine content. Figure 4 shows the results of an operating frequency of 42 KHz, an extraction time of 30 seconds, and temperatures of 5℃, 80℃ and 95℃. These extractions produced concentrations of 67ppm, 1120ppm and 1208ppm respectively, of caffeine content.

A comparison between Figure 3 and 4 illustrates that with all other variables constant, an increase in temperature results in higher concentrations of caffeine in the extraction. At the same time, the results show that an increase in the ultrasound frequency does not significantly affect the caffeine content.

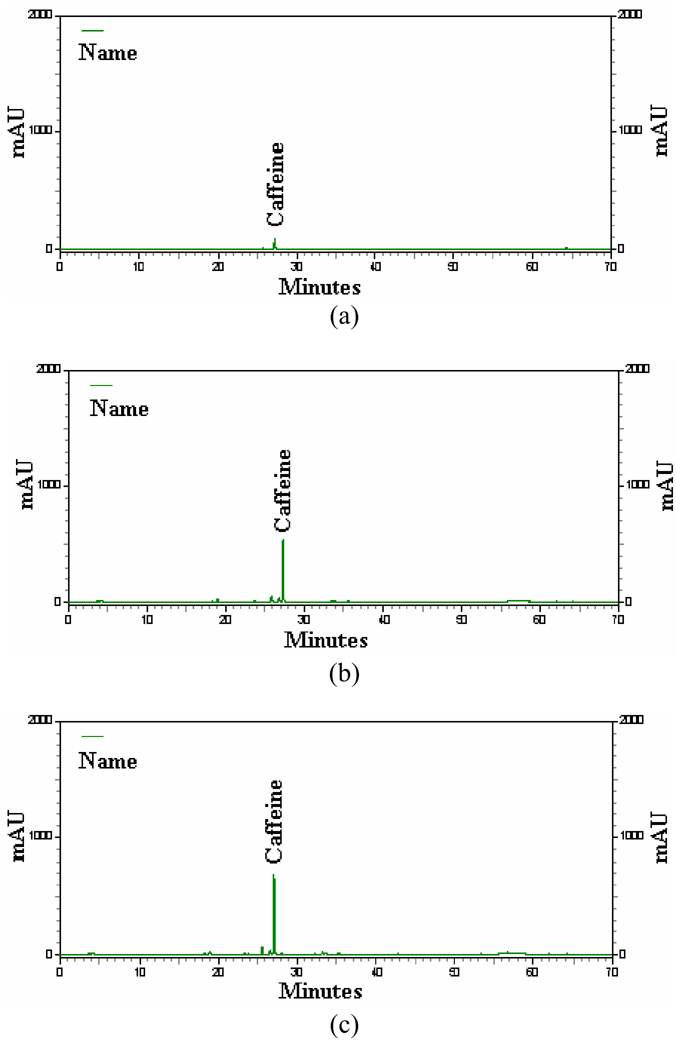

Fig. 3. Operating Frequency is 28KHz, work time at 30sec, pictures of caffeine extract content by control tank temperature in (a)5℃, (b)80℃, (c)95℃.

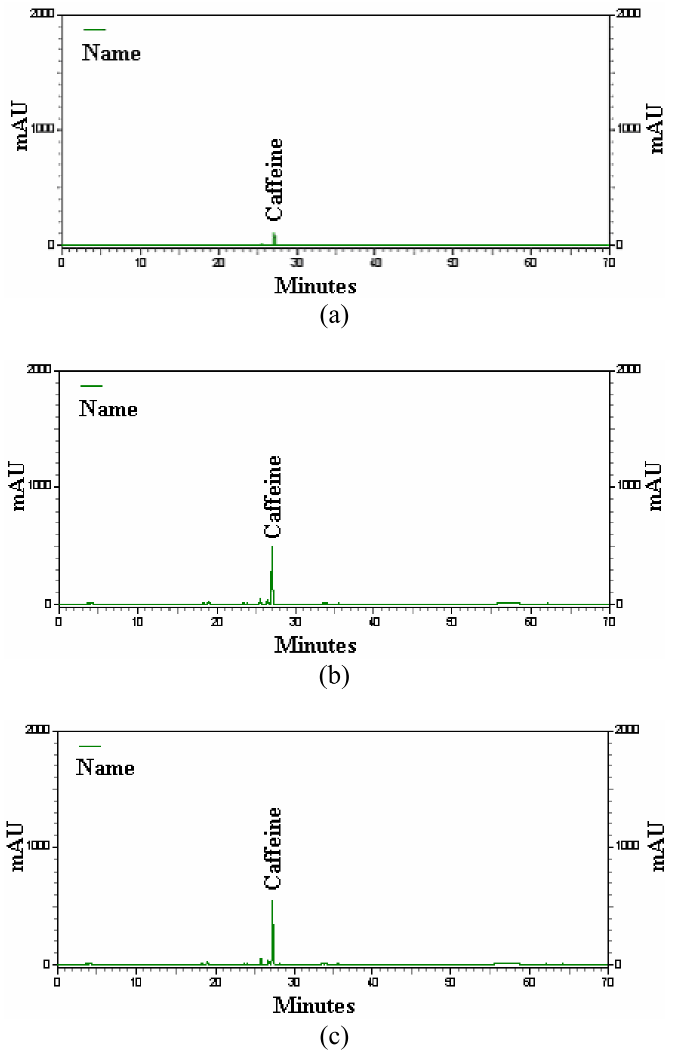

Fig. 4. Operating Frequency is 42KHz, work time at 30sec, pictures of caffeine extract content by control tank temperature in (a)5℃, (b)80℃, (c)95℃.

B. Influence of Ultrasound Operating Frequency on caffeine content :

Through the use of a standardized experimental procedure, and a 1:10 dilution of the liquid coffee extract, we can use an HPLC analysis, to plot a curve of caffeine content by converting the resultant data. (As shown in figure 5 and figure 6.) The experiment's data shows that caffeine content increases with an increase in temperature. When the operating temperature was 5℃, and the extraction time was 15 seconds, the caffeine content extracted from high operating frequency (42KHz) was nine times higher than when using a low operating frequency (28KHz) under the same conditions. In a low temperature state a higher frequency has a substantially better extraction result than when using a lower frequency. But as we reach higher operating temperatures the effect of frequency does not has as strong an effect. With all variables the same the largest gap between caffeine concentrations from low-frequency and high-frequency extracts, during a 30 second extraction, was only 15%.

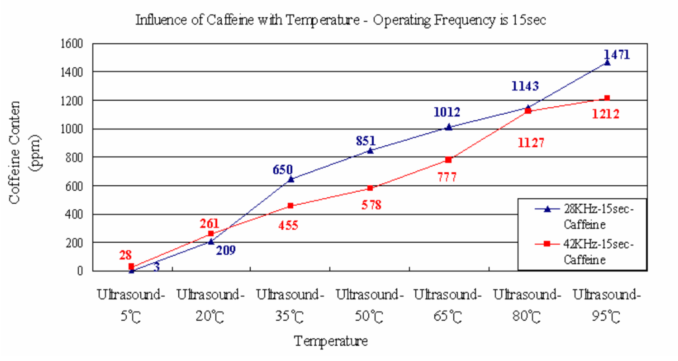

Fig. 5. Operating Frequency are 28KHz and 42KHz, temperature is 15 seconds.

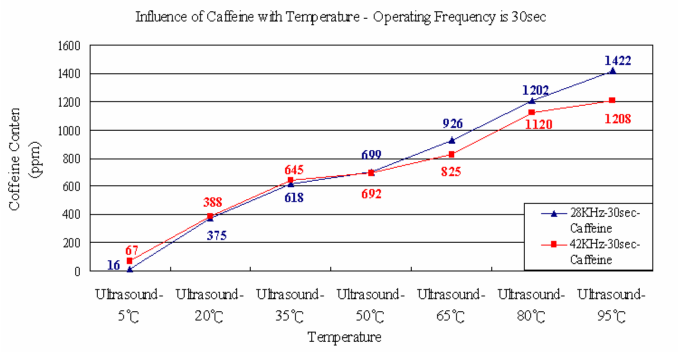

Fig. 6. Operating Frequency are 28KHz and 42KHz, temperature is 30 seconds.

C. Influence of extraction time on caffeine content :

The optimum temperature range for making coffee is between 80℃ and 95℃. Within this temperature range the coffee's aroma is fragrant and components and caffeine content smell very fragrant for consumer at this temperature. So, extraction time at 15 seconds and 30 seconds, the caffeine content of error value at 80℃ and 95℃ are about range of 5% (As shown in figure 7); and the error value was less than 1% (As shown in figure 8), apart from the extraction time longer with caffeine content of higher, at the same time, also proved that caffeine content to gain saturated at 15 seconds.

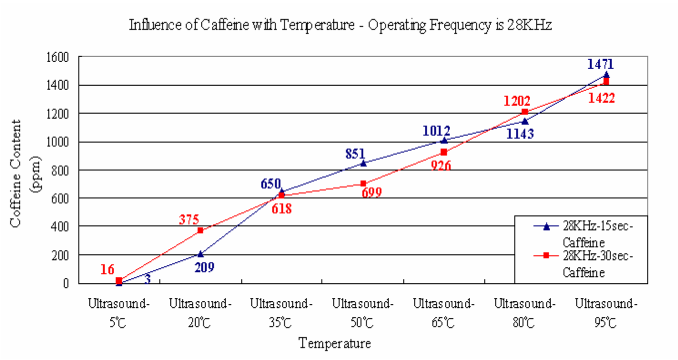

Fig. 7. Operating Frequency is 28KHz, temperature are 15 seconds and 30 seconds.

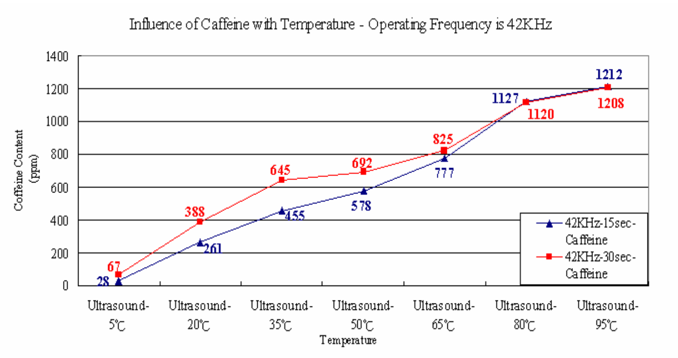

Fig. 8. Operating Frequency is 42KHz, temperature are 15 seconds and 30 seconds.

IV. Conclusion

From the results of these experiments we can conclude that, the influence of temperature is the biggest factor in determining caffeine content of an extraction. Specifically, an increase in temperature results in a higher concentration of caffeine. At the same time, the specific extraction time for specific coffee varieties and each also has a different saturation temperature. In particular, the caffeine concentrations that were produced by an extraction time of 15 seconds and an extraction time of 30 seconds were similar. The curve of the graphed results nearly overlaps. The results indicate that the caffeine content after a 15 second process nears the saturation point. The results indicate that by increasing the ultrasound frequency during a low temperature process the concentration of caffeine increase significantly. However, within a high temperature process an increase in the ultrasound frequency does not show a corresponding increase in caffeine concentration. This paper illustrates the results of experiments on caffeine extraction using ultrasound at various operating temperatures. The results indicate the influence of temperature, extraction duration, and ultrasound frequency. The provide insight into creating a more efficient, in terms of time and energy consumption, manufacturing process for caffeine extraction from coffee.

Acknowledgment

This paper supported in part by National Science Council in R.O.C. under grant No. NSC-97-2221-E-269-024 & NSC-96-2622-E-269-035-CC3, express many thanks.

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