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308 Lasani Town, Sargodha Road, Faisalabad - Pakistan
Mob: +92 300 3008585, Fax: +92 41 8815544
E-mail: editorpjn@gmail.com

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Effect of Time and Water Temperature on Caffeine Extraction from Coffee

Pham Phuoc Nhan and Nguyen Tran Phu
Department of Biochemistry and Plant Physiology, College of Agriculture and Applied Biology,
Cantho University, 3-2 Street, Cantho City, Vietnam

Abstract: The stimulating effect of coffee is mainly depended on caffeine availability in products. Caffeine levels in grains of two coffee species, *Coffea arabica* and *Coffea robusta*, have been quantified via the maximal absorbance of standard caffeine at 288 nm. Three popular coffee brands in Vietnam were selected for quality assessment based on the qualitative information printed on their labels. Caffeine level in *C. robusta* is always higher than that of *C. arabica* at any time durations and temperatures of extraction. Best combination between time and temperature for caffeine extraction is 15 min and 90°C or 100°C. About 90% of caffeine has been removed from coffee powder by routine percolated. The extracted caffeine is tightly correlated with extracting sequence by the equation of $y = 1.7647x^{-1.5023}$ with $R^2 = 0.9963$. The chosen coffee products have higher caffeine contents, at least 2.5 times than those appeared on their labels.

Key words: Caffeine, coffee, percolation, quality

INTRODUCTION

In 2009, Vietnam is the second most country for coffee production and export on the world after Brazil (FAO, 2010). Coffee culture is located mainly at highland in the central of the country. There are two important coffee varieties grown in Vietnam: *Coffea robusta* and *Coffea arabica* which are accounting for 90% and 10% of total production area, respectively. Human consumes coffee because it contains an actively biological compound called caffeine which is widespread non-medical used as the central nervous system stimulant or sedative (Nehlig *et al*., 1992; Croteau *et al*., 2000; Taiz and Zeiger, 2006). It acts as a blocker or an inhibitor of adenosine receptors in the central nervous system (Fredholm, 1995; Varani *et al*., 1999). Not only presence in coffee, caffeine is also presenting widely in many daily consumable products such as chocolate, tea, beverages, drugs, etc. Routine method in daily life for caffeine extraction from coffee powder is to percolate it by boiling water. Up to now, there is very few publications mentioned the method for caffeine extraction from coffee powder although hot water solvent has been applied for caffeine extract from green tea (Amra *et al*., 2006). In this study, we want to (1) investigate the effects of temperature and time on caffeine extraction from coffee powder of the most grown coffee varieties in Vietnam (2) examine how much caffeine would be released from coffee powder by traditional method of percolation with boiling water solvent (3) qualify some important qualitative criteria of popularly commercial brands of instant coffee on Vietnamese market.

MATERIALS AND METHODS

The standard caffeine was obtained from Merck (Germany) and diluted to the concentration of 30 ppm. The standard caffeine solution was scanned on HELIOSα spectrophotometer (Thermo Spectronic, England) from 200 to 400 nm to detect the wavelength of maximal absorbance. The wavelength of maximum absorbance was applied to quantify caffeine from coffee samples.

Dried coffee grains of two species, *C. arabica* and *C. robusta*, were bought from coffee retailer at Cantho city. Coffee grains were ground to powder and dried at 60°C until constant weight. About 5.0 g of dried coffee powder were transferred to a 50 mL glass beaker and 30 mL of boiling water were added. The beakers were sealed with plastic and kept in a water bath at 100°C for 5, 10, 15 and 30 min. Afterwards, the beakers were cooled down to room temperature. Extracted solution of 1 mL was pippetted to an eppendorf tube and centrifuged at 5000 rpm for 5 min. Then, 100 μL of supernatant solution were loaded on a silicagel plate with the size of 7 x 10 cm (resized from 20 x 20 cm silicagel plate, Merck, Germany). The standard caffeine of 10 μL was loaded parallelly on the same plate as the control. The thin layer silicagel plate was developed by the mixture of ethyl acetate : hexan : acetic acid with the ratio of 80 : 20 : 1 (v/v/v) for 20 min in a chromatography chamber. The plate was left to dryness at room temperature and caffeine carrying area on the silicagel plate was appeared in purple colour under UV light. This area was removed from the plate and placed in a tube containing 4 mL of distilled water. The tube was stopped, shaken

Corresponding Author: Pham Phuoc Nhan, Department of Biochemistry and Plant Physiology, College of Agriculture and Applied Biology, Cantho University, 3-2 Street, Cantho City, Vietnam

Table 1: Main quality descriptions printed on the labels of three commercial coffee products

Product name	Moisture (%)	Carbohydrate (%)	Caffeine (%)	Sachet weight (g)	Shelf life	Packing date
Milked Vinacafe	≤5	5.0	≥0.13	20	1 year	06.28.2010
Nescafé Viet	≤3	Not available	≥0.45	16	9 months	05.25.2010
Milked G7	≤5	8.56	≥0.24	16	2 years	07.13.2010

well and spinned down at 5000 rpm for 5 min. For spectral absorbance, 3 mL of solution were used.

After sufficient time for caffeine extraction was recognized from the above experiment, the effect of water temperature on caffeine extraction from coffee powder was investigated. The coffee powder of 5 g was mixed with 30 mL of water at 60, 70, 80, 90 and 100°C and maintained at respective temperatures for 15 min. The isolation and quantification of caffeine were carried out exactly the same as above descriptive procedures.

To investigate how much caffeine is liberated at the first time and the following sequences by the routine percolating method, 5 g of *C. robusta* powder was placed in an aluminum percolator and 30 mL of boiling water were poured to the percolator. The extracted solution was collected until no droplet falling. The percolator was extracted for 4 times more with the same volume of boiling water. The collected solutions were used for isolation and quantification of caffeine as described.

To assess the qualitative information printed on the labels of commercial coffee products on the market, three popular instant coffee brands have been chosen for the inspection. They were milked Vinacafé, Nescafé Viet and milked G7 which were bought randomly at a local coffee retailer. Details of important criteria on quality as listed in Table 1.

To determine caffeine level in each product, the same aforementioned procedures were applied. Moisture contents in products were evaluated by incubating at 60°C until unchanged in weight. The weight difference before and after the sample taken to dryness was accounted for humidity of the coffee product. In this experiment, instead of analysis of total carbohydrate, total soluble sugars were quantified according to the method from Dubois *et al*. (1956). All mentioned experiments were carried out with 3 replicates. For data analysis SPSS was used.

RESULTS AND DISCUSSION

The standard caffeine solution at 30 ppm had the absorbance pattern on spectrophotometer in the range from 200 to 400 nm as presented in Fig. 1. The maximum absorbance intensity was recorded at the wavelength of 288 nm and had a value of 0.495.

Time durations affected similarly on the caffeine extract from coffee powder by soaking with boiling water for both coffee species (Table 2). The highest caffeine levels were obtained at the time duration of 30 min but no significant difference with those of 15 min. Therefore,

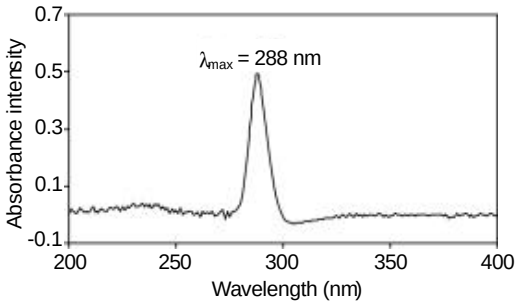

Fig. 1: Absorbance pattern of standard caffeine

Table 2: Effect of time on caffeine extraction at 100°C

Time (minutes)	Caffeine content (%)	
	C. arabica	*C. robusta*
5	1.16c	1.94c
10	1.17bc	2.00b
15	1.20ab	2.07a
30	1.21a	2.07a
P	*	**
CV (%)	0.63	0.17

** and *: significant difference at 1% and 5% level, respectively. In a column, the numbers followed by the same letter(s) are not significant difference by Duncan's multiple range test. CV: Coefficient of Variance

Table 3: Effect of water temperature on caffeine extraction at fixed time of 15 min

Temperature (°C)	Caffeine content (%)	
	C. arabica	*C. robusta*
60	1.11b	1.86c
70	1.12b	1.92b
80	1.13ab	1.95b
90	1.16a	2.04a
100	1.17a	2.08a
P	*	*
CV (%)	0.44	0.16

*: Significant difference at 5% level. In a column, the numbers followed by the same letter(s) are not significant difference by Duncan's multiple range test. CV: Coefficient of Variance

time efficiency for caffeine extraction from coffee powder by soaking with boiling water at the first time extraction was suggested at no longer than 15 min.

Not only time affected on caffeine release from coffee powder but also the temperature of water solvent using to extract it. Higher temperatures gave significantly more caffeine liberation (Table 3). The most effective temperature for caffeine extraction by water solvent was in the range from 90-100°C. However, by soaking at 60°C for 15 min, approximately 90% caffeine has been

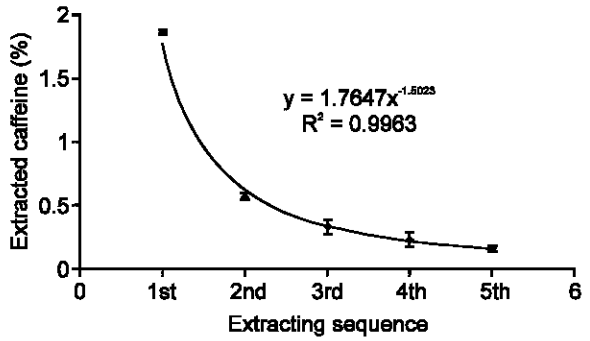

Fig. 2: The amount of caffeine release at each time of extracting sequence

Table 4: Basic components on quality of selected coffee products

Name of product	Moisture (%)	Total soluble sugars (%)	Caffeine (%)
Milked Vinacafe	4.51a	3.81a	0.84b
Nescafe Viet	2.10c	2.27c	1.37a
Milked G7	2.71b	3.38b	0.59c
$LSD_{0.01}$	0.123	0.122	0.027

In a column, the numbers followed by the same letter are not significant difference by Least Significant Difference (LSD) test at 1% level

freed from coffee powder when contrasting to that of 90°C. Therefore, hot water (not boiling water) was effective enough for extracting caffeine from coffee powder.

Caffeine removal by percolating *C. robusta* powder with boiling water at the first time was about 90% of that at 100°C and 15 min. Total caffeine contents in 5 times of percolation were more than 3.15% on dry weight. Routine percolating method in daily life at the first time removed no more than 59% of total caffeine presence in coffee powder. By soaking in water at 100°C for 15 min, caffeine release from coffee powder was nearly two thirds of total caffeine amount in coffee powder. At the forth and fifth time of extraction, there was no significant difference in caffeine liberations, 0.23% and 0.16%, respectively. The caffeine release and time of extracting sequence was tightly correlated by the equation of $y = 1.7647x^{-1.5023}$ with $R^2 = 0.9963$ (Fig. 2).

In three instant coffee products selected, the caffeine contents were always higher than those appeared on the product labels (compare the values in Table 4 with corresponding values in Table 1). The milked Vinacafé announced the lowest caffeine level to the customer among coffee brands; approximately 6.5 times lower than the value printed on its label. The actual caffeine content in milked G7 was about 2.5 times higher than that on the label. The "strongest coffee" Nescafé Viet contained a real caffeine amount only 3 times higher than the correspondent appeared on its brand. The selected products were overcome the quality examination for humidity but the carbohydrate was included unknown components besides the total soluble sugars. Two milked-instant coffee products were approved sweeter than the Nescafé Viet. There was no visible correlation between the quantified data on moisture or sugar content with the shelf life of these products.

Both time and temperature affect powerfully on the caffeine extract from coffee powder. They are also effective factors to remove caffeine from other plant material (Amra *et al*., 2006). The best combination between time and temperature to extract caffeine from coffee powder is 15 min and from 90-100°C. It has also been observed that caffeine is easily liberated from coffee powder by boiling water even at the short time duration of 5 min, mounting to at least 93% in comparison the caffeine level at 30 min of extraction. Routine method of caffeine extraction by percolation with boiling water is sufficient enough to remove caffeine from coffee powder at the first time of extraction. The efficiency of caffeine removal in the consequences of subsequent extraction is reduced clearly since caffeine potential in the material is poor in comparison with the total soluble caffeine content. Total amount caffeine in the last four times of extraction is accounted for only 40% total caffeine content in coffee powder and less than the caffeine value at the first percolation. It means the waste wetted coffee powder after the first percolation to be a highly prospective source of caffeine, about 1% on dry weight basis. The correlation in Fig. 2 is probably the perfect model to predict the amount of extracted caffeine from coffee powder at each percolation by boiling water.

At each time or temperature of extraction, the extracted caffeine amount from *C. arabica* coffee powder is always lower than the counterpart from *C. robusta*. Based on the analyzed data, *C. robusta* powder is composed of more 1.7 times in caffeine level than the *C. arabica*. Both coffee powders have relatively low but significantly different in water content 1.65% and 1.36% for *C. arabica* and *C. robusta*, respectively. The analyzed data have confirmed that instant coffee powders contain more water than the individual powder of coffee variety. In addition, the milk containing coffee brands are consisting of lower amounts of caffeine because of milk and carbohydrate component. It is also easily to understand that the investigated coffee products have lower caffeine levels than those in the powder of each coffee variety.

The results show that all producers of chosen coffee products assure their qualitative descriptions printed on the labels. If a Vietnamese consumes one sachet of Nescafé Viet, the caffeine quantity is comparable with the daily average consumption of an American of about 200 mg (Cooper *et al*., 1992). This caffeine level in a day is safety for preventing the increase of low density lipoproteins - cholesterol in the serum or coronary heart disease risk (Adebayo *et al*., 2007). In the term of

caffeine level, the two remaining products are definitely much safer to the consumers who are relatively sensitive to caffeine. However, a pregnant woman should not consume daily more than one sachet of those coffee products because of the caffeine level is higher than 71 mg; otherwise she delivers a lighter birth weight (Vlajinac *et al*., 1997). For children, these commercial instant coffees are absolutely not suitable as a result of causing unexpected behaviors if consumed daily one sachet in which caffeine amount is over 3 mg/kg of body weight if they are less than 30 kg (Hughes and Hale, 1998).

Conclusion: *C. arabica* has lower caffeine content on dry weight basis in comparing to that of *C. robusta*. Extraction of caffeine by routine percolation removes almost the potentially soluble caffeine of coffee powder. With these analyzed evidences, major commercially instant coffee brands sold on Vietnamese market are qualifying the nutritional values appeared on their labels.

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