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ANALYZING OF COFFEE QUALITY WITH DIFFERENT METHODS

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Abstract: The results obtained by the analysis of the samples of coffee mostly consumed on our market showed that the coffee quality corresponds to the values as given in the Law of Health Food.

It was not found that any of the parametres which determine food quality exceeded permitted values.

Content of heavy metals and aflatoxines is below permitted values.

The least content of caffeine was determined in a coffee sample roasted in the private roaster's shop, and only in it the presence of coffee surrogates/substitutes was not proved.

The obtained results of the coffee analysis showed that the coffees of most popular producers are very equal, and results of caffeine analysis are even more equal. This indicates the most probable fact that coffee is of the same origin, and that later, on during production, a different mixture was made, which affects the final product and gives aroma and taste to the liquid.

Key words: coffee, coffeine, heavy metals, HPLC, aflatoxin.

Introduction

During the process of roasting the raw coffee, under the exposure to the high heat (about 200°C), it comes to significant changes in the coffee content:

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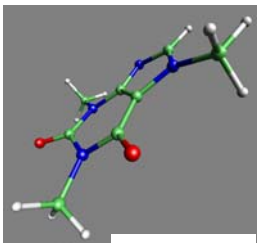
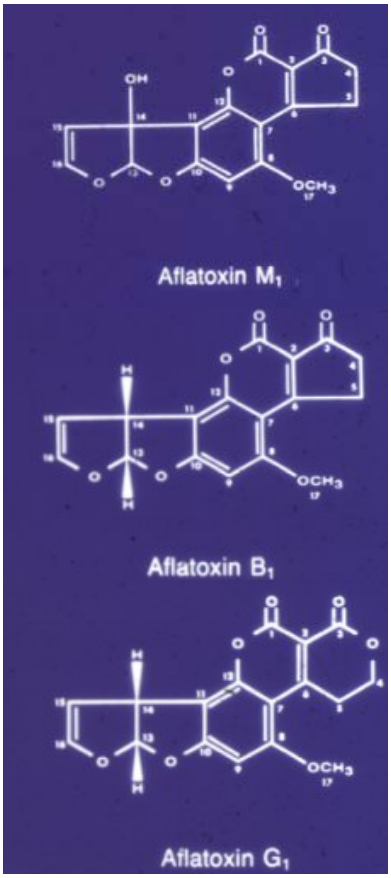
hundreds of different compounds give coffee a specific aromatic taste and scent. These compounds come from fat, carbohydrates and other compounds from raw coffee. Some compounds: pyridine, furfural, furfuril alcohol, methane acid, ethane acid, mercaptans, diethylketon, vanillin, ethanol, catechol, eugenol, phenol, diacetyl, hydroquinone, acetaldehyde, trimethylamine, ammonia etc. are present in very small amounts and in such ratios which give pleasant scent and taste to roasted coffee. (Maillard browning and Strecker's degradation reaction (Yayolayan and Lachambre, 1990; Hodge, 1967).



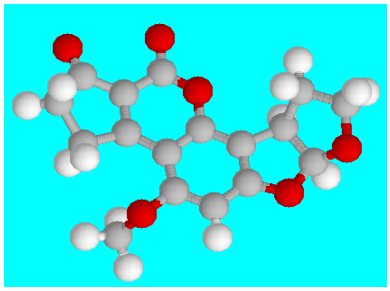
Coffeine

The average chemical composition of roasted coffee, according to U.B.Jacobs, (Trajković et al., 1983) is in wt. %: water (2.16), sugar (0.75), caffeine (1.20), raw fibres (13.03), ether extract (13.75), water extract (12.62), ash (12.62), dextrin (–, in raw coffee 0.86), tannin (–, in raw coffee 9.02) (Pekić, 1983; Wagner et al., 1974; Trajković et al., 1983).



The presence of mycotoxine – *aflatoxine* is possible in coffee. The name *aflatoxine* was given to metabolites of fungous *Aspergillus flavus* before it had been discovered that it was the mixture of related components and before their structure had been determined. From 18 known aflatoxines, aflatoxine B1 ($C_{17}H_{12}O_6$) is the most important for its presence and toxicity (LD_{50}=10-80 μg/kg). According to the result of structure determination, chemical characteristics and toxicity, aflatoxines were defined as a mixture of related chemical compounds of bisfurancumaric type. They represent metabolites of fungous *Aspergillus flavus* and *A. parasticus,* and their intake cause diseases or death in people or animals.

Aflatoxines have a wide range of biological effects. They could be: cancerous, mutogen, teratogen and embryo toxic. Aflatoxines taken with food pass through the gastric intestine system and enter the circulatory system in 30 min and into the



liver in 1 hour. A part of aflatoxines, to 60%, exude through gall, and a smaller part through stale and milk as metabolites M_1, P_1 and Q_1 or in the form of their conjugates (Krogh, 1987; Reddy et al., 1972; Purchase, 1974; Trucksess and Pohland, 2001; Kuiper-Goodman, 1991; Kuiper-Goodman et al., 1987; Rodricks and Stoloff, 1977; Diener and Davis, 1969).

Aflatoxines take their name according to the colour with which they fluorescent. Aflatoxine B fluorescent blue, and aflatoxine G green. Three structural variations of the aflatoxine molecules give a family of eight aflatoxines found in culture A. parasiticus. Aflatoxine serial with a mark B has in its molecule structure cyclopentane ring that is changed in the serial G with lactone. Index 1 determines a double connection in terminal furan ring, and in aflatoxines with index 2 such a double connection doesn't exist. Serial M contains hydroxylic group on a carbon atom on the connection of two furan rings. Combining possible combinations a serial of aflatoxines is received: B_1, B_2, G_1, G_2, M_1, M_2, GM_1, GM_2. There are authors who conclude that aflatoxine B_1 is the precursor of all other aflatoxines (Maggon and Venkitosubramanian, 1973).

In recent years on our market there is an intensive struggle between coffee sellers and producers who engage the best actors and sportsmen in spots broadcast in the prime time on all networks. The reason for this is probably in the fact that in Serbia 17,015,764 cups of "Turkish" (black) coffee are taken daily. That means that 5,300,861 citizens of Serbia, that is 87% of the population older than 15, perform their everyday ritual of drinking coffee. For that pleasure, defined as the dependence on caffeine, the market demands 102.1 tons of coffee, that is 850,788 liters of coffee per day! About four million tons of coffee beans are annually spent in the world. A Swede drink 12.5 kg of coffee annually, a Finn 8.5 kg, an American 7 kg, and there are not any valuable data about Serbia, but it could be certainly presumed that we are among the first consumers of coffee (Ignjatović et al., 2003).

That was the reason why we analysed the quality of coffees mostly advertised but also mostly consumed in our country: "Grand" coffee (according to the data of Strategic Marketing the most advertised coffee during 2002-2003. (41%), "Don" coffee (24%)). The received results are compared with those received from the analysis of coffee bought at small private roasters' shop (according to the same source, private roasters of coffee are represented with 13%).

Materials and Methods

Samples of roasted and ground coffee bought in stores were taken for the analysis of the quality of the coffees that could be found on our market:

Sample 1: coffee from a small private roaster's shop, Zemun;

Sample 2: "Don" coffee ("Minas" mixture), produced by GOLEX PRODUCT D.O.O., Belgrade;

Sample 3: "Grand" coffee, produced by GRAND PROM D.O.O., Belgrade.

In coffee samples it was done:

– organoleptic analysis by boiling and estimating of coffee without the addition of sugar;

– determination of moisture content by the method of drying in the vacuum dryer;

– determination of ash by the method of direct burning and determination of matter soluble in water (Trajković et al., 1983);

– The analysis of the presence of mycotoxine - aflatoxine in coffee was done by the chromatography method on a thin stratum of silica gel (AOAC Official Methods of Analysis, 1975). The 50 g of beforehand homogenized and milled sample was measured for the analysis, and brought into Erlenmeyer with shlif and the extraction by acetonitrol:water = 85:16 mixture was done and mixed on magnetic whisk for 30 min. After the extraction the sample was filtered through a filter paper into mencure of 100 cm^3. The extract was poured into the glass, $Fe(OH)_3$ and some infusorial earth were added and homogenization was done with magnetic whisk. The sample was put into the hopper for separation in which the departing from (with) chloroform was done. Fraction with mycotoxine was dissolved in the benzene: acetonitrol mixture and well mixed in supersonic bath. Then, on a silica gel sheet, activated for 1 hour on 105°C, racial standard solution of mycotoxine (G_1+B_1) and the extract of the sample were put. The sheet was put into chromatographic bath that contained the mobile phase acetone: chloroform: water = 12:88:1.5. After the separation was done, it was dried at room temperature and analysed under the UV lamp on 366 nm (Ostojin, 2000).

The limit of detection of this method is 2.5 μg/kg.

– The caffeine content in samples of roast and ground coffee was determined by reverse-phased liquid chromatography of high pressure (Federal Bureau for Standardization JUS ISO 10727; HP Application Note 5953-0004).

The work conditions of liquid chromatograph: HPLC Agilent 1100 apparatus, colon Symmetry Shield RP, 3.5 mm, 2.1x150 mm, the temperature of the colon: room temperature, flaw 0.3 cm^3/min, mobile phase: 30:70 = acetonitrol: water (pH value 4.50) (v/v), injected cubage: 2 μm, detector DAD *(Diode Array Detector),* in λ = 270 nm and λ_{ref} = 450 nm, duration of the analysis: 5 min, data arrangement Chem Station for LC, G2170AA.

The limit of detection 0.005%.

Heavy metals (lead and arsenic, according to regulations (Official Gazette of FRY, 1992) were determined by the process of acid digestion during determination of complete and resoluble metals by atomic absorption spectrophotometry (AAS) in flame technique and generation of the hydride technique (SW-846-*test methods for the evaluation of solid waste, method* 3005A).

To determine lead in coffee, 2-5 g of sample was measured, and then the burning of organic matter by the method of dry burning was done. Burning of the sample of ground coffee was done in a quartz or porcelain cup first on hot plate, and then in muffle furnace at 500-550°C (during 6 hours). Then 5.0 cm^3 HCl concentration of 6 mol/dm^3 was added twice and vaporized till dry, then 20.0 cm^3 HCl concentration of 0.5 mol/dm^3, well mixed with glass rod and through quantitative filter paper brought into a clean test tube.

Determination of arsenic in the coffee was done by the generation of the hydride method, method with boron hydride.

All analyses were done by *Atomic absorption spectrophotometer* Varian Spectra AA 200 (Varian Australia, Pty., Ltd., Mulgrave, Victoria, Australia).

Results and Discussion

The results of the analysis of coffee samples, compared with permitted values, according to the Law of Health Food, are shown in Table 1.

The given results showed that all the coffee samples meet the quality requirements according to the Law. It was confirmed that the first sample, from the private roaster, has the highest content of water, because it was neither packed nor vacuumed. In samples that were packed, the presence of substitute/surrogate was determined, which, on the other hand, was not found in the coffee from the private roaster.

Tab. 1. – Primary chemical composition of the coffee samples

Coffee sample	Moisture content (in wt. %)	Content of total ash (in wt. %)	Content of water-soluble substances (in wt. %)	Presence of substitute	Taste, aroma and flavour of beverage
sample 1.	4.80	4.80	24.00	non established	+ –
sample 2.	1.95	4.00	25.00	established	+
sample 3.	2.50	4.25	28.00	established	–
Permitted values	< 5.0	< 6.0	less 22%	–	*

* Roast coffee must not contain more than 2% overroasted (char) beans, it must not be mould, acetous, of odour scent or taste, and to give tasteless and odour liquid;

+ – The results of organoleptic test are satisfactory:

– The results of organoleptic test are not satisfactory.

As for organoleptic tests, not so good results come from the fact that the coffee sample was kept for some time, and not from the coffee itself.

Coffee, according to the Law of Health Food, may contain the traces of heavy metals, but in the amounts that are allowed by this regulation (Official Gazette of FRY, 1992). In the Regulation it is said that only lead and arsenic could be found in allowed concentrations, so their presence was determined in the samples, and results are shown in Table 2.

Tab. 2. - Analysis of the quantity of heavy metals in coffee (in mg/kg)

Sample	Lead	Arsenic
sample 1.	< 0.1	< 0.05
sample 2.	< 0.1	< 0.05
sample 3.	< 0.1	< 0.05
Permitted values (Official Gazette of FRY, 1992)	1	1

The analysis of the quantity of heavy metals present confirmed the accuracy of the analysed samples, considering that the amount of lead and arsenic is highly below the allowed values.

Through the analysis of caffeine a considerable peak in received chromatograph 1.426 mAU was observed. Comparing peak in this respect in the coffee samples, a good agreement with caffeine standard was observed, indicating that peak specifies caffeine and that it represents the content of the examined sample.

HPLC diagrams for the coffee samples are shown in Figure 1., and the results of quantitative determination of caffeine are shown in Table 3.

Tab. 3. - Results of the determined caffeine by HPLC method

Coffee samples	Number of measurements	Average values	Deviations		Rel. mean deviation (in %)
			Standard	Mean	
sample 1.	3	1.38	0.143	0.083	25.73
sample 2.	3	1.59	0.032	0.018	4.97
sample 3.	3	1.61	0.305	0.017	4.71

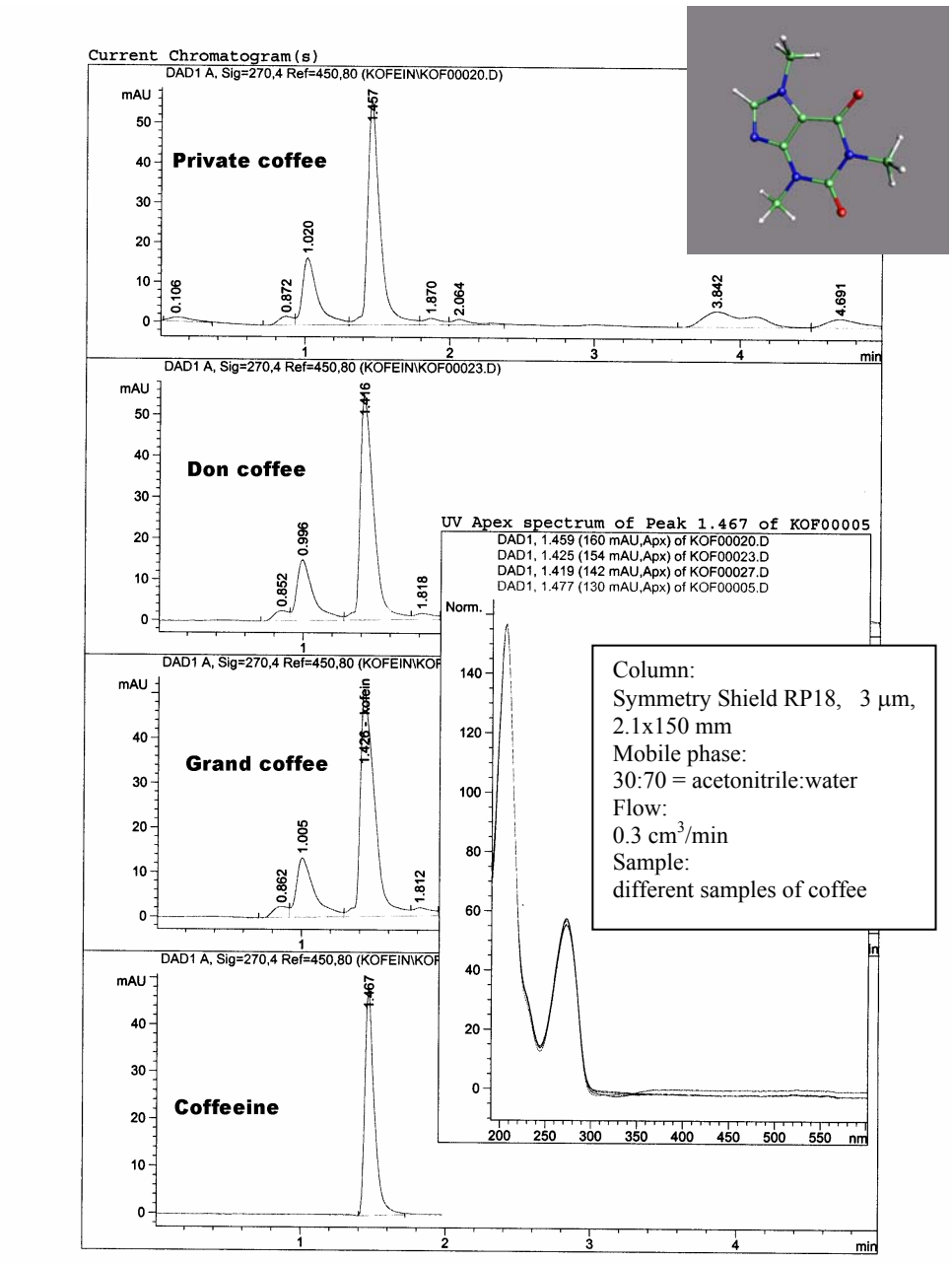


Fig. 1. - HPLC chromatograph received from the different coffee samples

Content of aflatoxines ($B+G_1$) in the analysed samples is shown in Table 4.

The analysis of received chromatographs, by observing under UV lamp on 366 nm, showed that fluorescence of the spots in the samples is less than fluorescence given by standard B_1, (2.5 μg/kg), which means that aflatoxine content in coffee samples is less than 2.5 μg/kg of aflatoxines B_1+G_1. Reference value for aflatoxine is 5.0 μg/kg, which means that the analysed samples are in accordance with article 8 of the Regulation of Health Food (Official Gazette of FRY, 1992).

Tab. 4. - Analysis of aflatoxine content ($B+G_1$) in coffee (in μg/kg)

Sample	Aflatoxine content ($B+G_1$) (in μg/kg)
sample 1.	< 2.5
sample 2.	< 2.5
sample 3.	< 2.5
Reference value	5.0
Mark of method	HE DM 0022

Conclusion

The obtained results of the analysis of coffee samples that are mostly consumed on our market showed that the coffee quality correspond to the values given in the Law of Health Food.

It was not found that any of the parametres, which determine food quality, exceeded permitted values.

Content of heavy metals and aflatoxines is below permitted values.

The least content of caffeine was determined in coffee sample roasted in the private roaster's shop, and only in it the presence of coffee surrogates/substitutes was not proved.

The obtained results of the coffee analysis showed that the coffees of most popular producers are very equal in quality, and results of caffeine analysis are even more equal. This indicates the most probable fact that coffee is of the same origin, and that laterer, on in the production process, different mixtures were made, which affects the final product and gives aroma and taste to the liquid.

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ISPITIVANJE KVALITETA KAFE RAZLIČITIM METODAMA

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R e z i m e

Dobijeni rezultati ispitivanja kvaliteta kafa koje se najčešće konzumiraju na našem tržištu pokazali su da kvalitet kafe odgovara vrednostima koje su preporučene Zakonom o zdravstvenoj ispravnosti namirnica.

Ovim ispitivanjima utvrdjeno je da nisu prekoračene dozvoljene granice nijednog od parametra koji utiču na kvalitet namirnica.

Sadržaj teških metala i aflatoksina je ispod dozvoljenih vrednosti.

Najmanji sadržaj kofeina odredjen je u uzorku kafe koja je proizvedena u privatnoj pržionici, a takodje jedino u njoj nije dokazano prisustvo surogata kafe.

Dobijeni rezultati ispitivanja kafe ukazuju da su kafe najpoznatijih proizvodjača veoma ujednačene, a rezultati ispitivanja kofeina čak i izjednačeni. To ukazuje na verovatnu činjenicu da je kafa istog porekla, a da je kasnije u pogonima pravljena različita mešavina, koja i utiče na konačni proizvod i koja daje aromu i ukus napitku.

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