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Dietary Fiber in Brewed Coffee

M. ELENA DÍAZ-RUBIO AND FULGENCIO SAURA-CALIXTO*

Departament of Metabolism and Nutrition, Instituto del Frío, Consejo Superior de Investigaciones Científicas (CSIC), Madrid, Spain

Coffee beans are rich in nondigestible polysaccharides (dietary fiber), which may partially pass into brewed coffee; however, to the authors' knowledge, there is not enough literature on dietary fiber in brewed coffee. A specific method to determine dietary fiber in beverages (enzymatic treatment plus dialysis) was applied to the coffees brewed by the most common methods (espresso, filter, soluble); results showed that brewed coffee contained a significantly higher amount of soluble dietary fiber (0.47−0.75 g/100 mL of coffee) than other common beverages. Coffee dietary fiber contains a large amount of associated antioxidant phenolics (8.7−10.5 mg/100 mL of brewed coffee).

KEYWORDS: Brewed coffee; dietary fiber; phenolics; antioxidant activity

INTRODUCTION

Coffee, an infusion of ground, roasted coffee beans, is one of the most widely consumed and appreciated beverages for its taste, its aroma, and also for its physiological effects and psychoactive properties (*1*). In fact, coffee is a complex chemical mixture reported to contain more than a thousand different compounds including carbohydrates, lipids, nitrogenous compounds, vitamins, minerals, alkaloids, and phenolic compounds (*2*), most of which may affect human health (*3*). The various compounds extracted under normal extraction conditions can be divided into nonvolatile compounds (caffeine, trigolein, chlorogenic acid, phenolics, amino acids, carbohydrates, and minerals) and volatile compounds (organic acids, aldehydes, ketones, esters, amines). Caffeine, polysaccharides, and Maillard compounds are the main groups extracted (*2*, *4*), and the amounts of these substances vary depending on coffee bean origin, roasting grade, extraction method and conditions, and cup concentration.

Experimental studies have shown positive and also negative effects of regular coffee drinking on several aspects of health (*5*, *6*). The results of epidemiological research suggest that coffee consumption may help to prevent many chronic diseases, including type 2 diabetes mellitus, arteriosclerosis, and neurodigestive diseases (*3*, *7*, *8*), and it has also been inversely related to the risk of cirrhosis (*9*); on the other hand, coffee consumption is associated with increases in several cardiovascular diseases risk factors, including high cholesterol (*6*). Polysaccharides, phenolics, caffeine, and Maillard compounds are considered to be key components in the physiological, nutritional, and organoleptic characteristics of coffee (*10*−*12*).

Hot water extractable polysaccharides are the main high molecular weight components of coffee infusions and play an important role in the viscosity of the brew, in the foam stability of espresso coffee, and hence in the retention of volatile substances (*13*).

Three types of polysaccharides—cellulose, type II arabinogalactan (AGII), and galactomannan (*4*)— predominate in coffee beans, whereas in brewed coffee only AGII and galactomannans predominate (*14*, *15*). Coffee polysaccharides have been widely studied in relation to their chemical, physiological, molecular, and organoleptic properties (*13*), but a nutritional approach may help to provide a more complete picture.

Food polysaccharides can be divided into two groups: digestible and indigestible. Digestible polysaccharides are hydrolyzed by digestive enzymes and metabolized after enzymatic hydrolysis; nondigestible polysaccharides are neither hydrolyzed nor absorbed in the small intestine, and they are the major constituents of dietary fiber (DF).

DF is the nondigestible part of vegetable foods and beverages and plays and important role in nutrition and health (*16*).

To the authors' knowledge there is no report focusing on the presence of dietary fiber in coffee beverage; indeed, food composition tables (*17*−*19*) report zero dietary fiber content for this infusion.

Also, coffee phenolics such as chlorogenic, caffeic, and ferulic acids have been studied mainly in relation to their antioxidant capacity (*20*). Several authors (*21*−*23*) have attributed the strong antioxidant properties of brewed coffee to the presence of phenolics such as chlorogenic acids and heat-induced polyphenol structure, which are formed during roasting.

Green and roasted coffee beans are rich in DF (*24*) and phenolics (*2*, *25*); also, phenolics possess an acknowledged ability to bind to other compounds such as polysaccharides, dietary fiber, or proteins (*26*−*28*) and may pass into the brewed coffee in the brewing process.

The aim of this work was to ascertain whether DF is a common constituent of brewed coffee and whether some phenolics (and their antioxidant activity) are associated with this fiber.

* Address correspondence to this author at the Department of Metabolism and Nutrition, Instituto del Frío, Consejo Superior de Investigaciones Científicas (CSIC), C/ José Antonio Novais, 10 Ciudad Universitaria, 28040 Madrid, Spain (telephone 34 915445607; fax 34 915493627; e-mail fsaura@if.csic.es).

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Table 1. Preparation of Brewed Coffees

brewed coffee	powdered coffee (g)	coffee brew[a] (mL)	solubilization[b] (%)
espresso	6.82	40	22
filtered	7.00	50	21
freeze-dried	2.00	50	100

[a] Cup of each coffee. [b] S (%) = $(C - RC)/C) \times 100$, where S = solubilitation, C = amount of coffee loaded in the coffee maker (dry matter), and RC = remaining matter in the coffee maker after the production of brewed coffee (dry matter).

MATERIALS AND METHODS

Chemicals. Pepsin was obtained from Merck (Darmstadt, Germany), and α-amylase and amylogucosidase were obtained from Sigma-Aldrich (Madrid, Spain). Gallic acid, glucose, inositol and *N*-methylimidazole were also from Merck. Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid), a water-soluble analogue of vitamin E, galacturonic acid, galactose, and also mannose were also from Sigma-Aldrich. 2,4,6-Tri(2-pyridyl)-5-triazine (TPTZ) was from Fluka Chemicals (Madrid, Spain). Dinitrosalicylic acid was from Panreac (Barcelona, Spain).

All reagents used were of analytical grade.

Samples. Two ground Colombian coffees [most widely consumed in Spain (*17*)] were selected and purchased from Cafes La Mexicana S.A. (Madrid, Spain): a 70%/30% mixture of medium-roasted and dark-roasted coffee and a freeze-dried coffee.

The most common means of extracting coffee in households, hotels, and restaurants is based on hot water or vapor. The three extraction methods normally used in Spain were tested (**Table 1**):

Espresso or Food Service Industry Coffeemaker. In this kind of coffee machine the internal temperature is in the range of 114−121 °C and the hot water must be delivered at the right pressure (8−9 bar).

Filter or Drip Electric Coffeemaker. The extraction takes 6−8 min at 90 °C, and when it is completed, the coffee in a jug is kept at 80 °C on the hot plate.

Freeze-Dried Coffee (Instant or Soluble Coffee). To produce an infusion, 2 g was solubilizated in 50 mL of hot water.

Soluble Dietary Fiber Determination. A specific method developed to determine DF in beverages was used (*29*). Briefly, infusions were treated with pepsin solution (100 mg of pepsin/mL of HCl−KCl buffer, pH 1.5); after 40 min at 40 °C in a water bath with constant shaking, α-amylase solution (40 mg of α-amylase/mL of Tris-maleate buffer, pH 6.9) was added, and the samples were incubated at 37 °C for 3 h. Finally, the infusions were treated with amyloglucosidase at pH 4.75 and incubated at 60 °C for 45 min. After the enzymatic treatments, samples were transferred into dialysis tubes (12000−14000 molecular weight cutoff; Dialysis Tubing Visking, Medicell International Ltd., London, U.K.) and dialyzed against water for 48 h at 25 °C (water flow = 7 L/h). Dialysates were then hydrolyzed with 1 M sulfuric acid at 100 °C for 90 min, and soluble DF was measured with dinitrosalicylic acid (*30*).

Neutral sugars were analyzed in the hydrolysates by gas−liquid chromatography (GLC) as alditol acetates (*31*). A Shimadzu GC-14 A chromatograph (Shimadzu Co., Kyoto, Japan) fitted with a flame ionization detector was used. An SP-2330 capillary column (30 m × 0.32 mm i.d., catalog no. 2-4073, Supelco, Bellefonte, PA) was used. Analytical conditions were as follows: column temperature, 240 °C (isothermal); injector temperature, 270 °C; detector temperature, 270 °C; carrier gas, nitrogen. Inositol was used as internal standard.

Uronic acids (constituents of SDF) were quantified in the hydrolysates by the Scott method (*32*), using galacturonic acid as standard.

Total Phenolics Determination. Total phenolics were determined in coffee beverages and dialysis retentants according to the Folin−Ciocalteu method (*33*). Test sample (0.5 mL) was mixed with 1 mL of Folin−Ciocalteu reagent and swirled. After 3 min, 10 mL of sodium carbonate solution (75 g/L) was added and mixed. Additional distilled water was mixed thoroughly by inverting the tubes several times. After 1 h, the absorbance at 750 nm was recorded. The results were expressed as gallic acid equivalents.

Antioxidant Activity Assays. Antioxidant activities of samples were estimated by ferric reducing antioxidant power (FRAP) (*34*) and $ABTS^{\bullet+}$ (free radical scavenging of the $ABTS^{\bullet+}$ radical cation) (*35*) methods.

In the FRAP assay, FRAP reagent (900 μL), freshly prepared and warmed at 37 °C, was mixed with 90 μL of distilled water and either 30 μL of test sample or standard or appropriate reagent blank. Reading at the absorption maximum (595 nm) was taken every 15 s, using a Beckman DU-640 spectrophotometer (Beckman Instruments Inc., Fullerton, CA). The readings at 4 and 30 min were selected for calculation of FRAP values. Methanolic solutions of known Trolox concentrations were used for calibration.

In the ABTS assay, ABTS radical cation ($ABTS^{\bullet+}$) was produced by reacting 7 mM ABTS stock solution with 2.45 mM potassium persulfate and allowing the mixture to stand in the dark at room temperature for 12−16 h before use. The $ABTS^{\bullet+}$ solution was diluted with methanol to an absorbance of 0.70 ± 0.02 at 658 nm. After the addition of 100 μL of sample or Trolox standard to 3.9 mL of diluted $ABTS^{\bullet+}$ solution, absorbance readings were taken every 20 s, using a Beckman DU-640 (Beckman Instruments Inc.). The reaction was monitored during 6 min. The percentage inhibition of absorbance versus time was plotted, and the area below the curve (0−6 min) was calculated. Methanolic solutions of known Trolox concentrations were used for calibration.

Statistical Analysis. Results are expressed as mean values ± standard deviation (SD). Comparison of means of three measurements, using a significance level of $p < 0.05$, was performed by one-way analysis of variance (ANOVA) using the Statgraphics Computer System, version 5.1.

RESULTS AND DISCUSSION

Soluble Dietary Fiber (SDF). SDF contents of coffee beverages (**Table 2**) showed that brewed coffee contains a significant amount of SDF (0.47−0.75 g/100 mL of coffee or 2.54−20% expressed as percent of powdered coffee bean). Instant or soluble coffee (freeze-dried coffee infusion) contains more SDF (0.752 g/100 mL of coffee) than espresso or filter coffee (0.65−0.47 g/100 mL of coffee) due to differences in the coffee brewing technique. Freeze-dried coffee is produced by means of a system that requires high temperatures (200 °C) to extract soluble compounds from roasted coffee; the infusion was made with 2 g of this freeze-dried extract, which was totally solubilized. The DF content in brewed coffee is higher than in other common beverages such as wine (0.14%) or orange juice (0.19%).

The method of dietary fiber determination used by the authors is based, with some modifications, on a procedure used to determine DF in wine (*29*) and beer (*36*), and there were three key steps: enzymatic treatments, dialysis to isolate soluble fiber, and analysis in dialysis retentant. Treatment with pepsin, α-amylase, and amyloglucosidase was necessary to hydrolyze protein, peptides, and digestible polysaccharides that may be present in beverages. SDF was isolated by dialysis, which eliminates all of the enzymatic hydrolysis products along with other coffee constituents able to pass through the dialysis membrane (molecular weight cutoff = 12000−14000 Da). Quantitative and qualitative analysis of fibers in the solution retained in the dialysis tubes was performed by spectrophotometric and gas−liquid chromatographic methods.

If beverages contain appreciable amounts of oligosaccharides, a specific method to determine these compounds would be necessary, because they would be dialyzed in our procedure; however, in the case of coffee brew, the content of oligosaccharides is negligible (*37*) and nutritionally insignificant, and therefore a specific determination is not necessary.

The AOAC enzymatic−gravimetric procedure is the one most commonly used for food labeling (*38*). This is based on the

Table 2. Content and Composition of Soluble Dietary Fiber in Brewed Coffee

	espresso	filtered	freeze-dried
SDF[a] (g/100 mL of coffee)	0.65 ± 0.03	0.47 ± 0.09	0.75 ± 0.04
SDF[a] (% powdered coffee)	3.08 ± 0.02	2.54 ± 0.07	20.20 ± 0.90
SDF composition			
uronic acids (g/100 mL of coffee)	0.012 ± 0.004	0.0097 ± 0.0003	0.01825 ± 0.0004
neutral sugars[b] (mol %)			
glucose	3.96 ± 0.04	3.88 ± 0.03	3.23 ± 0.09
galactose	24.1 ± 0.2	24.8 ± 1.3	56.8 ± 5.9
mannose	60.9 ± 0.9	60.1 ± 2.9	33.2 ± 2.9
xylose	nd[c]	nd	0.62 ± 0.04
arabinose	10.93 ± 0.2	11.1 ± 0.6	5.9 ± 0.5
rhamnose	0.17 ± 0.02	0.227 ± 0.006	0.08 ± 0.01

[a] SDF, soluble dietary fiber. Each value is the mean ± SD ($n = 9$). [b] Expressed as mole percentage in neutral sugars. [c] Not detected.

Table 3. Antioxidant Activity and Phenolics in Brewed Coffee and Dialysis Retentants[a]

		method	espresso	filtered	freeze- dried
brewed coffee	total phenolics GAE[b] (mg/100 mL coffee)		198.82 ± 4.56	218.28 ± 4.46	361.32 ± 4.19
	antioxidant activity (μmol of Trolox/100 mL of coffee)	FRAP	1945.5 ± 85	1565.2 ± 45	2593.0 ± 69
		ABTS	833.4 ± 43	1267.4 ± 48	1295.2 ± 16
diaysis retentant[b,c]	associated phenolics GAE (mg/100 mL of coffee)		103.73 ± 1.12	87.56 ± 1.05	105.42 ± 2.17
	antioxidant activity (μmol of Trolox/100 mL of coffee)	FRAP	564.9 ± 13	456.3 ± 68	610.7 ± 11
		ABTS	493.2 ± 14	313.7 ± 2.2	386.9 ± 2.2

[a] Each value is the mean ± SD ($n = 9$). [b] Expressed as gallic acid equivalents (GAE). [c] Dialysis retentant of brewed coffee after enzymatic treatment and dialysis.

removal of macronutrients by enzymatic treatment, and separation of soluble DF is achieved by precipitation in 80% ethanol−water. The AOAC method, currently used for labeling of solid foods, is not directly applicable to beverages because it entails major loss of fiber that is partially soluble in the medium used (*39*). In the case of samples rich in oligosaccharides a specific method is also needed, because these compounds escape analysis in the AOAC procedure.

The average DF intake in Europe ranges from 16 to 21 g/person/day; SDF in the Spanish diet is about 7 g/person/day (*17*, *40*, *41*). The contribution of brewed coffee to the dietary fiber intake in a common diet may be significant; a moderate daily consumption of three cups of espresso coffee is equivalent to 0.66 g of SDF, which accounts for about 10% of SDF intake in Spain.

The main nondigestible polysaccharides in coffee are arabinogalactan type II (AGII), galactomannan (GM), and cellulose. Cellulose is a major constituent of insoluble dietary fiber (IDF) but is found in only minor amounts in SDF; it was therefore to be expected that GM and AGII would be the main nondigestible polysaccharides in SDF from coffee.

Table 2 also shows monosaccharides and uronic acids in SDF in brewed coffee. Galactose (156−422 mg/100 mL) and mannose (395−246 mg/100 mL) were the major constituents in all infusions, which suggests GM is the main component of SDF. In fact, GM, which comprise nearly 70% of a roasted coffee infusion, are composed of a backbone of 1,4-linked mannans with a single unit of galactose side chains at C_6; AGII consists of a main chain of 1,3-linked galactose branched at C_6 with side chains containing arabinose and galactose residues (*14*, *15*). Small amounts of arabinose were found, suggesting that arabinogalactan was extracted from coffee during brewing, although the lone content of arabinose cannot be used to infer information about the arabinogalactans; in fact, because during roasting the arabinose side chains are preferentially cleaved from arabinogalactnas, it is not a surprise to find low levels of arabinose. Galactose came also from arabinogalactans, as the galactomannans present in roasted coffee infusions have a low percentage of galactose side chains (*40*).

The small amounts of uronic acids (0.012−0.009 g/100 mL) indicate that the pectic backbone is not a predominant structural element in coffee, although Redgwell and Fischer (*42*) have found that uronic acids can came also from the arabinogalactans in green and roasted coffee.

Total Phenolics and Antioxidant Activity. Phenolic compounds are ubiquitous constituents of higher plants found in a wide range of commonly consumed plant foods such as fruits, vegetables, cereals, and legumes and in beverages of plant origin such as wine, tea, and coffee. The presence of phenolics in DF of different vegetable materials has been reported elsewhere (*26*, *27*). In fact, a significant amount of total phenolics (140−184 mg/g of SDF) was retained in dialysis retentant associated with coffee SDF (**Table 3**), accounting for about 30−51% of total phenolics in coffee beverages.

The coffee polyphenols reported in the literature are single molecules (chlorogenic acid, ferulic acid) or oligomers (with molecular weights lower than the cutoff (12000 Da) of the dialysis membrane used in our experiment; they would therefore be dialyzed. Indigestible polysaccharides, dietary fiber, or Maillard compounds and protein are the main macromolecules that can link polyphenols (*26*−*28*). The soluble fraction of coffee beans contains melanoidins (Maillard compounds), but their molecular weights are <10000 Da. We may therefore expect that only DF polysaccharides will be undegraded by these enzymes, whereas associated polyphenols may remain in the dialysis retentant.

We found that all infusions contained significant amounts of associated phenolics, the concentrations in each case depending on the brewing technique and the type of associated phenolic (87−105 mg/100 mL of coffee) (**Table 3**).

Coffee polyphenols are potent antioxidants (*25*) and endow brewed coffee with considerable antioxidant activity (*20*). Our results showed that a significant part of the antioxidant activity is associated with DF (**Table 3**); this suggests that a fraction of

antioxidant polyphenols from coffee is bioaccessible in the small intestine, whereas the part associated with DF (30−51%) will be bioaccessible in only the large intestine after fermentation by colonic microflora.

In summary, dietary fiber is a quantitatively important constituent of brewed coffee and contains appreciable amounts of antioxidant phenolics. Further research on the biological properties of this fiber is needed.

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