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Polyphenols and energy recovery from spent coffee grounds

Antonio Zuorro, Roberto Lavecchia*

Dipartimento di Ingegneria Chimica, Materiali e Ambiente – Università "La Sapienza"
Via Eudossiana 18, 00184 Roma (Italy)
roberto.lavecchia@uniroma1.it

Spent coffee grounds were investigated as a potential source of high-value phenolics and energy. Phenolic extraction was performed by the use of 60% (v/v) ethanol in water as the solvent. Under the best conditions (temperature = 60 °C; extraction time = 180 min; liquid-to-solid ratio = 50 mL/g) over 1500 mg of phenolics per 100 g of dry coffee grounds were obtained. Calorimetric measurements showed that coffee grounds had a high calorific value (about 24 MJ/kg) and that this property did not change on extraction of phenolic compounds. Overall, these results suggest that phenolic-rich extracts can be obtained from spent coffee grounds using a low-cost, environmentally benign and non-toxic extraction procedure and that the resulting solid residue can be further exploited for energy production.

1. Introduction

Spent coffee grounds, i.e. the solid residue remaining after the production of the coffee beverage, are generated in large amounts worldwide, as attested by the over 100 million bags of coffee produced in the world each year (ICO, 2009). *Coffea arabica* and *Coffea canephora* var. *robusta* are the two main species of commercial interest (Thurber, 2010). They are usually referred to as Arabica and Robusta, respectively, with the former accounting for about 70–75% of production. Coffee grounds have no commercial value and are currently disposed of as conventional solid waste or, to a limited extent, sent to compost facilities. However, the presence in this material of high levels of phenolic compounds with significant antioxidant activity (Yen et al., 2005; Ramalakshmi et al., 2009) suggests the possibility of using the waste as an inexpensive source of natural antioxidants. Furthermore, considering that the extraction of phenolics from coffee grounds would generate a solid residue to be disposed of, it would be highly desirable to combine the recovery of phenolics with some form of exploitation of the inert material. Such an approach would offer both environmental and economic advantages, the saving of landfill space being accompanied by the obtainment of value-added products (Laufenberg et al., 2003; Arvanitoyannis and Varzakas, 2008).
In view of the above considerations and as a continuation of our studies in this field (Lavecchia and Zuorro, 2008a, 2008b; Zuorro and Lavecchia, 2010), we conducted an exploratory research study with the aim of: (a) assessing the feasibility of obtaining a phenolic-rich extract from spent coffee grounds by an environmentally friendly procedure, and (b) determining the calorific value of the waste material before and after

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extraction in view of its energetic exploitation. With regard to the first point, we developed a procedure based on the use of ethanol–water mixtures as the extraction solvent. A factor influence study was also performed in order to estimate the contribution of the main process variables to the phenolic extraction yield.

2. Experimental

2.1 Chemicals

Gallic acid (3,4,5-trihydroxybenzoic acid) and the Folin-Ciocalteu's phenol reagent were obtained from Sigma-Aldrich Co. (St. Louis, Mo, USA). Ethanol, hydrochloric acid and sodium carbonate were purchased from Carlo Erba (Milano, Italy). All chemicals were analytical grade and used without further purification.

2.2 Spent coffee grounds

Coffee beverages were first prepared by an automatic espresso machine utilizing pre-loaded coffee capsules. Spent coffee grounds were then recovered from the capsules, partially dried in air for a few hours and stored in plastic bags at 4 °C until use.

2.3 Determination of total phenolics

The amount of total phenolics in the extracts was determined by the Folin-Ciocalteu method according to the procedure described by Singleton et al. (1999). Measurements were made at 525 nm by a benchtop colorimeter (Hanna Instruments, Italy).

2.4 Measurement of calorific value

The calorific value was determined by an automatic adiabatic bomb calorimeter (C5000, IKA) controlled by C5040 CalWin software. Measurements were made at least in duplicate using an amount of solid of 0.5-1 g.

2.5 Phenolic extraction procedure

The extraction experiments were carried out in batch mode. Appropriate amounts of coffee grounds and the ethanol–water mixture were loaded into 50 or 100 mL screw-top pyrex flasks. The flasks were placed in a water bath thermostated at ± 0.1 °C and magnetically stirred. At the desired time, a sample of the liquid was taken, passed through a 45-µm nylon filter and assayed for phenolic content. A first series of experiments was undertaken to determine the optimal composition of the extraction mixture. The mixture giving the highest yield was then used to evaluate the influence of the main process variables on the extraction of phenolics. To this end, a full-factorial design was used including three factors and two levels (Table 1). Four center-point replicates were also considered, for a total of $2^3 + 4 = 12$ runs. The overall experimental design layout is reported in Table 2, where the "run" column shows the formal order of runs in the design and the "trial" column indicates the randomized order in which the experiments were carried out.

3. Results and Discussion

A preliminary evaluation of the total amount of phenolics (c_{tot}) in spent coffee grounds was performed using different waste samples. They were subjected to a three-stage

extraction procedure at T = 80 °C, t = 30 min, m_S = 2 g, V_L = 100 mL. Total phenolics were expressed as gallic acid equivalents (GAE) We obtained: c_{tot} = 1725 ± 16 mg GAE/100 g.

Experiments aimed at determining the optimal solvent composition (T = 25 °C, t = 60 min, L/S = 15 mL/g) gave the results shown in Figure 1.

Table 1: Factors, codes and levels for the full-factorial design

Factor	Code	Level		
		–1	0	1
Temperature (°C)	X1	20	40	60
Extraction time (min)	X2	30	105	180
Liquid-to-solid ratio (mL/g)	X3	10	30	50

Table 2: Experimental design layout

Run	Trial	X1	X2	X3
1	5	–1	–1	–1
2	10	+1	–1	–1
3	6	–1	+1	–1
4	2	+1	+1	–1
5	11	–1	–1	+1
6	3	+1	–1	+1
7	8	–1	+1	+1
8	12	+1	+1	+1
9	1	0	0	0
10	9	0	0	0
11	4	0	0	0
12	7	0	0	0

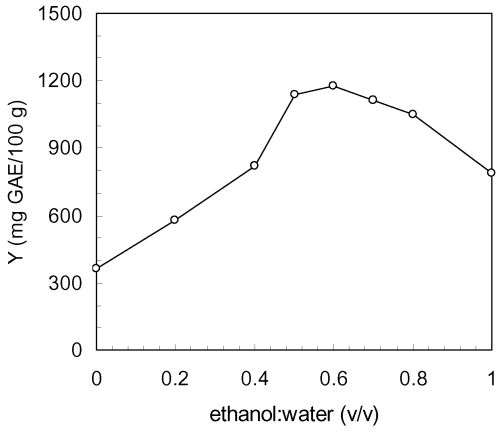

Figure 1: Effect of solvent composition on phenolic extraction yield (Y)

As can be seen, the extraction yield exhibited a maximum in aqueous mixtures containing 60% (v/v) ethanol. The position of the maximum was found to be essentially independent on the liquid-to-solid ratio and on temperature. Accordingly, this mixture was used for all the subsequent experiments.

The results concerning the influence of the experimental conditions (temperature, extraction time and liquid-to-solid ratio) on phenolic extraction are summarized in Table 3. The levels associated to each factor were chosen to cover a range of values of practical interest. So, temperatures below 20 °C or above 60 °C were not considered for kinetic reasons or for the need to avoid phenolic degradation, respectively.

Inspection of the results reveals that the maximal extraction yield (1540 mg GAE/100 g, corresponding to about 90% of the total amount of phenolics in the waste) was achieved at 60 °C, 180 min and a liquid-to-solid ratio of 50 mL/g, i.e., at the upper extreme of the range of values considered. However, a reduction of the extraction time, from 180 to 30 min, at this temperature resulted in a nearly identical yield (1475 against 1540 mg GAE/100 g), suggesting that phenolic extraction occurs mainly at the first stage of the process. Similar results were observed at 20 °C, where the extraction yields at 30 and 180 min were 796 and 830 mg GAE/100 g, respectively.

To quantitatively estimate the contribution of the three factors to the extraction yield and their binary and ternary interactions we used the following equation (Lewis et al., 1999):

$$Y = a_0 + a_1X_1 + a_2X_2 + a_3X_3 + a_{12}X_1X_2 + a_{13}X_1X_3 + a_{23}X_2X_3 + a_{123}X_1X_2X_3 \qquad (1)$$

where a_1, a_2 and a_3 are the coefficients associated with the main effects; a_{12}, a_{13} and a_{23} are those related to binary interactions and a_{123} is the ternary interaction coefficient. These coefficients were determined from the data of runs 1-8, obtaining the values presented in Table 4. The standard deviation of the experimental response was calculated from the four replicated center points, yielding: σ_y = 19 mg GAE/100 g. This value was then used to perform the Student's *t* test (Lewis et al., 1999), which provided the following 95% confidence interval: [–21.4; +21.4].

Table 3: Phenolic extraction yields (Y) under the conditions of the factorial design

Run	T (°C)	t (min)	L/S (mL/g)	Y (mg GAE/100 g)
1	20	30	10	796 ± 10
2	60	30	10	1072 ± 11
3	20	180	10	830 ± 10
4	60	180	10	1102 ± 12
5	20	30	50	992 ± 29
6	60	30	50	1475 ± 21
7	20	180	50	1117 ± 20
8	60	180	50	1540 ± 22
9	40	105	30	1225 ± 38
10	40	105	30	1234 ± 14
11	40	105	30	1245 ± 26
12	40	105	30	1269 ± 13

Table 4: Estimated coefficients for the model described by eq. (1)

Coefficient	Effect	Value
a_0	–	1115.5
a_1	Temperature	181.8
a_2	Extraction time	31.8
a_3	L/S	165.5
a_{12}	Temperature–Extraction time	–8.0
a_{13}	Temperature–L/S	44.8
a_{23}	Extraction time–L/S	15.8
a_{123}	Temperature–Extraction time–L/S	–7.0

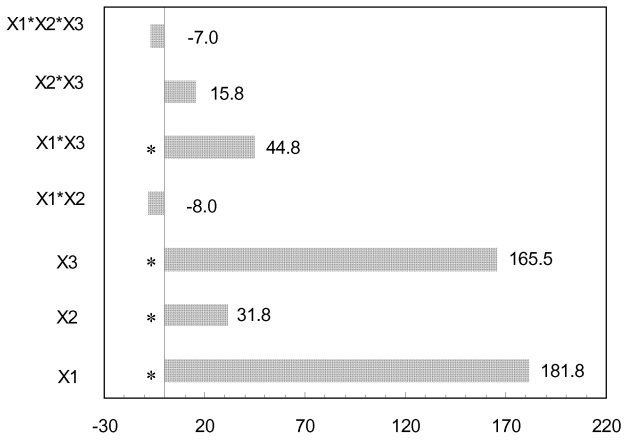

Figure 2: Pareto chart for parameter effects on phenolic extraction. Significant effects ($p < 0.05$) are marked with an asterisk

Examination of Table 4 and the Pareto chart shown in Figure 2 reveals that only four coefficients, a_1, a_2, a_3 and a_{13}, can be considered significant ($p < 0.05$). Their positive sign indicates that all of the three main factors have a positive effect on phenolic extraction, with temperature and liquid-to-solid ratio exerting a stronger influence. In addition, there appears to be a significant positive interaction between the latter variables, suggesting that an increase in temperature has a more pronounced effect on extraction at higher liquid-to-solid ratios.

Finally, measurement of the calorific value of spent coffee grounds before (Q) and after (Q') phenolic extraction provided the following results (expressed per dry weight of solid): Q = 24.3 ± 0.8 MJ/kg and Q' = 23.4 ± 0.5 MJ/kg. These results suggest the following considerations: (a) the calorific value of spent coffee grounds is only marginally affected by the extraction of phenolic compounds (the observed percent reduction being approximately 4%) and (b) the energy released by their combustion is very large. In particular, it appears to be higher than those of sawdust and most biomass

wastes and residues, which are roughly in the range of 15-21 MJ/kg (Demirbas, 1997; Poskart and Szecówka, 2008).

4. Conclusions

The results from the present study strongly support the possibility of obtaining phenolic-rich extracts from spent coffee grounds by an environmentally friendly and cost-effective method. Due to its high calorific value, the extraction residue could further be exploited for energy pruduction purposes. Such an approach would allow a considerable reduction in the amount of waste to be disposed of and the obtainment of a new product of potential interest to the cosmetic, pharmaceutical and food industries.

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