Valorisation of spent coffee grounds using hot compressed water

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Introduction

Coffee is the second most traded commodity in the world being only second to oil.

The coffee industry generates a large amount of residues. Spent coffee grounds (SCG) is the by-product obtained in the preparation of instant coffee, when coffee is treated with hot water for the extraction of flavor substances. Every year 6 million tons of this residue are generated worldwide and most of it is still discarded as waste, or burned. There is potential for the valorisation of this residue, given that SCG is a rich source of phenolic compounds, bioactive compounds with interest for food, pharmaceutical and cosmetic industries (Machado et al. 2012). SCG is also rich in carbohydrates, which are present in a complex lignocellulosic matrix. The most abundant polysaccharide present in SCG is hemicellulose, which is an heterogeneous polymer of xylose, arabinose, mannose, galactose and glucose (Mussatto et al. 2011). Several methods are reported for the extraction of phenolic compounds from SCG and hydrolysis of lignocellulose, mostly using organic solvents and acidic solutions.

Hot compressed water (HCW) is an environmental friendly alternative to these conventional solvents. HCW is liquid water at high temperatures, above its vapor pressure. Compared to water at ambient conditions, HCW has lower viscosity and surface tension, leading to increased mass transfer rates, a lower dielectric constant, which increases the solubility of less polar molecules, such as many phenolics, and also a higher ionic product, which makes HCW a more reactive medium for the hydrolysis of lignocellulosic matrices (Brunner 2009).

Materials and Methods

Before the treatment of SCG with HCW, several conventional methods were used for the chemical characterization of this residue. Before analysis, SCG was dried in an oven at 80°C for 3 days, to obtain a dry weight basis. The nitrogen content was determined by elementary analysis and to determine protein content, a nitrogen-to-protein conversion factor of 6.25 was used. Ash content was determined through mass difference, before and after the samples were placed in a muffle at 550 °C. The determination of lipid content was achieved through Soxhlet extraction with hexane and weighing the extracted oil. The carbohydrate content was determined by a two-step acid hydrolysis of the remaining residue from the Soxhlet extraction, and Klason lignin was determined by subtracting from the remaining hydrolysis residue the resistant protein and acid insoluble ash. The original dry residue was treated with a 60% v/v methanol:water solution at 60 °C for the identification and quantification of phenolic compounds.

For the extraction/hydrolysis with HCW, a semi-continuous apparatus was used, comprising an approximately 270 mL reactor loaded with 60 g of SCG. To evaluate the influence of temperature on the extraction/hydrolysis process, several assays were performed until maximum temperatures of 150, 180, 200 and 220 °C, while pressure was kept constant at around 100 bar. Samples were collected during the heating stage and after reaching the maximum temperature. For the analysis of total phenolic compounds, the Folin-Ciocalteu method was used, while for reducing sugars the phenol-sulfuric method was applied. HPLC was used for the identification and quantification of individual compounds.

The antioxidant activity of the extracts obtained was determined by the DPPH (1,1-diphenyl-2picrylhydrazyl) method.

Results and Discussion

Before the experiments with HCW, the chemical characterization of SCG was performed. SCG has a content of 50% in polysaccharides, mostly hemicellulose (42%). The other components present are proteins (13.4%), lipids (12.3%), lignin (12.8%) and ash (1%). These results agree with those of other authors (Mussatto et al. 2011). Extraction of SCG with the methanol:water solution yielded 22 mg/g of phenolic compounds.

Several assays were performed with HCW at different temperatures (150, 180, 200, 220 °C). The increase in temperature led to an increase in the yield of phenolic compounds and carbohydrates recovered, up to 200 °C. The results obtained at 200 and 220 °C were very similar, leading to around 32% of carbohydrates recovered from SCG and 36 mg of phenolic compounds per g of SCG. These results represent a recovery of 65%

of the total amount of carbohydrates present in SCG, and a higher recovery of phenolic compounds when compared with the conventional extraction method used.

It was observed that the extracts collected at lower temperatures, until 150 °C, had a higher content in phenolic compounds, around 150 mg/g of dry extract, while the samples collected at higher temperatures had a lower content of phenolic compounds but were richer in carbohydrates. This is in agreement with water becoming more reactive at higher temperatures, thereby increasing the hydrolysis of the lignocellulosic matrix.

These results were also confirmed by the determination of the antioxidant activity of the extracts, which was higher for the extracts richer in phenolic compounds, with a half maximum effective concentration (EC_{50}) of 25 mg_{dry extract}/L, which compares with an EC_{50} 83 mg_{dry extract}/L for the extracts with a lower content in phenolic compounds.

Conclusions

SCG is an important source of phenolic compounds and carbohydrates, and HCW is an environmental friendly alternative for the recovery of both types of compounds. A phenolic-rich extract was obtained at 150 °C, while for an efficient hydrolysis of the polysaccharides higher temperatures are needed. The antioxidant activity of phenolic-rich extracts was evaluated, yielding an EC_{50} of 25 mg/L. The sugar-rich extracts can be used as an alternative carbon source for the growth of microorganisms.

References

Brunner, G. 2009. "Near Critical and Supercritical Water. Part I. Hydrolytic and Hydrothermal Processes." *Journal of Supercritical Fluids* 47 (3): 373–81. doi:10.1016/j.supflu.2008.09.002.

- Machado, Ercília M S, Rosa M. Rodriguez-Jasso, José A. Teixeira, and Solange I. Mussatto. 2012. "Growth of Fungal Strains on Coffee Industry Residues with Removal of Polyphenolic Compounds." *Biochemical Engineering Journal* 60. Elsevier B.V.: 87–90. doi:10.1016/j.bej.2011.10.007.
- Mussatto, Solange I., Livia M. Carneiro, João P A Silva, Inês C. Roberto, and José A. Teixeira. 2011. "A Study on Chemical Constituents and Sugars Extraction from Spent Coffee Grounds." *Carbohydrate Polymers* 83 (2). Elsevier Ltd.: 368–74. doi:10.1016/j.carbpol.2010.07.063.