



A study on chemical constituents and sugars extraction from spent coffee grounds

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ARTICLE INFO

Article history:

Received 12 July 2010

Received in revised form 26 July 2010

Accepted 27 July 2010

Available online 6 August 2010

Keywords:

Spent coffee grounds

Chemical composition

Hemicellulose

Dilute acid hydrolysis

Experimental design

ABSTRACT

Spent coffee grounds (SCG), the residual materials obtained during the processing of raw coffee powder to prepare instant coffee, are the main coffee industry residues. In the present work, this material was chemically characterized and subsequently submitted to a dilute acid hydrolysis aiming to recover the hemicellulose sugars. Reactions were performed according to experimental designs to verify the effects of the variables H_2SO_4 concentration, liquid-to-solid ratio, temperature, and reaction time, on the efficiency of hydrolysis. SCG was found to be rich in sugars (45.3%, w/w), among of which hemicellulose (constituted by mannose, galactose, and arabinose) and cellulose (glucose homopolymer) correspond to 36.7% (w/w) and 8.6% (w/w), respectively. Optimal conditions for hemicellulose sugars extraction consisted in using 100 mg acid/g dry matter, 10 g liquid/g solid, at 163 °C for 45 min. Under these conditions, hydrolysis efficiencies of 100%, 77.4%, and 89.5% may be achieved for galactan, mannan, and arabinan, respectively, corresponding to a hemicellulose hydrolysis efficiency of 87.4%.

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1. Introduction

Coffee is one of the world's most widely consumed beverages, and spent coffee grounds (SCG), the solid residues obtained from the treatment of coffee powder with hot water to prepare instant coffee, are the main coffee industry residues with a world-wide annual generation of 6 million tons (Tokimoto, Kawasaki, Nakamura, Akutagawa, & Tanada, 2005). Considering this huge amount of coffee residue produced all over the world, the reutilization of this material is a relevant subject. Some attempts for reutilization of SCG have been made, using it as fuel in industrial boilers of the same industry due to its high calorific power of about 5000 kcal/kg (Silva, Nebra, Silva, & Sanchez, 1998), as an antioxidant material source (Yen, Wang, Chang, & Duh, 2005), or as a source of polysaccharide with immunostimulatory activity (Simões et al., 2009). Kondamudi, Mohapatra, and Misra (2008) demonstrated that SCG can be used for the production of biodiesel and fuel pellets. SCG was also considered an inexpensive and easily available adsorbent for the removal of cationic dyes in wastewater treatments (Franca, Oliveira, & Ferreira, 2009). However, none of these strategies have yet been routinely implemented, and most of these residues remain unutilized, being discharged to the environment where they cause severe contamination and environmental pollu-

tion problems due to the toxic nature (presence of caffeine, tannins, and polyphenols) (Leifa, Pandey, & Soccol, 2000). Nowadays, there is great political and social pressure to reduce the pollution arising from industrial activities. In this sense, conversion of SCG to value-added compounds is of environmental and economical interest.

Hemicelluloses, the second most common polysaccharides in nature, are heterogeneous polymers of pentoses (xylose and arabinose), hexoses (mannose, galactose, glucose), and sugar acids. In recent years, bioconversion of hemicellulose has received much attention because of its practical applications in various industrial processes, such as for the production of fuels and chemicals (Saha, 2003). Hemicelluloses are usually found in the nature in association with other polymeric fractions, namely the cellulose and lignin. To be efficiently used in bioconversion processes, the hemicellulose polysaccharide needs to be separated from these other structures. Different processes may be used for this purpose, among of which, dilute acid hydrolysis stands out as one of the most efficient to selectively release hemicellulose sugars (Mussatto & Roberto, 2004). The major problem of acid hydrolysis is that the decomposition of monomeric sugars produced during the reaction takes place simultaneously with the hydrolysis of polysaccharides. To prevent sugars decomposition, it is very important to conduct the process under adequate reaction conditions. The experimental design statistical methodology is a useful tool to define such conditions performing a minimal number of experiments. This methodology has been employed in several works to maximize the sugars recovery from agro-industrial residues through the establishment of the

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Table 1

2⁴ experimental design with the real and coded values of the variables used for dilute acid hydrolysis of SCG hemicellulose, and responses obtained to each experimental condition.

Assay	Variables				Responses								
	Real values (coded values) ^a				Sugars (g/l) ^b					Efficiencies (%) ^c			
	X ₁	X ₂	X ₃	X ₄	Cel	Glu	Arab	Gal	Man	η _{Gal}	η _{Man}	η _{Arab}	η _{Hemi}
1	10 (−1)	100 (−1)	15 (−1)	100 (−1)	0	0	0.08	0	0	0	0	4.8	0.2
2	14 (+1)	100 (−1)	15 (−1)	100 (−1)	0.03	0	0.06	0	0	0	0	4.8	0.2
3	10 (−1)	140 (+1)	15 (−1)	100 (−1)	0	0	0.13	0	0	0	0	7.8	0.4
4	14 (+1)	140 (+1)	15 (−1)	100 (−1)	0.05	0	0.05	0	0	0	0	4.2	0.2
5	10 (−1)	100 (−1)	45 (+1)	100 (−1)	0.01	0.06	1.32	0.15	0.11	1.2	0.6	79.8	4.6
6	14 (+1)	100 (−1)	45 (+1)	100 (−1)	0	0	1.03	0.07	0.34	0.8	2.4	87.4	5.9
7	10 (−1)	140 (+1)	45 (+1)	100 (−1)	0.05	0.11	1.85	0.27	0.13	2.1	0.7	100.0	6.0
8	14 (+1)	140 (+1)	45 (+1)	100 (−1)	0	0	1.04	0.07	0.09	0.8	0.6	88.2	4.9
9	10 (−1)	100 (−1)	15 (−1)	140 (+1)	0.14	0.27	1.96	0.68	0.30	5.3	1.5	100.0	7.7
10	14 (+1)	100 (−1)	15 (−1)	140 (+1)	0	0	0.87	0.11	0.11	1.2	0.8	73.8	4.5
11	10 (−1)	140 (+1)	15 (−1)	140 (+1)	0.10	0.09	1.91	0.51	0.24	4.0	1.2	100.0	7.0
12	14 (+1)	140 (+1)	15 (−1)	140 (+1)	0.02	0	1.11	0.18	0.13	2.0	0.9	94.2	5.8
13	10 (−1)	100 (−1)	45 (+1)	140 (+1)	0	0.28	2.12	14.26	11.24	100.0	56.6	100.0	74.9
14	14 (+1)	100 (−1)	45 (+1)	140 (+1)	0.21	1.08	1.79	5.12	4.68	55.8	33.1	100.0	44.8
15	10 (−1)	140 (+1)	45 (+1)	140 (+1)	0	0.25	2.54	13.64	14.31	100.0	72.1	100.0	83.9
16	14 (+1)	140 (+1)	45 (+1)	140 (+1)	0.11	0.94	2.06	9.45	4.74	100.0	33.5	100.0	61.6
17	12 (0)	120 (0)	30 (0)	120 (0)	0.10	0.13	1.79	0.59	0.21	5.5	1.3	100.0	7.6
18	12 (0)	120 (0)	30 (0)	120 (0)	0.09	0.09	1.67	0.43	0.19	4.0	1.2	100.0	7.0
19	12 (0)	120 (0)	30 (0)	120 (0)	0.08	0.08	1.64	0.43	0.18	4.0	1.1	100.0	6.9

^a X₁: liquid-to-solid ratio (g/g); X₂: acid concentration (mg/g); X₃: reaction time (min); X₄: temperature (°C).

^b Cel: cellobiose; Glu: glucose; Arab: arabinose; Gal: galactose; Man: mannose.

^c η: efficiency of hydrolysis; Hemi: hemicellulose.

best hydrolysis operational conditions (Mussatto & Roberto, 2005; Neureiter et al., 2004; Roberto, Mussatto, & Rodrigues, 2003).

In view of the aforementioned, the present work evaluated the sugars extraction from SCG hemicellulose, as a first step to explore the use of this material in fermentative processes. Initially, the material was chemically characterized, and its hemicellulose content was determined. In a subsequent stage, extraction reactions were performed by using dilute acid under different operational conditions (liquid-to-solid ratio, acid concentration, reaction time and temperature), which were proposed according to experimental designs. The condition able to maximize the extraction results was established by statistical analysis.

2. Material and methods

2.1. Raw material characterization

Spent coffee grounds (SCG) were supplied by NovaDelta-Comércio e Indústria de Cafés, S.A. (Campo Maior, Portugal). The material with around 80% humidity was dried at 60 ± 5 °C to 10% moisture content, being thus stored until be required for processing or analysis.

For chemical characterization, dried SCG was subjected to a quantitative acid hydrolysis with 72% (w/w) sulfuric acid. In this method, 2 g of sample were first added to 10 ml 72% H₂SO₄ and maintained at 50 °C for 7 min. After this pre-treatment, distilled water was added to the mixture to dilute the H₂SO₄ to 1N, and incubated at 121 °C for 45 min. The monosaccharides and acetic acid contained in hydrolysates were determined by HPLC in order to estimate the contents of samples in cellulose (as glucan), hemicellulose (as mannan + galactan + arabinan + xylan) and acetyl groups. Protein content was estimated by the Kjeldahl nitrogen method, and a factor of 6.25 was used to convert nitrogen into protein. Ashes were determined by weight difference before and after incineration of the SCG sample in a muffle furnace at 550 °C for 4 h. Before weighing, samples were placed in a desiccator for 50 min. The mineral content was determined by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES). All determinations were carried out in triplicate.

2.2. Dilute acid hydrolysis

Dried SCG was submitted to hydrolysis reactions under different conditions of H₂SO₄ concentration (100–140 mg/g dry matter), liquid-to-solid ratio (10–14 g/g), temperature (100–180 °C), and reaction time (15–75 min), which were combined as proposed in experimental designs. For the experiments, SCG and the required amount of acid solution were placed in 200-ml stainless steel batch cylindrical reactors. Under the desired temperature, the dully covered reactors were introduced into a silicone oil bath where they were maintained during the necessary time. At the end of each reaction, the reactors were immediately cooled in ice bath, and the resulting solid material was separated by filtration. The filtrates (hemicellulosic hydrolysates) were analyzed for sugars (cellobiose, glucose, arabinose, mannose, galactose, and xylose), degradation products (furfural, hydroxymethylfurfural, and total phenols), and acetic acid determination.

2.3. Experimental designs

Initially, a 2⁴ full-factorial design with three levels leading to 19 sets of experiments was made to evaluate the effect of four variables, namely the liquid-to-solid ratio (X₁), acid concentration (X₂), reaction time (X₃), and temperature (X₄), on the hydrolysis of SCG hemicellulose. For statistical analysis, the variables were coded according to Eq. (1), where each independent variable is represented by x_i (coded value), X_i (real value), X₀ (real value at the center point), and ΔX_i (step change value). The levels of the variables investigated in this study are given in Table 1. Three assays in the center point were carried out to estimate the random error needed for the analysis of variance, as well as to examine the presence of curvature in the response surfaces. Hydrolysis efficiency of galactan, mannan, arabinan, and hemicellulose were taken as responses of the design experiments.

$$x_i = \frac{X_i - X_0}{\Delta X_i} \quad (1)$$

Based on the results obtained in the 2⁴ design, a 2² central composite design was proposed to maximize the sugars recovery from

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