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Analytical Methods

Sediments in coffee extracts: Composition and control by enzymatic hydrolysis

P.A. Delgado [a], J.A. Vignoli [b], M. Siika-aho [c], T.T. Franco [a,*]

[a] School of Chemical Engineering, State University of Campinas, UNICAMP, P.O. Box 6066, 13081-970 Campinas, SP, Brazil
[b] Cia. Iguaçu de Café Solúvel, BR 369, Km 88, 86300-000 Cornélio Procópio, PR, Brazil
[c] VTT Biotechnology, FIN-0204, P.O. Box 1500, Espoo, Finland

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Abstract

The water-insolubility of some coffee extract components is one of the major limitations in the production of instant coffee. In this work, fractions from coffee extracts and sediments were prepared, and their chemical composition determined. Based on the carbohydrate analysis, galactomannan was found to be the main polysaccharide component of the insoluble fractions and probably responsible for sediment formation. The suitability of twelve commercial enzymes for the hydrolysis of the insoluble fractions was investigated. Pectinase 444L was the most effective enzyme in releasing sugars, mainly mannose and galactose, from these substrates. Biopectinase CCM, Rohapect B1L, Pectinase 444L and Galactomannanase ACH were found to be the most effective enzymes for reducing the sediment of coffee extracts. The highest sediment reduction was obtained using Rohapect B1L and Galactomannanase ACH, at enzyme concentrations of 0.3 and 0.1 mg protein/g substrate, respectively.

Keywords: Coffee polysaccharides; Galactomannans; Instant coffee; Enzymatic hydrolysis; Coffee composition; Sediment

1. Introduction

Coffee is one of the world's most widely consumed beverages. The chemical composition of the coffee cell wall has not been studied in detail, since it is difficult to dissolve, extract and digest (Kasai, Konishi, Iwai, & Maeda, 2006). Polysaccharides comprise nearly 50% of the green coffee bean weight (Fischer, Reimann, Trovato, & Redgwell, 2001; Nunes & Coimbra, 2001; Nunes, Reis, Domingues, & Coimbra, 2006), and those found in the coffee cell wall are mainly galactomannan, arabinogalactan and cellulose (Fischer et al., 2001; Oosterveld, Harmsen, Voragen, & Schols, 2003; Redgwell, Trovato, Curti, & Fischer, 2002). Arabinogalactans consist of a main chain of 1→3 linked galactose branched at C-6, with side chains containing arabinose and galactose. Galactomannans consist of a main chain of 1→4 linked mannan with galactose unit side chains linked at C-6, and different degrees of branching (Bradbury & Halliday, 1990; Navarini et al., 1999; Nunes et al., 2006). The structures of the polysaccharides of industrialized coffee products depend on the degree of roasting (Nunes & Coimbra, 2002; Oosterveld, Voragen, & Schols, 2003; Redgwell, Trovato, et al., 2002).

The main obstacle to characterizing the coffee cell wall is the high proportion of insoluble polymers (Bradbury & Atkins, 1997; Fischer et al., 2001; Redgwell, Curti, Fischer, Nicolas, & Fay, 2002). The solubility increases with increasing degree of branching and decreasing molecular weight (Nunes & Coimbra, 2001). Arabinogalactans dissolve better than do linear mannans, which can easily precipitate, and one of the reasons for this non-dissolution is an association of linear mannans to form crystalline regions (Bradbury & Atkins, 1997). This could be the reason for the formation of sediment during the manufacture of instant coffee. According to Fischer et al. (2001), the

* Corresponding author. Tel.: +55 19 3521 3966; fax: +55 19 3521 3965.
E-mail address: franco@feq.unicamp.br (T.T. Franco).

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difficulty in dissolving the cell wall polysaccharides indicates an intimate association between some of the arabinogalactan, galactomannan and cellulose molecules.

Proteins are another important component of coffee extracts. The roasting process causes degradation of the proteins into smaller products (Nunes & Coimbra, 2001). In espresso coffee, the protein content was shown to be correlated with the foam volume (Nunes, Coimbra, Duarte, & Delgadillo, 1997), and the protein is usually covalently linked to arabinogalactans (Fischer et al., 2001; Redgwell, Curti, et al., 2002; Navarini et al., 1999; Redgwell, Schmitt, Beaulieu, & Curti, 2005). Information on coffee lipids is very limited, but it has been speculated that poor quality of coffee is also due to the hydrolysis of triacylglycerols (TAGs) with the release of free fatty acids, which, in turn, are oxidized (Jham, Velikova, Muller, Nikolova-Damyanova, & Cecon, 2001; Nikolova-Damyanova, Velikova, & Jham, 1998; Segall, Artz, Raslan, Jham, & Takahashi, 2005). The main classes of lipids present in green coffee are triacylglycerols (75%) and terpene esters (14%) (Jham et al., 2001; Nikolova-Damyanova et al., 1998). There are still no reports available on the lignin content of coffee, but the lignin is found closely associated with the cellulose and hemicellulosic polysaccharides (de Vries & Visser, 2001; Dóka, Bicanic, & Bunzel, 2004; Juhász, Szengyel, Réczey, Siika-aho, & Viikari, 2005).

In Brazil, coffee extracts are processed into instant coffee or concentrated extract for exportation. However, during storage and commercial circulation, sediment is sometimes observed in the extracts, which is considered to be a quality defect and limits the utilization of the product.

These days, enzymes are commonly used in many industrial applications, including the degradation of plant cell walls. Cellulases, hemicellulases and pectinases are industrially important enzymes that are sold in large amounts for many applications. Different authors have investigated the use of enzymes to hydrolyze coffee polysaccharides. Nunes et al. (2006) isolated the galactomannans from light and dark roasted coffee infusions, and hydrolyzed them with endo-mannanase, decreasing the molecular weight of these polysaccharides. Mannanase can also be used to reduce the viscosity of the extract in the production of instant coffee, improving the effectiveness of the concentration process and reducing drying costs (Sachslehner, Foidl, Foidl, Gübitz, & Haltrich, 2000). These authors hydrolyzed the coffee mannan with free and immobilized mannanase from *Sclerotium rofsii*.

The aim of this work was to determine the composition and study the enzymatic hydrolysis of coffee fractions using different commercial enzyme preparations, and then to apply the enzymatic treatment to the whole extract in order to reduce the sediment formed during coffee processing.

2. Materials and methods

2.1. Materials

Coffee extract containing sediment was supplied by Cia Iguaçu de Café Solúvel (Cornelio Procópio, Paraná, Brazil). Enzyme preparations were obtained from different sources, and are described in Table 1. Monosaccharide standards were purchased from Sigma and Fluka, and all other reagents and solvents were of the highest purity.

2.2. Preparation of the coffee and sediment fractions

In the process used by the Cia Iguaçu, green coffee beans were roasted and ground, and the ground coffee then percolated by hot water under high pressure to extract the solids. The extract obtained was stored in tanks at 4 °C, where the sediment formed. The whole extract (containing sediment) and the sediment alone were the samples used in the present work, being fractionated according to Fig. 1. All the fractions were freeze-dried.

2.3. Chemical analysis

Neutral and acidic sugars were analyzed according to the Saeman hydrolysis (Selvendran, March, & Ring, 1979). In this method, 10 mg of sample were first added to 0.5 ml of

Table 1
Source, major activity and protein content of the commercial enzyme preparations

Enzyme	Source	Major activity[a]	Protein content (mg/ml)
Econase CE	AB enzymes	Cellulase	110 ± 4.24
Protease GC 106	Genencor	Protease	86.0 ± 1.41
Novo Shape	Novozymes	Pectinase	28.2 ± 0.21
Pectinex 3XL	Novozymes	Pectinase	17.7 ± 0.42
Pectinex Ultra	Novozymes	Pectinase	50.6 ± 0.85
Biop. CCM	Biofincon	Pectinase	40.6 ± 1.70
Biop. Super 8x	Quest	Pectinase	57.9 ± 1.46
Pectinase 444L	Biocatalysts	Pectinase	16.9 ± 0.04
Rohapect B1L	AB enzymes	Pectinase	37.9 ± 0.14
Rohapect D5L	AB enzymes	Pectinase	12.9 ± 0.33
Rohapect 10L	AB enzymes	Pectinase	67.6 ± 2.62
Galactomannanase ACH	Sumizyme	Galactomannanase	0.3 ± 0.01[b]

[a] According to the manufacture.
[b] mg/mg (enzyme powder).