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Analytical Methods

Extraction and characterization of pectins from cocoa husks: A preliminary study

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Abstract

Cocoa husks, a by-product of cocoa processing, were investigated as a source of pectins. Preliminary results of pectin recovery and characterization are shown; they constitute the first part of a study for the optimization of pectin extraction from this by-product. Husks of two different origins (Ghana and Venezuela) were used whole or minced and pectins were extracted under various conditions (pH 7.0, 4.0, 2.5, 1.5 and 1.0; extraction periods 1–3 h): the highest yield is obtained with minced husks after 1 h of extraction at pH 2.5. A preliminary characterization of pectins, in terms of methyl and acetyl ester contents, was also carried out in order to investigate the influence of different extraction conditions on the chemical composition of the extracts.

Keywords: Cocoa husks; Pectins; Extraction; Characterization

1. Introduction

Pectins are complex carbohydrate molecules; they are used mainly as gelling agents in the food industry, however pharmaceutical and cosmetic uses are also known. Usually, industrial pectins are extracted from citrus peels and apple pomace with a multiple-stage physical-chemical process characterized by an extraction step with hot dilute mineral acid and recovery through alcohol precipitation (Daniel, Voragen, & Pilnik, 1994; May, 1990). Recently, non-traditional pectin sources have been investigated. Pectins have been extracted from various food industry by-products and, considering that food processing is characterised by large amounts of waste material, by this way the extraction process could represent an efficient and environmental friendly matter recovery for the production of functional compounds (Kroyer, 1995; Schieber, Stintzing, & Carle, 2001).

In the present study, cocoa husks, a by-product of the chocolate industry, have been investigated as a potential source of pectins. Cocoa husks represent a disposal problem, since increased processing of cocoa beans brings increasing wastes and this is a good reason to come up with a useful outlet for this by-product (Redgwell et al., 2003; Serra Bonvehì & Escolà Jordà, 1998). Cocoa husks contain a lower amount of pectins (about 9% dry weight) than citrus peels and apple pomace (about 30% and 15%, respectively), hence it is necessary to optimise the extraction process for improved yield, purity and functional properties, but at the same time, due to their low humidity, they present many advantages (e.g. they can be stored and transported without further processing). Cocoa husks pectins can be recovered by adapting the hot acid extraction method used for production from conventional raw materials; however, considering that little is known about this recovery, it is necessary to investigate the influence of extraction parameters on pectin yield and quality and their possible improvement through additional processing (e.g. pre-treatment of the raw material with microwave heating or enzymes). Finally, impurities like pigments or residual

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acids contained in the precipitated pectins, must be determined and reduced (Kratchanova, Pavlova, & Panchev, 2004; Sahari, Akbarian, & Hamedi, 2003).

The main objectives of this research were to define optimum conditions for cocoa husks pectins extraction and to give a preliminary chemical characterisation of the obtained product in terms of quality and purity.

2. Materials and methods

2.1. Raw materials and chemicals

Husks of cocoa imported from Ghana and Venezuela were utilized; both whole or minced husks were applied in the extraction procedure (husks were ground in a grinding mill).

All the chemicals and reagents were of analytical grade (Sigma-Aldrich S.r.l. Milano, Italy). The kit for the enzymatic analysis of acetic acid (Cat. No. 10 148 261 035) was purchased by Boehringer Mannheim/R-BIOPHARM AG, Darmstadt, Germany.

2.2. Pectins extraction and recovery

Pectins extraction was based on the method proposed by De Giorgi, Tomasicchio, and Andreotti (1985). Husks were extracted with water (1:25, w:v), acidified or not with HCl, at 95 °C, for 1 h or more. The complete set of experiments and extraction conditions is summarized in Table 1.

After extraction, the slurries were filtered through four layers of gauze and then the filtrate was passed through a Büchner funnel under vacuum with a Whatman No.1 qualitative filter paper. Pectins were precipitated by adding seven volumes of boiling ethanol 99.8%. The precipitate was filtered under vacuum through a mixed cellulose ester filter (0.45 μm) (Advantec MFS, Inc., USA) and washed with 10 volumes of an ethanol solution (63%). Coagulated pectins were finally solubilized with 0.05 N NaOH.

2.3. Microwave heating of cocoa husks

One set of extraction experiments was carried out by pre-treating Ghana cocoa husks by microwave heating (Kratchanova et al., 2004). Minced husks (20 g) were hydrated with 50 ml of distilled water; after 30 min the excess water was discharged and husks were heated in a microwave oven for 15 min at maximum power (750 W).

Table 1
Extractions conditions

Extraction time (hours)	Cocoa husks							
	Ghana			Venezuela				
	Whole pH	Minced pH		Minced pH				
1	7.0	7.0	2.5	–	4.0	2.5	1.5	1.0
2	7.0	7.0	–	–	–	–	–	–
3	7.0	7.0	–	7.0	–	–	–	–

The entire quantity of irradiated ground husks was extracted as described above (extraction at pH 2.5 for 1 h).

2.4. Pectins characterization

Anhydrogalacturonic acid (%AGA) content of pectins was determined by the colorimetric phenyl–phenol method (Blumenkrantz & Asboe-Hansen, 1973) with an UV/VIS spectrophotometer, at 524 nm (ABS_{524}).

The determination of the ester methoxyl content (%DM) was carried out by two different analytical methods. It was firstly determined by saponification with 0.25 N NaOH and titration with 0.1 N NaOH in the presence of Hinton's indicator according to the method proposed by McCready, 1970. This content was also determined from the methanol liberated upon its alkaline hydrolysis through an enzymatic method with alcohol oxidase, peroxidase and the chromogen 2,2′-azinobis(3-ethylbenzthiazoline-6-sulfonic acid) (Mangos & Haas, 1996).

Acetyl ester content was determined by an enzymatic UV-method based on the formation of an amount of NADH, measured by the increase in light absorbance at 340 nm, which is proportional to the acetic acid concentration.

The presence of polyphenols in solubilized pectins was determined with an UV/VIS spectrophotometer, at 325 nm, as reported by Petrus and Dougherty (1973).

All analyses of pectins were performed in triplicate.

3. Results and discussion

3.1. Influence of the extraction pH and time on pectin yield

The extraction process for cocoa husks pectins was optimised on the basis of two different conditions: pH and extraction time.

A first set of extractions was performed with Ghana cocoa husks. Firstly, whole or minced husks were extracted at a neutral pH and at three different extraction times (1–3 h); in this way the applicability of the process was demonstrated. Secondly, taking into account that hot acid extraction is usually the most efficacious, the procedure was repeated with minced husks at pH 2.5 for 1 h. Fig. 1 shows the obtained results expressed as %AGA by husks dry weight. Considering the extraction at neutral pH with whole husks, the pectins quantity is influenced by the extraction time; in fact, when the time passes from 1 to 2 h also the extracted quantity is doubled (2.0% and 4.0%, respectively), and when the extraction lasts 3 h a little improvement is obtained (4.7%). When the same runs are repeated with minced husks, as expected, the amount of recovered pectins rises appreciably because of the higher contact surface during the extraction; as a consequence, all the subsequent experiments were conducted with minced husks. On the other hand, the influence of time was lower with respect to results obtained with whole husks; in this case, pectins quantity passes from 5.0% to 6.0% for 1 and 2 h of extraction, respectively, while with 3 h 6.5% is