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Effect of addition of green coffee extract and nanoencapsulated chlorogenic acids on aroma of different food products

Grażyna Budryn*, Donata Zaczyńska, Joanna Oracz

Institute of Food Technology and Analysis, Faculty of Biotechnology and Food Sciences, Lodz University of Technology, 90-924, Lodz, Poland

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ABSTRACT

Between food polyphenols and proteinaceous compounds occur noncovalent interactions. The interactions can affect some properties of these compounds, such as nutritional value, bioactivity and organoleptic characteristics. When polyphenols are used as food additive, the changes can by limited by their encapsulation. The aim of the study was to determine, how the addition of polyphenols, i.e. chlorogenic acids, from green coffee in free form or nanoencapsulated by β-cyclodextrin and further the addition of hydrolysate of ovalbumin, whey proteins or soy proteins will affect the content and the profile of volatile aroma compounds of six food products: bread, cookies, caramel cottage cheese, nutty filling, and mushroom or meat stuffing. Considering the six food products a general trend of the content of volatile compounds caused by supplementing with the used additives was observed. The increase of aroma volatiles content in the headspace of the products increased in the following order: nanoencapsulated chlorogenic acids < control < protein hydrolysates < free chlorogenic acids < chlorogenic acids in free form with protein hydrolysates.

1. Introduction

Polyphenols are often used to enrich food products because of the high activity to prevent oxidative changes in food and to limit oxidative stress in human organism (Maat et al., 2005). Coffee is one of rich sources of polyphenols, especially phenolic acids (Daglia, Papetti, Gregotti, Berté, & Gazzani, 2007). Green coffee beans contain mainly hydroxycinnamic acids, such as caffeic and ferulic acids, as well as their esters with quinic acid, referred to as chlorogenic acids (CHAs) (Budryn, Żyżelewicz, Nebesny, Oracz, & Krysiak, 2013). They exhibit anti-inflammatory and anti-mutagenic effects, which prevent chronic disorders, such as tumors, cardiovascular and rheumatologic diseases (Cheng, Dai, Zhou, Yang, & Liu, 2007). Consumption of coffee can also be helpful in the fight against obesity and in limiting the effects of type 2 diabetes (Bassoli, et al., 2008).

To achieve the positive effects of polyphenols consumption, their average daily intake has to be on a level not lower than 1 g (Williamson & Holst, 2008). Many studies considering supplemented food showed that such a large amount of polyphenols in one serving can strongly interact with proteinaceous components (Renouf et al., 2010). The interactions could limit the functionality of both proteins and polyphenols, especially they decrease their absorption and reduce the antioxidant activity (Rawel & Rohn, 2010). Because of these facts it may be preferable to nanoencapsulate CHAs by forming inclusion complexes with β-cyclodextrin (β-CD) and adding in such form to foods (Nasirullah, Kumar & Shariff, 2011; Szejtli & Szente, 2005). The aromatic ring of CHAs located inside the β-CD can be protected against reactions with food proteins. Such properties of β-CD-CHAs inclusion complexes during processing with food proteins and protein hydrolysates were already confirmed in model studies (Budryn et al., 2014; Budryn, Zaczyńska, Rachwał-Rosiak, & Oracz, 2015; Budryn et al. 2015; Budryn et al., 2016; Budryn, Zaczyńska, & Rachwał-Rosiak, 2016). Inclusion of CHAs with β-CD presumably does not limit their bioavailability and antioxidant activity (Paramera, Konteles, & Karathanos, 2011).

The interactions of polyphenols with proteinaceous preparations could change not only pro-health activities but also organoleptic properties of foods and forming of some specific volatiles could take place (Ahmad et al., 2015; Noor-Soffalina, Jinap, Nazamid, & Nazimah, 2009). Thus the form of added polyphenols and their ability to interact with proteins would be significant for aroma volatiles formation. So far no studies on the effect of food

* Corresponding author.
E-mail address: grazyna.budryn@p.lodz.pl (G. Budryn).

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supplementation with phenolic compounds in different form on aroma volatiles profile were conducted. The aim of this study was to supplement six food products with free or nanoencapsulated CHAs, and also with protein hydrolysates, to compare the composition of headspace aroma volatiles of the obtained products depending on the used additives.

2. Materials and methods

2.1. Chemicals and raw materials

Analytical-grade ethanol, methanol and ethyl acetate were purchased from Poch (Gliwice, Poland), β-cyclodextrin (β-CD, ≥98%) from Sigma Aldrich (St. Louis, MO, USA) and nylon filters from Chromacol (Herts, UK).

Green Robusta coffee beans (*Coffea canephora* L.) harvested in Brazil in 2012, hulled by dry method were purchased from Bero Polska (Gdynia, Poland). Protein hydrolysates: whey protein hydrolysate Amino 4500 (WPH) was purchased from Trec Nutrition (Gdynia, Poland), egg ovalbumin hydrolysate A 6710 (EOH) from Sigma (St. Luis, MO, USA) and soy protein hydrolysate S 1674 (SPH) from Fluka (St. Luis, USA). All the used ingredients for obtaining food products were purchased from the local market.

2.2. Preparation and purification of green coffee extract (GCE)

The aqueous extract from green coffee beans was obtained as previously described (Budryn et al. 2014). The extract contained isomers of caffeoylquinic, feruloylquinic and dicaffeolyquinic acids. The solution was frozen at −80 °C, freeze-dried in a DELTA 1-24LSC Christ freeze drier (Osterode am Harz, Germany) and purified by centrifugal partition chromatography (CPC) method with SPOT Prep II 50 chromatograph from Armen Instrument (Saint-Avé, France) integrated with UV/VIS detector and a fraction collector. Briefly, the two-phase system of solvents was prepared from water, ethanol and ethyl acetate (5:1:4, v/v/v). Elution of CHAs occurred from 20 to 24 min and from 29 to 32 min of the analysis, detected by UV absorption at 320 nm. Collected fractions were concentrated in ScanMaxiVac concentrator Labogene (Lynge, Denmark) and again freeze-dried. The content of CHAs in the purified GCE was 564.8 mg g^{-1} db. (dry basis) and caffeine was eliminated. The remainder of the extract were proteins (29.5 mg g^{-1} db.), sugars (92.8 mg g^{-1} db.), soluble fiber (121.6 mg g^{-1} db.) and minerals (191.3 mg g^{-1} db.).

2.3. Preparation of inclusion complexes of β-CD with CHAs from GCE (β-CD-CHAs)

Inclusion complexes of β-CD with CHAs were prepared with accordance to the previously developed method (Budryn et al. 2014). Briefly 113.5 mg of β-CD and 130.3 mg of GCE, which contained 70.8 mg of CHAs were dissolved in 2 mL of water. The complexation was conducted for 2 h at 50 °C in a Pierce Reacti-Therm TS-18821 reactor from Thermo Scientific (Palo Alto, CA, USA). After complexation the solution was left for 24 h at 0 °C and the suspension was centrifuged in MIKRO 22R centrifuge from Hettich (Kirchlengern, Germany) at 4 °C for 20 min at 10 000 × g. The precipitate was washed twice with 5 mL of methanol, suspended in 5 mL of ice-cold water, frozen at −80 °C and freeze-dried. A mixture of β-CD complexes with particular CHAs was obtained and characterized by ESI-MS/MS method (Budryn et al. 2014). The concentration of CHAs in complexes amounted 237.7 mg g^{-1}.

2.4. Food products formulations and obtaining

Types of food products were chosen due to their characteristic taste properties harmonizing with those of CHAs. The obtained products included: bread (BR), cookies (CO), caramel cottage cheese (CT), nutty filling (NF), mushroom (MU) and meat stuffing (ME), and each product was prepared in five modifications comprising: control (C), supplemented with GCE (GCE), supplemented with protein hydrolysate (PH), supplemented with both GCE and PH (GCE-PH), and supplemented with CHAs in the form of inclusion complexes (β-CD-CHAs). Three different protein hydrolysates were used, each of them was added to two products, taking into account organoleptic and functional properties accordant with those of the products. BR and CO were supplemented with egg ovalbumin hydrolysate (EOH), CT and NF with whey protein hydrolysate (WPH), MU and ME with soy protein hydrolysate (SPH).

The formulations of all prepared food products are given in Table 1 (Budryn et al., 2016).

Cookies: all ingredients were mixed with blender, formed into round biscuits with diameter of 40 mm and a height of 3 mm and baked in a baking oven Piccolo PRO II Winkler Wachtel (Düsseldorf, Germany) at 220 °C for 10 min.

Bread: a half of water amount was mixed with yeast, sugar and a quarter of the flour amount. After 20 min of pre-fermentation at 33 °C remaining ingredients were added. Prepared mixture was left for 30 min to continue fermentation. Afterwards the loaf was formed and baked in a baking oven Piccolo PRO II Winkler Wachtel (Düsseldorf, Germany) at 200 °C for 55 min.

Caramel cottage cheese: all the ingredients were mixed with blender.

Nutty filling: fat was liquefied at 40 °C and mixed with the remaining components with blender.

Mushroom and meat stuffing: shredded onion was fried in rape oil with mushrooms or pork depending on a kind of stuffing. After cooling spices, CHAs and optionally soy protein hydrolysate were added.

2.5. HS-SPME-GC-MS analysis of aroma volatile compounds

The aroma volatiles from headspace (HS) layer were absorbed by solid phase microextraction (SPME) technique. Absorption of headspace volatiles was conducted using a Divinylbenzene/Carboxen/Polydimethylsiloxane (DVB/CAR/PDMS) fiber (50/30, 80 μm). Before each analysis, the SPME fiber was conditioned in the dispenser of a gas chromatograph at 250 °C. A sample of 500 mg of a product was taken into a glass vial of 2 mL capacity. The vial was closed with a cap and PTFE-silicone seal and conditioning for 45 min at 40 °C. Subsequently the SPME fiber was placed in HS layer over the product for 40 min at 25 °C. After that the fiber was inserted to the injector system operating in the splitless mode at 220 °C. The fiber was left for 1 min to desorb the volatile substances.

The analysis of the volatile compounds desorbed from the SPME fiber was performed using a Varian gas chromatograph GC-450 coupled with a mass spectrometer Saturn 220-MS Varian (GC-MS) (Palo Alto, CA, USA). GC was equipped with a capillary column ZB-WAX 30 m × 0.25 mm × 0.25 mm. Helium was used as GC carrier gas with a flow of 0.7 mL min^{-1}. The used temperature of the operating program included initial 35 °C maintained for 5 min, then increased by 5 °C min^{-1} to 50 °C and further increased by 5.5 °C min^{-1} to 230 °C maintained for 10 min. Mass spectra were obtained under electron ionization (EI) at 70 eV with *m/z* range of 30–200. The temperature of the ion source and that of the transfer line was 180 °C and 200 °C, respectively. The identification of volatiles was performed by comparing the mass spectra of the