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Bioactive compositions of extracts from cabbage outer leaves as affected by drying pretreatment prior to microwave-assisted extraction

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ABSTRACT

In order to add value to cabbage outer leaves, which are generally considered as a waste product, extraction and use of their health-beneficial bioactive compounds should be made. Among many extraction alternatives microwave-assisted extraction (MAE) is one of the most promising and has indeed been applied to extract bioactive compounds from cabbages. However, study is lacking on the effect of drying prior to MAE, which is sometimes required since extraction cannot always be performed on fresh materials. This study was divided into 2 parts, with the first part investigating the effects of selected drying methods, i.e., hot air drying (HD) and low-pressure superheated steam drying (LPSSD), on selected bioactive compounds, namely, glucosinolates (GLS), sulforaphane, phenolics, as well as the total antioxidant activity (TAA) of the dried cabbages. In the second part the effects of the drying methods on subsequent MAE were assessed. The results suggested that HD but not LPSSD led to a significantly decreased GLS content of the dried cabbages. Sulforaphane and total phenolics contents as well as TAA of the dried cabbages decreased upon both HD and LPSSD. The GLS and total phenolics contents as well as TAA of the extracts from the dried cabbages prepared by HD and LPSSD were lower than those from the fresh sample. The results on the sulforaphane content of the extracts were nevertheless opposite.

1. Introduction

Cabbages (*Brassica oleracea* L. var. capitata) are generally sold with their outer leaves removed; the leaves are normally discarded or used only as animal feed or fertilizer. Nevertheless, cabbage outer leaves have been reported to contain various health-beneficial bioactive compounds, including various phenolic compounds. The leaves also contain glucosinolates and sulforaphane [10,13,14]. Glucosinolates (GLS) are a group of sulfur-containing secondary plant metabolites. When cabbage tissues are damaged, GLS are hydrolyzed by enzyme myrosinase into degradation products with different bioactivities. GLS and their breakdown products, in particular, sulforaphane (4-methylsulfinybutyl isothiocyanate) are known to possess anticarcinogenic properties [11].

A number of extraction methods can be used to extract the above-mentioned compounds from cabbages or indeed any compounds from any plant materials. Among many alternatives microwave-assisted extraction (MAE) is one of the most promising and has indeed been applied to extract bioactive compounds from cabbages. Tanongkankit et al. [14], for example, investigated the use of MAE to extract sulforaphane from white cabbages; the results were compared with those of conventional solvent extraction. MAE resulted in a higher extraction yield of sulforaphane within a much shorter extraction time. The results are in agreement with those of Dahmoune et al. [3] who studied MAE of phenolics from *Myrtus communis* leaves. The total phenolics content and antioxidant activity of the extract obtained via MAE were noted to be higher than those of the extract obtained via ultrasound-assisted extraction and solvent extraction.

In many cases, however, extraction cannot be performed on fresh materials. This may be because of the excessive supply of fresh plant materials during a harvesting season. Limited capacity of an extraction unit and hence its inability to process incoming fresh materials within a short period of time results in the need to preserve the fresh materials for later extraction. Drying is required to preserve the fresh materials in such a situation. Although hot air drying (HD) is commonly used for plant materials

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preservation, HD is known to cause much degradation to bioactive compounds due to the required long drying time and the oxygen-rich nature of the process [1]. For this reason many alternative drying methods have been proposed. Among the possible alternatives, low-pressure superheated steam drying (LPSSD) has proved successful in preserving the contents and activities of a wide array of heat- and oxygen-sensitive bioactive compounds in plant materials [4]. Niamnuy et al. [9] indeed investigated the use of different drying methods (hot air drying, infrared-hot air drying and LPSSD) to prepare dried centella leaves for subsequent conventional solvent extraction and reported that the phenolics content and antioxidant activity of the extracts obtained from the leaves dried by LPSSD were the highest.

So far limited information is available on the effects of drying methods prior to MAE (or any extraction methods) on the extractability and activity of bioactive compounds from cabbages (and actually other plant materials). The aim of this work was therefore to study the effects of HD and LPSSD prior to MAE on the contents of bioactive compounds extractable from cabbage outer leaves. GLS and sulforaphane as well as phenolics were focused as the main bioactive compounds. The antioxidant activity (TAA) of the extracts was also investigated. The effects of the selected drying methods on the bioactive profiles of the dried cabbages prior to MAE were also assessed.

2. Materials and methods

2.1. Chemicals

DEAE-Sephadex A-25, gallic acid and 2,2-diphenyl-1-picrylhydrazyl (DPPH) were obtained from Sigma–Aldrich (Steinheim, Germany); sinigrin standard and sulforaphane standard were purchased from Sigma–Aldrich (St. Louis, MO); Folin–Ciocalteu reagent was obtained from Carlo Erba (Milan, Italy); 2,2′-azinobis (3-ethylbenzothiazoline-6-sulfonic acid) diammonuim salt (ABTS) was purchased from Sigma–Aldrich (Oakville, Canada); 6-hydoxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox) was purchased from Sigma–Aldrich (Buchs, Switzerland).

2.2. Sample preparation

Outer leaves of cabbages were obtained from Pak Klong market in Bangkok. Raw leaves were kept at 4 °C until the time of an experiment. Prior to each experiment the leaves were washed under running tap water and drained on a screen to get rid of excess water.

Fresh sample for extraction was prepared by chopping the leaves using an electric chopper (Moulinex, DPA141, Ecully, France) for 2 min to obtain an average size of 1.7–2.5 mm. Dried cabbages, on the other hand, were prepared by slicing the fresh leaves to obtain a sample with the dimensions of 0.5 × 5 cm. The slices were introduced to either the hot air dryer (Memmert, ULM500, Schwabach, Germany) at 60, 70 and 80 °C or the low-pressure superheated steam dryer proposed by Devahastin et al. [5] at 70 and 80 °C at an absolute pressure of 10 kPa. Drying experiments were conducted until a sample final moisture content of less than 0.1 kg/kg (d.b) was reached. A dried sample was ground into powder to obtain the particle size of around 300 μm. The powder was packed in an aluminum packet and kept at −18 °C until extraction.

2.3. MAE experiments

A domestic microwave oven (Samsung, GE-872D, Port Klang, Malaysia) was modified for MAE as described by Hiranvarachat et al. [6]. A 1000-mL round-bottom flask containing an extraction solvent (99.9% (v/v) ethanol) and a sample was placed inside the microwave oven cavity. The flask was fitted with an external condenser to condense the vaporized extract, which was then collected in a graduated cylinder. Cold water (∼4 °C) was used as a condensing medium.

Five g of either fresh or dried cabbage sample was placed in the flask, which was filled with 50 mL of 99.9% (v/v) ethanol and extracted under microwave radiation. The tested set microwave power was 100 W, whereas the extraction time was 5 min; preliminary experiments revealed that this condition yielded the maximum content of sulforaphane from the fresh cabbages (2.14 ± 0.36 mg/100 g d.b.). After extraction the condensed extract and the remaining solvent in the flask were mixed and evaporated by a rotary evaporator (Buchi, R-215, Flawil, Swizerland) at 50 °C for 7 min to produce crude extract [6]. The content was diluted with another type and amount of solvent, depending on the required subsequent chemical analysis. Five mL of 99.9% (v/v) ethanol was added if the GLS content, total phenolics content (TPC) and TAA were to be determined. For sulforaphane analysis, 2 mL of acetonitrile was used for the dilution. A diluted sample was kept at −18 °C until further analysis.

2.3.1. Preparation of crude extracts for bioactive compounds contents determination

2.3.1.1. Preparation of crude extract for GLS content determination. Extraction of GLS was performed according to the modified method of Tanongkankit et al. [13]. Either fresh (1 g) or dried cabbages (0.3 g) were weighed into a tube, which was closed by a screw cap and placed in a water bath at 95 °C for 15 min. Four mL of boiling deionized water was then added; the whole content was immediately mixed by a vortex mixer (Scientific Industries, model G-560, Bohemia, NY) for 10 s. The tube was heated again at 95 °C for 3 min. After cooling the sample was centrifuged at 600 × g for 15 min; supernatant was transferred to a different test tube containing 150 μL of 0.5 M barium/lead acetate. The sample was re-extracted by adding 4 mL of boiling deionized water and placed at 95 °C for 3 min and again centrifuged at 600 × g for 15 min. The extract was combined with the first supernatant and made up to 10 mL with deionized water. The content was kept at room temperature until further analysis.

2.3.1.2. Preparation of crude extract for sulforaphane content determination. Extraction of sulforaphane was performed following the method of Tanongkankit et al. [14] with some modification. Fresh cabbages (5 g) or dried cabbages (3 g) were stirred with 50 mL of dichloromethane, which contained 2.5 g of sodium sulfate anhydrous, for 30 min. The extract was filtered through Whatman No. 1 filter paper. The residue was re-extracted with 50 mL of dichloromethane and 2.5 g of sodium sulfate anhydrous for another 15 min; the extract was again filtered. The extracts were mixed and dehydrated using the rotary evaporator at 50 °C. The residue was dissolved in 2 mL of acetonitrile and kept at room temperature until further analysis.

2.3.1.3. Preparation of crude extract for TPC determination. Extraction of phenolics was performed according to the modified method of Tanongkankit et al. [12]. Five g of a cabbage sample was shaken in a shaker (New Brunswick Scientific, model Innova 4230, Edison, NJ) with 50 mL of acetone-deionized water solution (1:1, v/v) at 120 rpm for 15 h at ambient temperature (∼30 °C). The acetone extract was filtered and kept in dark at room temperature until further analysis.

2.3.1.4. Preparation of crude extract for TAA determination. Ten g of either fresh or dried cabbages was shaken with 90% (v/v) ethanol