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How Different Cooking Methods Affect the Phenolic Composition of Sweet Potato for Human Consumption (*Ipomea batata* (L.) Lam)

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Abstract: In recent years, there has been increasing interest in the functional components of sweet potato because of its nutritional and medicinal value. The aim of this study is to analyse how much sweet potato phenolic compounds composition (derived from caffeoylquinic acids) varies as a result of cooking. Traditional techniques such as: boiling, oven roasting and more recent processing techniques such as microwave cooking were tested. Three sweet potato varieties were cooked for different periods of time and under different conditions. Ultrasound-assisted extraction (UAE) was used to extract the compounds of interest and then, a chemometric tool such as Box-Behnken design (BBD) was successfully used to evaluate and optimise the most influential factors in the extraction, i.e., temperature, solvent composition and sample-to-solvent ratio. The optimal settings for UAE were: solvent 100% methanol, a temperature of 39.4 °C and a mass/volume ratio of 0.5 g per 10 mL solvent. Oven roasting of sweet potatoes resulted in increased levels of caffeoylquinic acids, whereas prolonged cooking times in water resulted in decreasing levels of the same.

Keywords: sweet potato; caffeoylquinic acid; ultrasound-assisted extraction (UAE); *Ipomea batata* (L.) Lam; cooking

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1. Introduction

Sweet potato (*Ipomoea batata* (L.) Lam) is a dicotyledonous species that belongs to the Convolvulaceae family. It is a perennial crop and one of the main sources of human and animal food [1]. In fact, in terms of worldwide production, it ranks sixth after rice, wheat, potato, maize and tapioca, with a total annual crop of over 90 million tonnes (FAOSTAT).

Sweet potato consumption has increased in recent years, not only as a traditional food but also at an industrial level through different processed products [2]. Its high energy content is explained by its high nutrient content [1,3]. In recent years, there has been increasing interest in its production and consumption in developing countries [4,5]. It is also a rich source of components with anti-carcinogenic, cardiovascular [6], anti-inflammatory [7], anti-tumour [8], anti-microbial [9] and antioxidant [3] properties, which protect against neuronal degeneration [10], liver damage from alcoholic beverages [11] and kidney failure [12].

The various bioactive components present in sweet potatoes are susceptible to changes, and those changes that they undergo as a result of storage [13,14], cooking [15–18] or certain

processing methods, such as drying [19], encapsulation by spray drying [2] or even cutting patterns [20], were evaluated.

The phenolic compounds contained in sweet potatoes which make of them an excellent source of antioxidant compounds are mainly found in its leaves [21,22]. Even so, their edible roots also contain derivatives of chlorogenic acid, in its mono- and di-esterified forms of caffeoylquinic acid (CQA) [23,24] (Figure 1) [25], as well as anthocyanins in the case of the purple-fleshed varieties [26–28], mainly in the form of acylated cyanidin and peonidin glycosides [29].

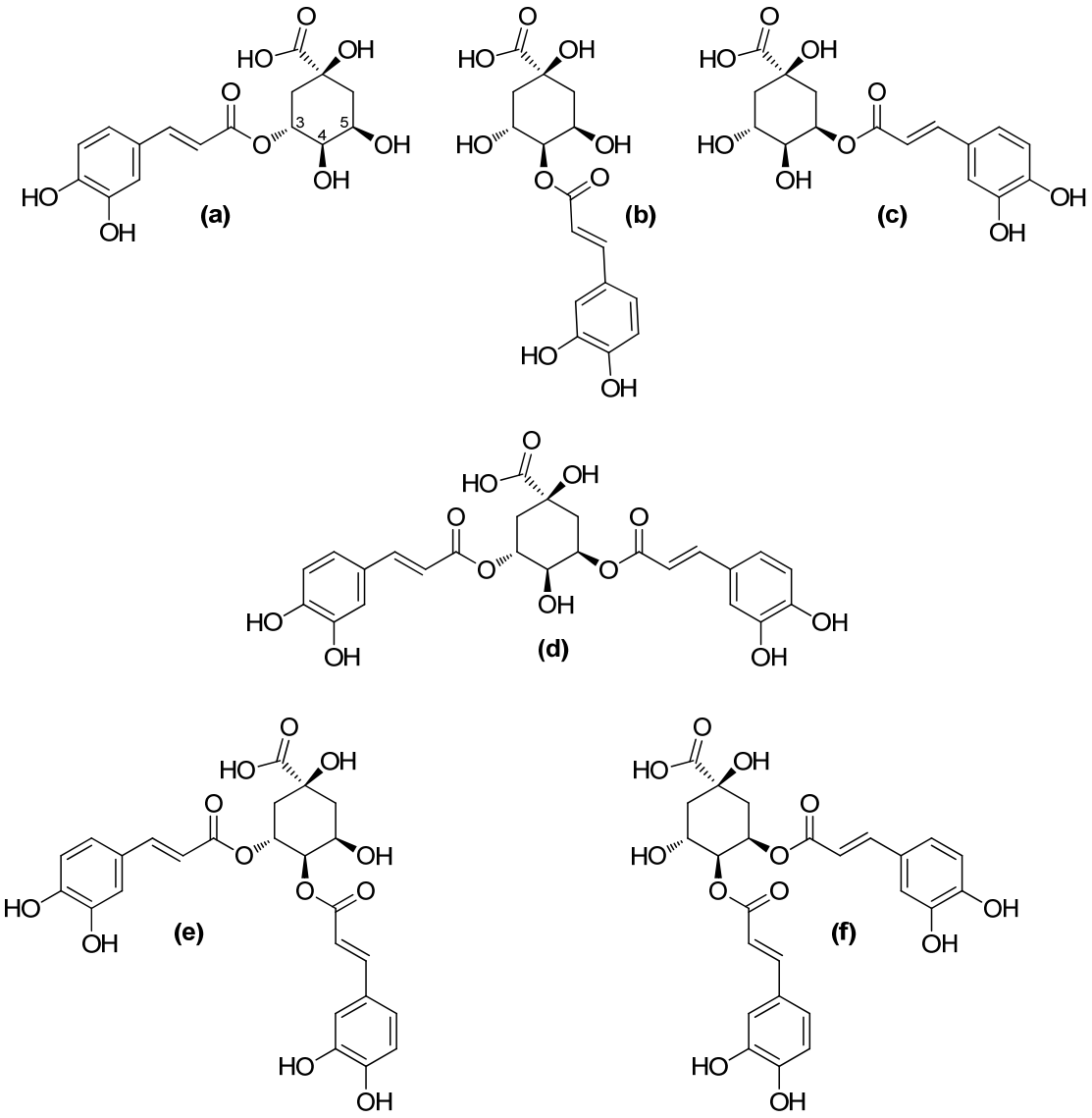

Figure 1. Derivatives of caffeic acid present in the roots of sweet potatoes. (**a**) 3-*O*-caffeoylquinic acid (3-CQA or chlorogenic acid), (**b**) 4-*O*-caffeoylquinic acid (4-CQA or cryptochlorogenic acid), (**c**) 5-*O*-caffeoylquinic acid (5-CQA or neochlorogenic acid), (**d**) 3,5-dicaffeoylquinic acid (3,5-diCQA or isochlorogenic acid A), (**e**) 3,4-dicaffeoylquinic acid (3,4-di-CQA or isochlorogenic acid B), (**f**) 4,5-dicaffeoylquinic acid (4,5-di-CQA or isochlorogenic acid C).

This study aims to evaluate the changes that such phenolic acid derivatives, which are present in the roots of three varieties of orange-fleshed sweet potato, undergo during different cooking processes.

2. Materials and Methods

2.1. Solvents and Reactants

For the extractions and chromatographic separations, the solvents used were: 100% acetic acid (glacial) (Merck, Darmstadt, Germany), Milli-Q water obtained by means of a Millipore water purification system (Bedford, MA, USA), acetronitrile and HPLC-grade methanol (Panreac, Barcelona, Spain). Chlorogenic acid (>95% purity, Sigma-Aldrich Chemical Co., St. Louis, MO, USA) was used as a standard for the quantification of mono-CQAs in the extracts. The standard used for the quantification of di-CQAs in the extracts was 3,4-diCQA (>90% purity, Sigma-Aldrich Chemical Co., St. Louis, MO, USA).

2.2. Sweet Potato Samples

Three varieties of sweet potato (*Ipomoea batata* L.), all of them of orange flesh, were tested. The sweet potato varieties were as follows: California (Agricultural Cooperative "Las Virtudes", Conil de la Frontera, Spain); Beauregard (Agricultural Cooperative "Virgen del Rocío", Sanlúcar de Barrameda, Spain); and Covington (purchased at a supermarket in Cadiz, origin USA).

2.3. Preparation/Cooking of the Sweet Potatoes

For the final preparation of the sweet potatoes, three types of cooking methods were chosen: boiled and roasted in the oven, as the traditional ways of consumption, and steamed in the microwave, as a new preparation technique.

Each preparation was carried out in duplicate. For each preparation, 10 sweet potatoes without any bruises or cuts were selected, and once the cooking process was finished, their pulp was homogenised in order to produce a consistent and representative sample. For the experiments with sliced sweet potatoes, the ends were removed, and the rest was cut into 3 cm thick pieces.

The cooking of the sweet potatoes was carried out using either whole sweet potatoes of similar size, or pieces of them, which were boiled for 7, 12 and 17 min. The boiling process was stopped by submerging the sweet potatoes into ice water at the setup time. The time ranges used were those traditionally used for the preparation of boiled sweet potato for human consumption.

The sweet potatoes were roasted in a domestic oven at 240 °C for 30, 35 and 40 min. This temperature and time range were selected as they are the most commonly used for the preparation of roasted sweet potato for human consumption. Microwave preparation was conducted according to the instructions on the commercial packaging, i.e., 7 min at 800 W.

2.4. Optimisation of the Extraction Process by Ultrasound-Assisted Extraction (UAE)

The optimal extraction conditions were determined using a Box-Behnken design of experiments on a sample of sweet potato of the Covington variety, roasted in an industrial oven purchased from a local retailer in Cadiz. Once roasted, the sweet potatoes were crushed and homogenised using a conventional hand blender (750 W) until a fine mash was obtained. This sample was stored at −20 °C until further analysis.

The ultrasonic extractions (UAE) were carried out using a UP 200S (200 W, 24 kHz) probe (Ultraschallprozessor, DR. Hielscher, Berlin, Germany) allowing amplitude and cycle control, coupled to a temperature-controlled thermostatic bath, 7 Litre refrigerator circulator (PolyScience, Niles, IL, USA).

In this study, the Box-Behnken design (BBD) was used to evaluate the effect of independent variables on the efficiency of ultrasound-assisted extraction (UAE). The three optimised variables were temperature, solvent composition and sample quantity to solvent volume ratio. Table 1 shows the range used for the three independent variables. For the optimisation of these variables, a total of 15 experiments were performed in duplicate.

Table 1. Range of variables selected for the Box-Behnken design.

	Range Applied		
Factor	**Lower Level (−1)**	**Intermediate Level (0)**	**Top Level (+1)**
Temperature (°C)	10	35	60
Solvent (% MeOH)	0	50	100
Ratio (g/10 mL solvent)	0.5	0.75	1.0

2.5. Analysis of the Phenolic Compounds by Ultra-High Performance Liquid Chromatography (UHPLC)

The analysis of the phenolic compounds present in the sweet potatoes was carried out using ACQUITY UPLC H-Class liquid chromatography equipment (Waters Corporation, Milford, MA, USA) coupled to an ACQUITY UPLC photodiode array (PDA) detector. The PDA detector was adjusted to a wavelength range of 240–400 nm for 3D scanning applying a sampling rate of 40 pts s^{-1}.

The column used for the separation of the phenolic compounds was an ACQUITY UPLC® BEH C18 (2.1 × 100 mm, particle size 1.7 µm, Waters, Milford, MA, USA). The temperature was set at 47 °C and the mobile phase used was a binary solvent system consisting of Milli-Q water acidified with 2% acetic acid as solvent A and acetronitrile acidified with 2% acetic acid as solvent B. The analysis was performed in 8 min using the following gradient sequence (time, %B): 0 min, 0%; 1 min, 0%; 3 min, 5%; 4 min, 10%; 4.5 min, 10%; 5 min, 20%; 7 min, 20%; 8 min, 30%. The flow rate was set at 0.6 mL/min.

Chlorogenic acid (3-CQA) was the standard selected for the quantification of the three extracted mono-esterified caffeoylquinic derivatives, according to the following calibration curve: y = 26,442x + 11,415, with a coefficient of determination R^2 = 0.9999. For the di-esterified derivatives, 3,4-diCQA was the standard used for the quantification, obtaining the following calibration curve: y = 117,249x + 31,108, with a coefficient of determination R^2 = 0.9994. Figure 2 illustrates a typical chromatogram of the Covington variety showing the identified compounds.

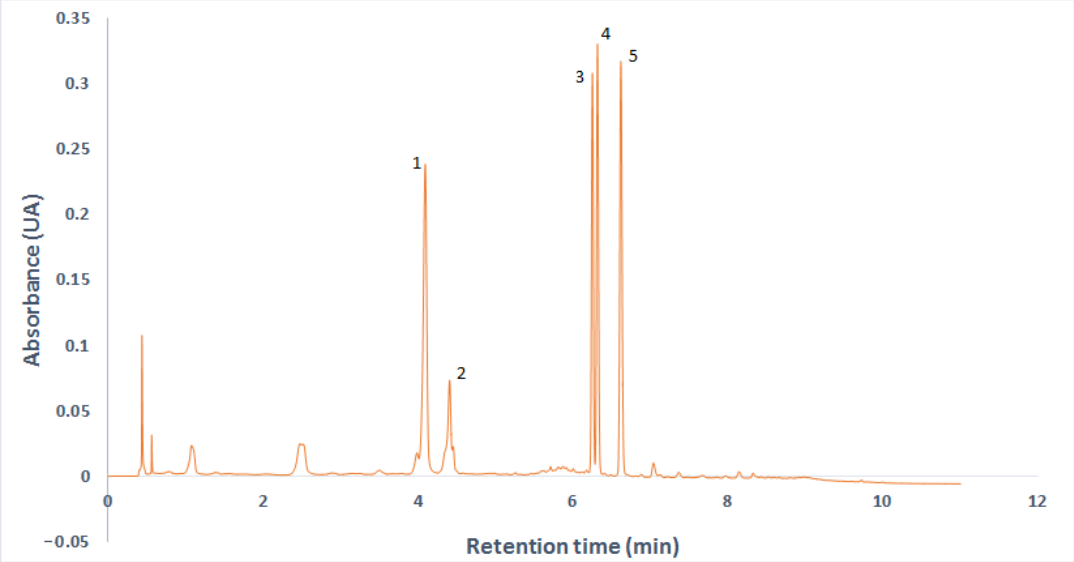

Figure 2. Chromatogram of the Covington variety (320 nm). (**1**) 5-*O*-caffeoylquinic acid (5-CQA or neochlorogenic acid), (**2**) 4-*O*-caffeoylquinic acid (4-CQA or crytochlorogenic acid), (**3**) 3,4-dicaffeoylquinic acid (3,4-di-CQA or isochlorogenic acid B), (**4**) 3,5-dicaffeoylquinic acid (3,5-di-CQA or isochlorogenic acid A), (**5**) 4,5-dicaffeoylquinic acid (4,5-di-CQA or isochlorogenic acid C).

2.6. Validation of the Extraction Method

For the validation of the method, the accuracy of the ultrasound-assisted extraction (UAE) was evaluated. The repeatability was evaluated by completing nine extractions on the same day (n = 9), while the evaluation of the intermediate precision was based on six daily extractions on three consecutive days (n = 6 + 6 + 6). The accuracy of the method was expressed as the coefficient of variation (%CV) in the responses obtained (normalized areas).

2.7. Identification of the Phenolic Compounds by Means of UHPLC-QToF-MS

The phenolic compounds present in the sweet potato samples were identified by ultra-high performance liquid chromatography coupled to a quadrupole time-of-flight mass spectrometer (UHPLC-QToF-MS, Xevo G2, Waters Corp., Milford, MA, USA). The mass spectra were acquired in negative ion mode under the following conditions: desolvation gas flow = 700 L h^{-1}, desolvation temperature = 500°C, cone gas flow = 10 L h^{-1}, source temperature = 150°C, capillary voltage = 700 V, cone voltage = 30 V and collision energy = 20 eV. The full scan mode (m/z = 100 − 1200) was used.

3. Results and Discussion

3.1. Identification of the Phenolic Compounds Present in the Samples

The sweet potato extracts were gathered and concentrated in a rotary evaporator until a signal intensity was reached that would allow the mass spectra to be obtained on the UHPLC-PDA-QToF-MS equipment (approximately a signal of 0.5 AUFS at maximum peak absorption: 320 nm).

A chromatogram similar to that obtained by the UHPLC-PDA system was obtained and the identification of the peaks was based on their mass spectra, on their maximum absorption in the UV-Vis spectrum and, given that the mono- and di-esterified derivatives showed common signals in their mass spectra, on their elution order and the subsequent comparison with those found in the literature [21,30–33]. Table 2 includes the signals found in the mass spectra of the phenolic compounds present in the sweet potato samples.

Table 2. Phenolic compounds identified by UHPLC-QToF-MS and their presence in the different sweet potato varieties studied.

Compound	Molecular Ion (m/z) [M-H]-	Main Ion Fragments (Relative Intensity %)	Var. California	Var. Beauregard	Var. Covington
5-*O*-Caffeoylquinic acid	353	191 (100)	+ [1]	+	+
4-*O*-Caffeoylquinic acid	353	191 (20), 173 (100)	+	+	+
3-*O*-Caffeoylquinic acid	353	191 (100), 179 (44)	+	+	− [2]
3,4-Dicaffeoylquinic acid	515	353 (100), 191 (2), 179 (6)	+	+	+
3,5-Dicaffeoylquinic acid	515	353 (100), 191 (9)	+	+	+
4,5-Dicaffeoylquinic acid	515	353 (100), 173 (5)	+	+	+

[1] + Indicates the presence of the compound.; [2] − Indicates the absence of the compound.

3.2. Optimization of the Ultrasound-Assisted Extraction Method

For the optimisation of the method by means of the Box-Behnken design, the factors that most frequently affect the efficiency of ultrasound-assisted extraction were selected, such as temperature (10–60 °C), solvent composition (0–100% MeOH in water) and sample-to-solvent ratio (0.5:10–1:10 g/mL). According to our research group's previous experience with these type of compounds in plant matrices, the extractions were performed by setting the probe amplitude to 33% of its maximum value (200 W), the cycle was setup at 0.5 pulses per second and 10 min was the extraction time established.

Following the Box-Behnken design for the three experimental variables that had been selected, 15 experiments were conducted in duplicate, including three central points. This would allow for the determination of the optimal extraction conditions according to the effect of each of the main variables as well as that of these variables in combination.

Table 3 shows the experiments that were performed, and the chromatographic re-

sponse obtained, expressed as the area of the chromatographic peaks measured at 320 nm per gram of the extracted sample (relative area), which is directly related to the concentration of the compounds analysed in the extract in each of these experiments.

Table 3. Box-Behnken design of the experiments for three factors and responses (n = 2) observed.

Experiment	Temperature (°C)	Solvent (% MeOH)	Ratio (g/10 mL Solvent)	Observed Relative Area	Predicted Relative Area
1	35	50	0.75	542,767	480,007
2	60	0	0.75	398,138	410,201
3	10	0	0.75	280,807	284,828
4	35	100	0.50	542,409	554,472
5	60	50	0.50	549,945	50,867
6	10	50	1.00	433,755	432,885
7	60	100	0.75	556,268	532,143
8	35	0	1.00	302,029	293,987
9	10	100	0.75	501,395	501,395
10	10	50	1.00	436,036	432,885
11	35	50	0.75	465,321	480,007
12	35	100	1.00	484,902	496,965
13	60	50	0.50	471,423	508,674
14	35	50	0.75	423,891	480,007
15	35	0	0.50	461,722	453,680

An Analysis of variance (ANOVA) was carried out to assess the effect of each variable both separately and as the result of the interactions between them. The equation of the model obtained for optimisation by the UAE is as follows:

Relative Area = 643,130 − 189 temperature + 1874 solvent – 502,236 ratio − 14.5 temperature × temperature − 13.6 solvent × solvent + 72,616 ratio × ratio − 12.5 temperature × solvent + 3050 temperature × ratio + 2044 solvent × ratio.

Analysis of variance (ANOVA, p = 0.05) was used to determine the degree of significance of each variable considered. The developed model showed a high correlation between the actual values and those obtained by the model, with a correlation coefficient of 88%. Figure 3 shows the principal component graph for each of the factors, where it can be seen that the extraction solvent is the most influential variable.

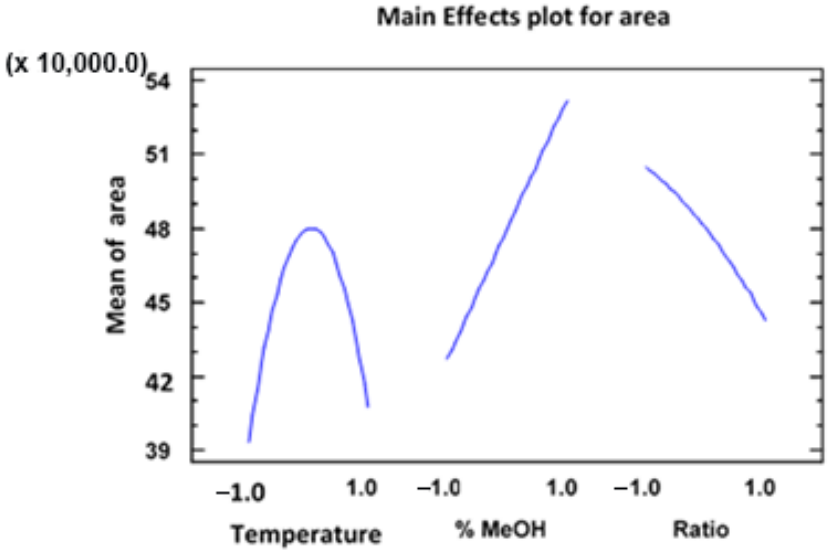

Figure 3. Principal component concentration curve according to each variable.

Based on the BBD analysis, the optimal values obtained for the maximum extraction of the caffeoylquinic acids in sweet potato were: 39.4 °C, 100% MeOH extraction solvent and 0.5 g of sample per 10 mL of solvent.

It was observed that the optimal extraction value for the % MeOH in the extraction solvent is at the upper limit of the studied range, while the sample/solvent ratio it is at the lower limit of the studied range. No additional experiments beyond these ranges were required since, since in the case of the solvent, the percentage of MeOH could not be increased, while a lower ratio, i.e., a smaller amount of sample, would not produce a minimal valid signal from the chromatographic analysis of the phenolic compounds by UHPLC.

3.3. Validation of the Extraction Method

In order to validate the ultrasound-assisted extraction method, a series of extractions were carried out to determine the repeatability (n = 9) and intermediate precision (n = 6 + 6 + 6) under optimal extraction conditions. Table 4 shows the relative standard deviation (%) values obtained.

Table 4. Repeatability (n = 9) and intermediate precision (n = 6 + 6 + 6) of the ultrasound-assisted extraction method.

Compound	5-*O*-Caffeoylquinic Acid	4-*O*-Caffeoylquinic Acid	3,4-Dicaffeoylquinic Acid	3,5-Dicaffeoylquinic Acid	4,5-Dicaffeoylquinic Acid
Repeatability (% RSD)	6.5	6.7	8.3	7.2	8.0
Intermediate precision (% RSD)	6.2	7.0	6.2	6.0	5.3

The results show that in both cases the relative standard deviation stays within the maximum recommended error for those analytes whose concentration levels are in the order of mg/L, which is 10%. The coefficient of variation in the repeatability shows a minimum value of 6.5 for 5-*O*-caffeoylquinic acid and a maximum of 8.3 for 3,4-dicaffeoylquinic acid. Regarding the intermediate precision, the relative standard deviation ranged from a minimum of 5.3 for 4,5-dicaffeoylquinic acid and a maximum of 7.0 corresponding to 4-*O*-caffeoylquinic acid.

3.4. Implementation of the Method to Actual Samples

Once the optimal conditions for the extraction of the major phenolic compounds present in sweet potatoes were defined, different types of cooking methods were applied to the three sweet potato varieties (Covington, California and Beauregard) so as to evaluate the effect of cooking on the phenolic compound composition of the samples. The experiments consisted of three different types of cooking methods: boiling, oven roasting and microwave processing.

3.4.1. Effect of Cooking Method on Sweet Potato Phenolic Compounds

In this study, the Covington variety was cooked following the three different cooking methods described (boiling, oven roasting and microwave processing). The cooked sample was boiled for 17 min; the oven-roasted sample was boiled for 30 min at 240 °C; and the microwave prepared sample was microwaved for 7 min at 800 W. These three preparations were carried out in duplicate. The effect of each cooking technique on the phenolic compounds present in the sweet potatoes is shown in Figure 4.

According to the results obtained with each of the three different cooking methods, oven-roasted produced the sweet potatoes with the highest content of caffeoylquinic acids. Particularly 5-caffeoylquinic acid appears as the major compound detected after subjecting the sweet potato samples to the three cooking techniques. Temperature and time may be the factors to increase the content of caffeoylquinic acids in the oven-roasted sample, since

a temperature of 240 °C was applied for 30 min in this process (with a considerable water loss through evaporation). In any case, and despite the definite differences, it is surprising that such ample temperature variations are reflected in such narrow deviations in the caffeoylquinic derivative composition. Microwave cooking produced intermediate results, with similar concentration levels to those obtained in oven-roasted sweet potato samples regarding 5-CQA and 3,5-di-CQA derivatives as well as comparable levels regarding the rest of the derivatives found in the rest of the samples after cooking. Boiled sweet potatoes, however, generally present a lower concentration of caffeoylquinic acids, which may be due to the extraction effect of water during cooking.

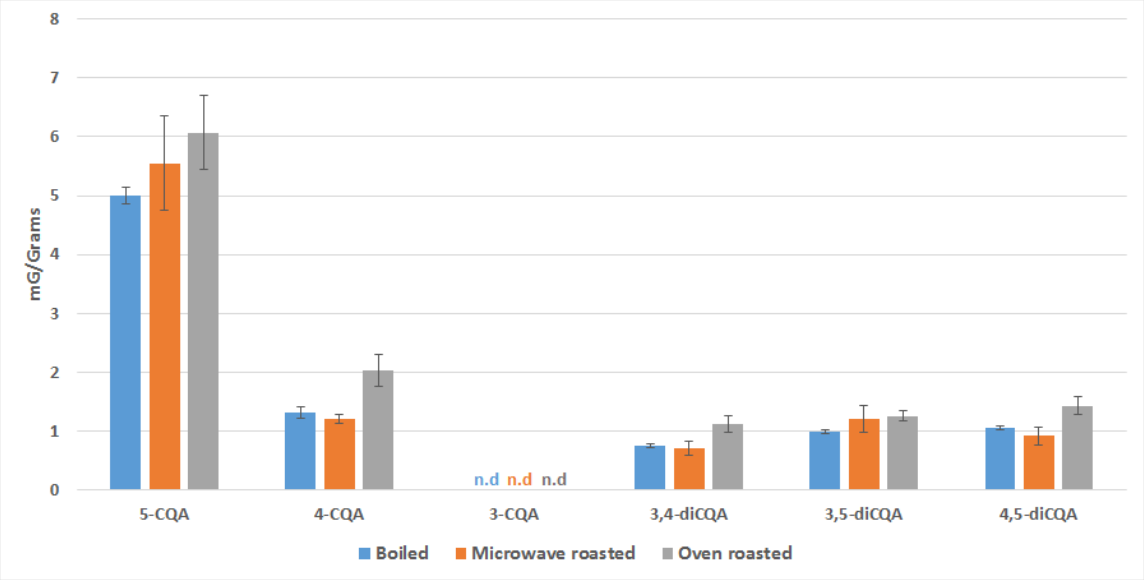

Figure 4. Effect of different cooking methods (n = 2) on the concentration of caffeoylquinic acids.

3.4.2. Effect of Boiling Time and Presence or Absence of Sweet Potato Peel

In this study, uniform 3 cm slices of the California variety of sweet potato with and without skin were cooked in boiling water. The samples were boiled for three different periods of time, 7, 12 and 17 min. Each experiment was carried out in duplicate. Figure 5 shows the results from the cooking experiments of the California sweet potato with skin after the three boiling times indicated.

In view of Figure 5A, a decrease in the different components is observed with increasing boiling time. It is therefore ruled out that the caffeoylquinic derivatives are generated during boiling, while in fact, the opposite effect is being observed with a loss of concentration. Such a concentration drop, at least when cooking in water, can be due either to thermal degradation or to extraction into the boiling water. Figure 5B shows the results of a similar experiment where California sweet potato was boiled without skin.

It can be observed, as in the previous case, that there is a decrease in the components with increasing boiling time. However, the levels of caffeoylquinic acids recovered after 7 min of boiling are much lower than those obtained when boiled with skin. This may be due to the protective effect of the skin against the extraction of the compounds into the boiling water.

The cluster analysis (Figure 5C) shows that 5-CQA was the compound with a different behaviour under the cooking conditions. It must be noted that the concentration of 5-CQA decreased 60% if cooked with skin, however it was only 20% lower if cooked without skin for 17 min. It must be noted that the lower differences were due to a much lower concentration after 7 min of boiling time (4.2 mg/g to 1.5 mg/g of 5-CQA after 7 min of boiling time). It does not matter the cooking time; boiling the sweet potatoes without skin (Figure 5B) produced lower levels of the caffeoylquinic acids. No important differences

were found for the other compounds cooked with or without skin, but similar reduction after additional cooking periods were obtained, ranging from 30% (4-CQA and 4,5-diCQA) to 60% (3,5-diCQA).

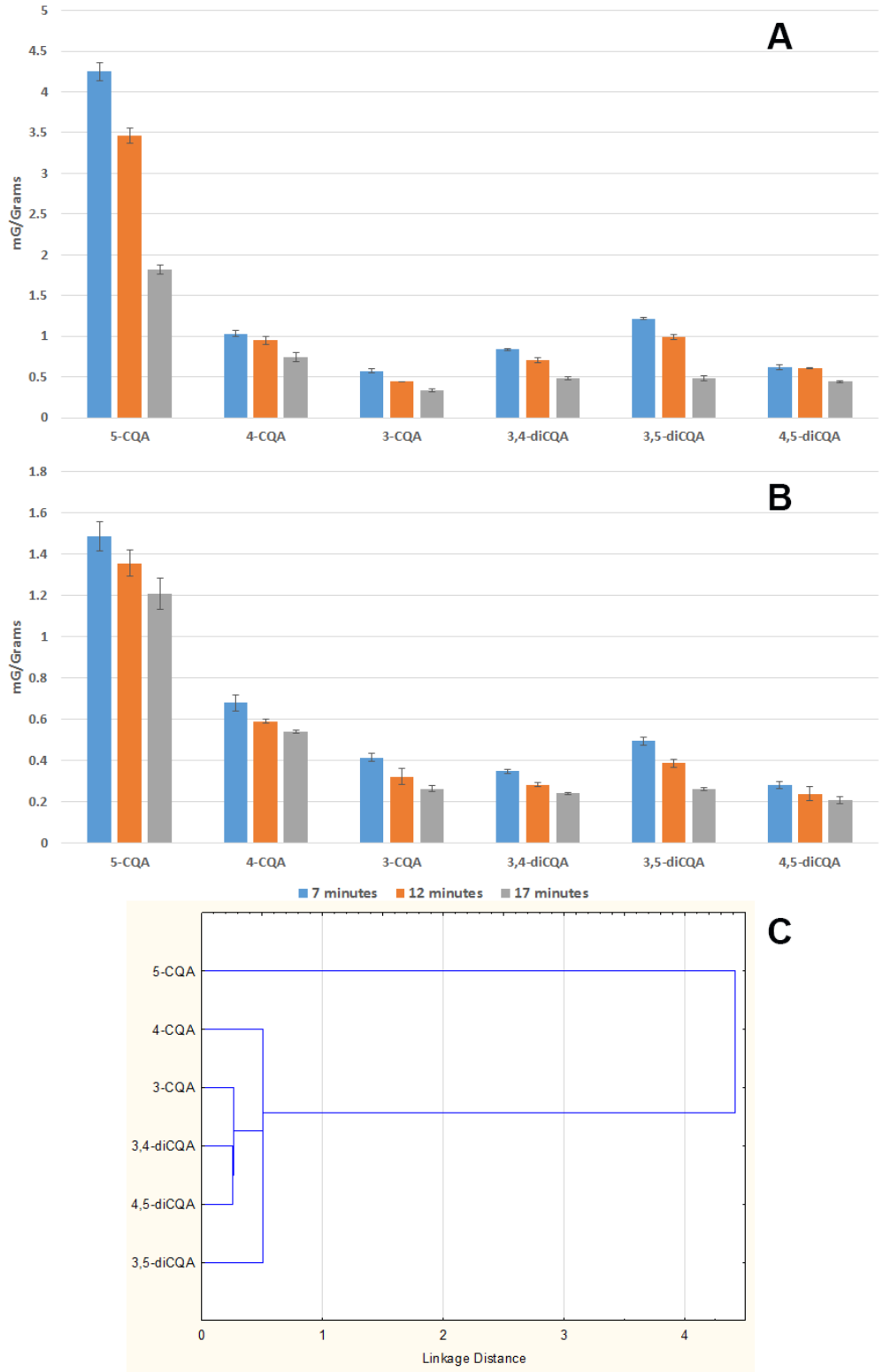

Figure 5. Concentration of caffeoylquinic acids in boiled California sweet potatoes with skin (n = 2) (**A**) and without skin (n = 2) (**B**), after cooking for 7, 12 and 17 min. (**C**) Dendrogram of caffeoylquinic acids obtained from boiled California sweet potatoes with skin (sample A) and without skin (sample B).

In order to further investigate the hypothesis that the skin prevents the extraction and washing out of these components during the cooking process, two experiments were carried out that consisted in boiling 3 cm sweet potato slices with skin and whole sweet potatoes with skin (Figure 6A) and, 3 cm sweet potato slices without skin and whole peeled sweet potatoes (Figure 6B), all of them for 17 min. These two experiments were carried out using the California variety.

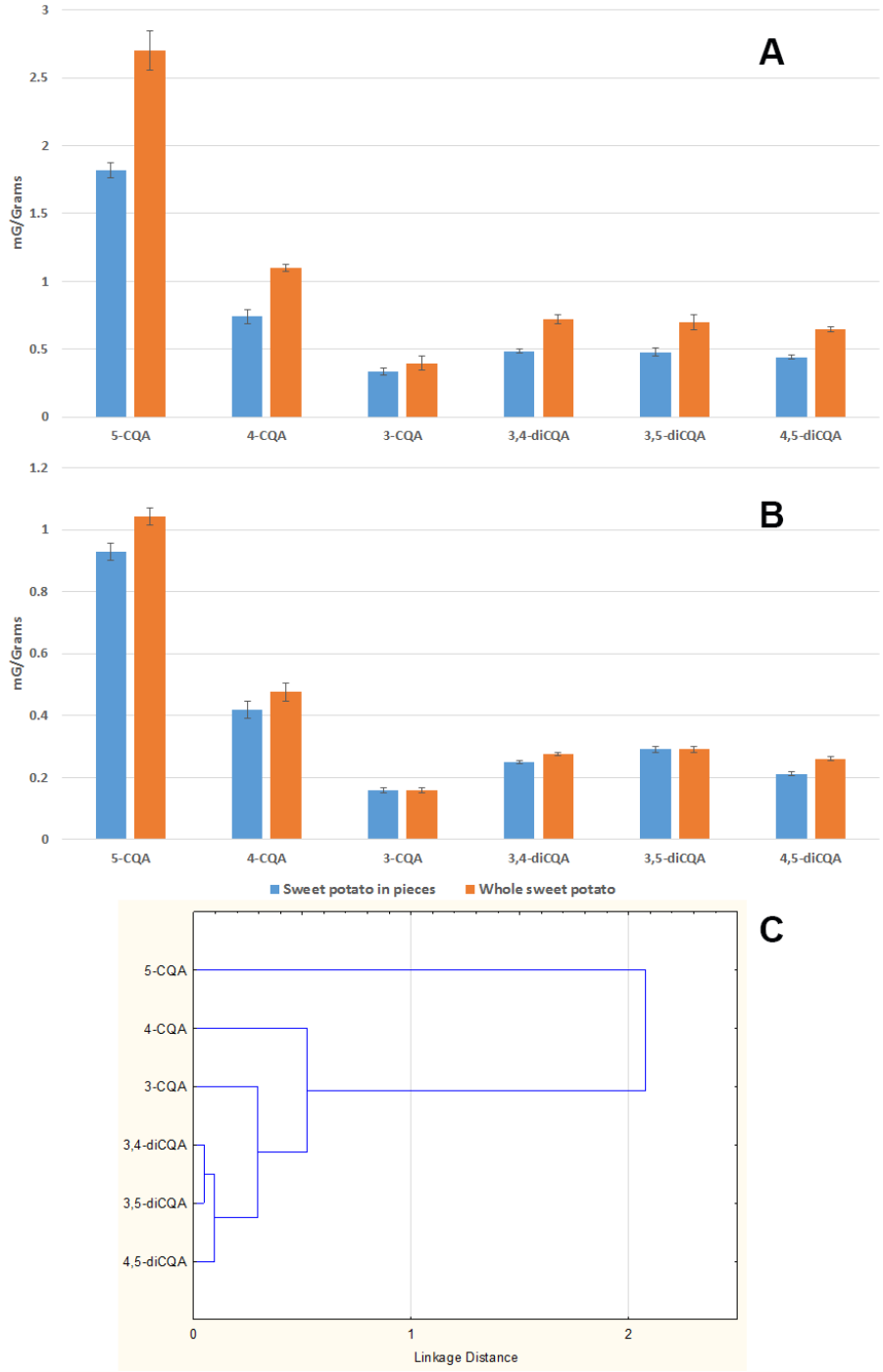

Figure 6. Concentration of caffeoylquinic acids in sweet potatoes of the variety California boiled in pieces vs. boiled whole (n = 2). With skin (**A**), without skin (**B**). (**C**) Dendrogram of caffeoylquinic acids obtained from California sweet potatoes boiled in pieces vs. boiled whole with skin (sample A) and without skin (sample B).

The results show that the whole cooked sweet potato with skin retains more caffeoylquinic acids than the boiled pieces with skin, due to a greater coverage effect of the skin, as it wraps around the whole sweet potato. The levels of caffeoylquinic acids recovered from the skinless pieces and from the whole peeled sweet potato are very similar, due to the absence of the barrier effect provided by the skin.

The cluster analysis (Figure 6C) shows again the different behaviour of 5-CQA. It can be seen that if cooked with skin, a much higher level was found for 5-CQA cooked as a whole piece (Figure 6A). However, rather similar values were found if cooked without skin.

3.4.3. Oven Roasting Time Effect

In this study, sweet potatoes of the California and Beauregard varieties were baked at 240 °C for different lengths of time (30, 35 and 45 min). The baking of these two varieties was conducted in duplicate.

Figure 7A shows the results obtained from the California variety. As the baking time increased, there was an increment in the levels of caffeoylquinic acids in the sample; therefore, this is the opposite effect to the one found when cooking by boiling in water. In this case, there is no extraction by water, but a concentration enhancement of the compounds due to the evaporation of the water.

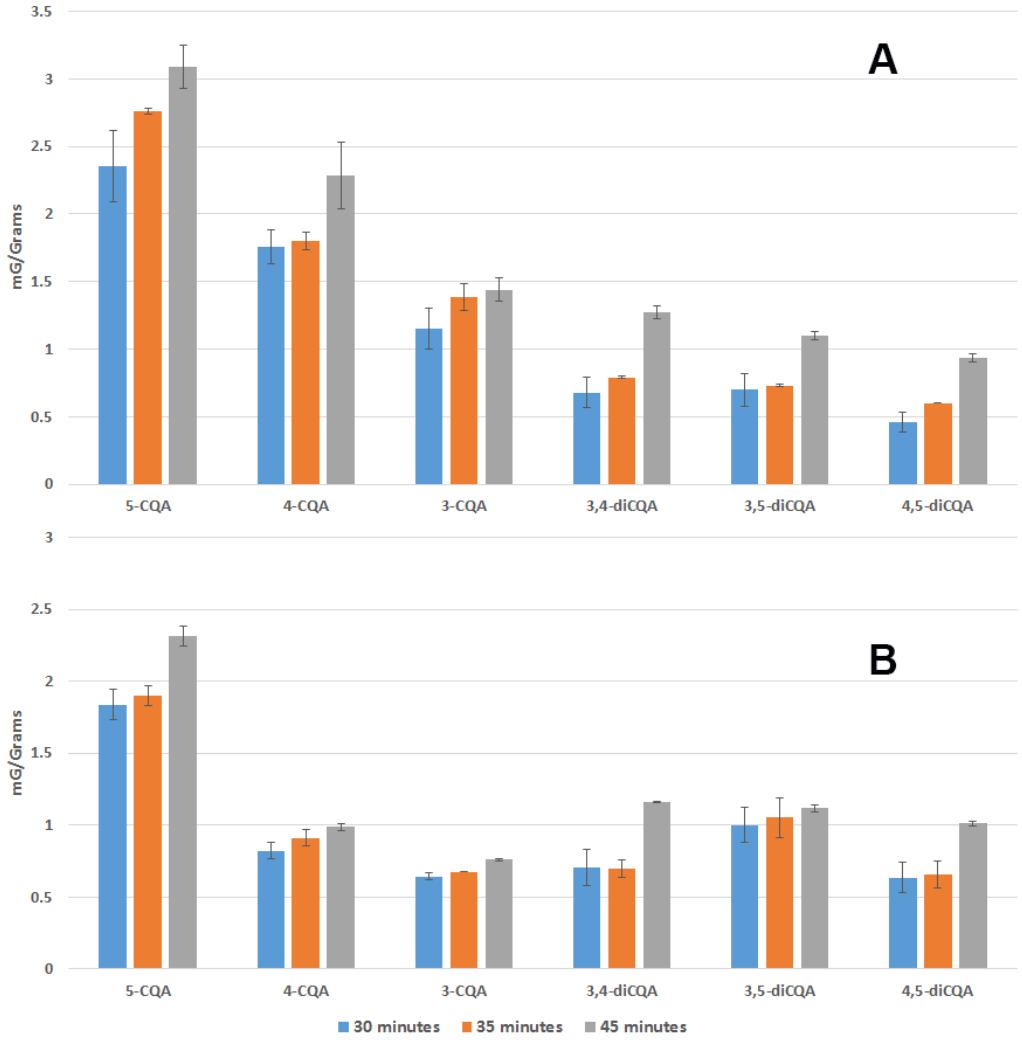

Figure 7. *Cont.*

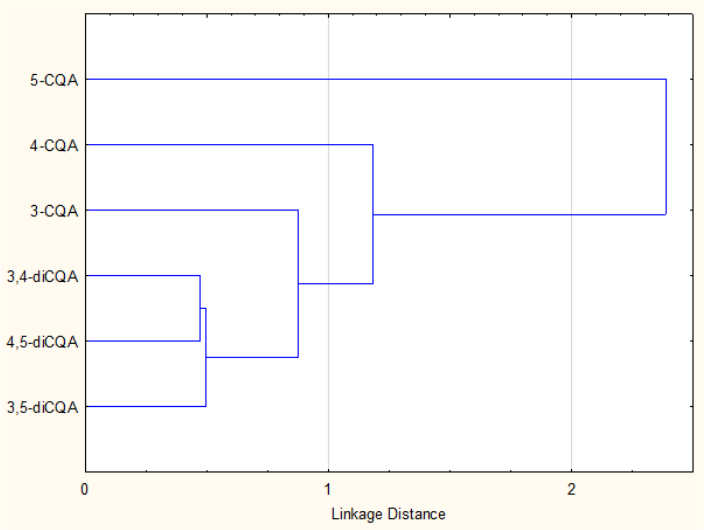

Figure 7. Concentration of caffeoylquinic acids in oven-roasted California sweet potatoes (n = 2) (**A**) and Beauregard sweet potatoes (n = 2) (**B**). (**C**) Dendrogram of caffeoylquinic acids obtained from oven-roasted California sweet potatoes (sample A) and Beauregard sweet potatoes (sample B).

Figure 7B shows the results for the Beauregard variety, where a similar behaviour to that of the California variety is observed, with an increment in caffeoylquinic acid levels as oven time was also increased.

It can also be observed that in both varieties the levels of di-esterified derivatives are similar; however, mono-esterified caffeoylquinic acids show higher levels in the California variety compared with those of the Beauregard variety.

The cluster analysis (Figure 7C) shows a heterogeneous behaviour for the different caffeoylquinic acids. It can be seen that the three monocaffeoyl derivatives had specific differences vs. the three dicaffeoyl derivatives that showed a very similar behaviour.

4. Conclusions

- The method developed for the extraction of sweet potato compounds was proven to be effective to extract caffeoylquinic compounds, with repeatability and intermediate precision properties below 10%;
- The cooking method clearly affects the resulting levels of caffeoylquinic compounds, with oven roasting producing cooked sweet potatoes with higher levels of caffeoylquinic derivatives;
- In the case of water boiling, longer cooking time results in lower levels of caffeoylquinic derivatives, whereas in the case of oven roasting, the opposite effect occurs. An explanation is proposed involving two phenomena with opposite effects, namely extraction by hot water and concentration due to water evaporation at high temperature;
- From the consumer's point of view, the main point to note is that a longer baking time in the oven increases the levels of caffeoylquinic derivatives, while boiling in water reduces them.

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