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# Impact of blanching, sweating and drying operations on pungency, aroma and color of *Piper borbonense*

M. Weil<sup>a,\*</sup>, A. Shum Cheong Sing<sup>b</sup>, J.M. Méot<sup>c</sup>, R. Boulanger<sup>c</sup>, P. Bohuon<sup>d</sup>

<sup>a</sup> CIRAD – UMR Qualisud, Food Process Engineering Research Unit, 7 chemin de l'Irat, 97410 Saint Pierre – BP 180, Reunion

<sup>b</sup> Université de la Réunion – Faculté des Sciences et Technologies Laboratoire de Chimie des Substances Naturelles et des Sciences des Aliments (LCSNSA), 15, avenue René Cassin, Moufia, Reunion

<sup>c</sup> CIRAD, UMR QualiSud, Food Process Engineering Research Unit, TA B-95/16, 73°rue J.F. Breton, F-34398 Montpellier cedex 5, France

<sup>d</sup> Montpellier SupAgro, UMR QualiSud, Food Process Engineering Research Unit, 1101°av. Agropolis, B.P. 5098, F-34093 Montpellier cedex 5, France

## ABSTRACT

Low pungency, high aromatic potential and red color, give to *Piper borbonense* its originality when compared to *Piper nigrum*. Effects of blanching, sweating and drying on these characteristics were assessed. The three operations had no impact on the concentration of piperine and essential oil but affected the composition of essential oil slightly and considerably affected the color of the pepper. The “wet process”, including blanching, sweating and drying, had the largest impact on the composition of aroma, increasing para-cymene content by 89% and reducing safrole content by 33% in dried pepper compared to fresh. Blanching increased the drying rate thus reducing drying time. Drying had a major impact on color, which changed from red to brown. The biggest differences observed led to reductions of 2.2, 7.9 and 8.4 units in  $L^*$ ,  $a^*$  and  $b^*$  values, when chromatic values measured in fresh pepper were compared to those of dried pepper.

## 1. Introduction

Pepper (*Piper* spp.) is the most common spice worldwide; 472 500 tons were produced in 2013 (FAO Statistics Division., 2015). Although, also known for its medicinal properties (Ahmad et al., 2012), pepper is mainly used to enhance the taste and flavor of food. The quality of pepper as a spice is measured throughout pungency, aroma and color (Gu, Tan, Wu, Fang, & Wang, 2013). Although more than 700 species grow in tropical and subtropical regions, most of which are wild (Sumathykutty, Rao, Padmakumari, & Narayanan, 1999), one single domesticated species – *Piper nigrum* is by far the most widely consumed. A wild pepper, named *Piper borbonense* grows in Reunion Island but has not been collected until now. Some very closely related wild species of pepper, local name Tsiperifery, grow in Madagascar and are picked for both local consumption and for sale, including for export. These wild peppers differ from domesticated *Piper nigrum* in their low piperine content, high essential oil content and particular red color (Weil et al., 2014). Although they are sold at high prices in Europe, these

Malagasy peppers are of heterogeneous quality which could affect their reputation and valorization. As pepper quality varies with the species, origin, agricultural system (when domesticated), climate, or maturity, it may also be influenced by postharvest treatments. Dhas and Korikanthimath (2003) described the different types of processing of pepper and the advantages of each, but few studies have focused on the impacts of processing on pepper quality. Existing studies generally tested domestic cooking, and reported contradictory results. Wild pepper is currently not processed in Reunion Island. In Madagascar, wild peppers are processed according to “dry” and “wet” processes (Weil et al., 2014). The “dry” process only consists in drying, whereas the “wet” process includes blanching and sweating prior to drying. Traditionally, sweating, i.e. keeping the hot blanched product in a blanket for 24 h is widely used in the treatment of Malagasy vanilla beans. However, it is not used elsewhere on pepper and not described either in the literature. The objective of our study was thus to assess the impact of blanching, sweating and drying in controlled conditions on the quality of wild *Piper borbonense* pepper originating from Reunion Island. The quality characteristics considered in this study were pungency (piperine content), aroma (essential oil content and composition) and color (judged by eye and through  $L^*$ ,  $a^*$  and  $b^*$  chromatic values). The influence of blanching and sweating on drying kinetics was also assessed.

\* Corresponding author.

E-mail addresses: mathieu.weil@cirad.fr (M. Weil), alain.shum@univ-reunion.fr (A. Shum Cheong Sing), jean-michel.meot@cirad.fr (J.M. Méot), renaud.boulanger@cirad.fr (R. Boulanger), philippe.bohuon@supagro.fr (P. Bohuon).

## 2. Materials and methods

### 2.1. Plant material

We defined three maturity stages according to pepper color: A (immature green pepper), B (orange pepper – intermediate maturity) and C (red mature pepper). Wild mature (C) pepper spikes were picked in the south of Reunion Island. Spikes picked on different occasions were frozen at  $-80\text{ }^{\circ}\text{C}$  (freezer Froilabo – Bio Memory, 690 L) before being pooled and mixed to form a single homogenous batch. Before processing, the peppercorns with their peduncles were separated from the fruit stems by hand and defrosted for two hours at room temperature. The defrosted pepper is called “fresh” pepper in the rest of this article.

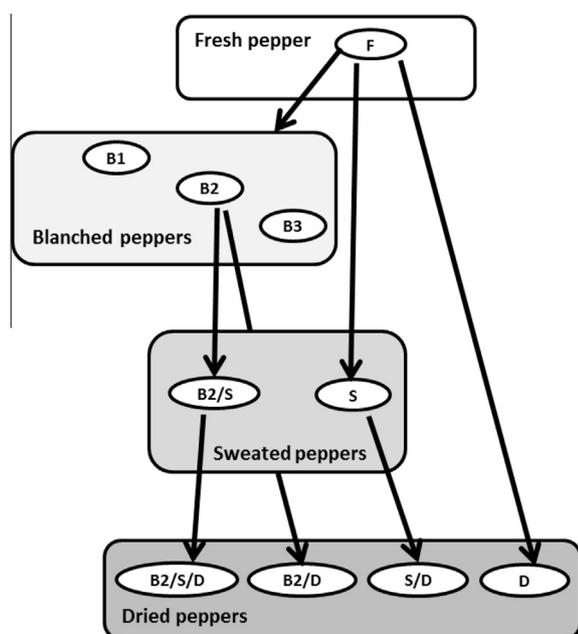
### 2.2. Processing experiments

#### 2.2.1. Blanching, sweating and drying

The processes consisted in three unit operations that were applied (alone or combined) to obtain different samples (Fig. 1). F: fresh pepper; B: blanched (B1:  $60\text{ }^{\circ}\text{C}/30\text{ s}$ ; B2:  $75\text{ }^{\circ}\text{C}/180\text{ s}$ ; B3:  $100\text{ }^{\circ}\text{C}/300\text{ s}$ ); S: sweated ( $35\text{ }^{\circ}\text{C}$ , 99% RH, 24 h); D: dried ( $60\text{ }^{\circ}\text{C}$ , 20% RH, 39 h). Blanching consisted in soaking the peppercorns in a hot water bath (Mettmert GmbH type WB 22 Schwabach, Germany) at a ratio of 1:36 peppercorns to water in three different conditions: at  $60\text{ }^{\circ}\text{C}$  for 30 s;  $75\text{ }^{\circ}\text{C}/180\text{ s}$ ; and  $100\text{ }^{\circ}\text{C}/300\text{ s}$ . Sweating consisted of storing the peppercorns in a climatic chamber (BIA Climatic – Type CL 125, Conflans Sainte Honorine, France) at  $35\text{ }^{\circ}\text{C}$  and 99% RH for 24 h. Drying was performed by placing aluminum trays ( $300\text{ cm}^2$ ) containing 250 g of peppercorns arranged in a compact 1 cm thick layer for 39 h at  $60\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ , RH  $20\% \pm 2\%$  in the same climatic chamber. Hot air ( $60 \pm 1\text{ }^{\circ}\text{C}$ , RH  $20 \pm 2\%$ ) was circulated over the surface of the layer.

#### 2.2.2. Drying kinetics

Drying of peppercorn samples used a cross flow pilot dryer, developed in our laboratory. In the treatment chamber ( $0.25\text{ m long} \times 0.25\text{ m wide} \times 0.92\text{ m high}$ ), 150 g of peppercorns were



**Fig. 1.** Processes applied to pepper F: fresh pepper; B: blanched (B1:  $60\text{ }^{\circ}\text{C}/30\text{ s}$ ; B2:  $75\text{ }^{\circ}\text{C}/180\text{ s}$ ; B3:  $100\text{ }^{\circ}\text{C}/300\text{ s}$ ); S: sweated ( $35\text{ }^{\circ}\text{C}$ , 99% RH, 24 h); D: dried ( $60\text{ }^{\circ}\text{C}$ , 20% RH, 39 h).

placed on a sieve ( $0.25 \times 0.25\text{ m}^2$ ) in a single thin non-compact layer. Hot air ( $60 \pm 1\text{ }^{\circ}\text{C}$ , RH  $20 \pm 2\%$ ) was circulated downwards at  $2.7 \pm 0.1\text{ ms}^{-1}$  through the layer of peppercorns by a high-capacity fan. The air velocity was just high enough to have no significant effect on temperature when passing through the layer of peppercorns to ensure a proper treatment, and to enable statistical analyses. When the heat treatment was complete, the peppercorns were cooled by ventilation with air at ambient temperature. The water content, which was measured on a dry basis (noted  $X$ ) as a function of time, was estimated in line, using the mass reading of the sieve. Water content kinetics  $X^{(t)}$  were fitted with a cubic smoothing spline (Matlab<sup>®</sup> Version 5.2, The Mathworks Inc., USA). The drying rate ( $dX/dt$ ) was calculated as the direct analytical derivative of the cubic smoothing spline function on  $X^{(t)}$ .

### 2.3. Sample preparation

The samples resulting from the different processing operations were frozen at  $-80\text{ }^{\circ}\text{C}$  for further preparation and analysis. The pepper samples were ground for 10 s at 10,000 rpm in mill (Retsch – Grindomix GM200, Retsch GmbH, Germany) for all analyses except color which was measured on whole peppercorns.

### 2.4. Analytical methods

#### 2.4.1. Dry matter content

The dry matter content (mean “essential oil free dry matter”) was obtained by drying 5 g of ground pepper in an aluminum cup in the oven (ULE 400, Mettmert GmbH, Germany) at  $105\text{ }^{\circ}\text{C}$  for 30 h (i.e., until constant weight). Initial and final mass was determined with a precision balance (Scaltec SBC 22 model, Scaltec GmbH, Germany). The mean standard deviation of repeatability was  $\pm 0.6\%$  ( $n = 3$ ). Water content expressed on a dry basis was deduced from essential oil and dry matter content.

#### 2.4.2. Piperine content

The piperine content, expressed on a dry basis, was determined according to the spectrophotometric method described in ISO 5564 (International Standard Organization., 1982). The spectrophotometer used was a Thermospectronic Helios  $\alpha$  v4.60 (Thermo Fisher Scientific, USA). The mean relative deviation of repeatability was  $\pm 7.3\%$  ( $n = 3$ ).

#### 2.4.3. Essential oil content

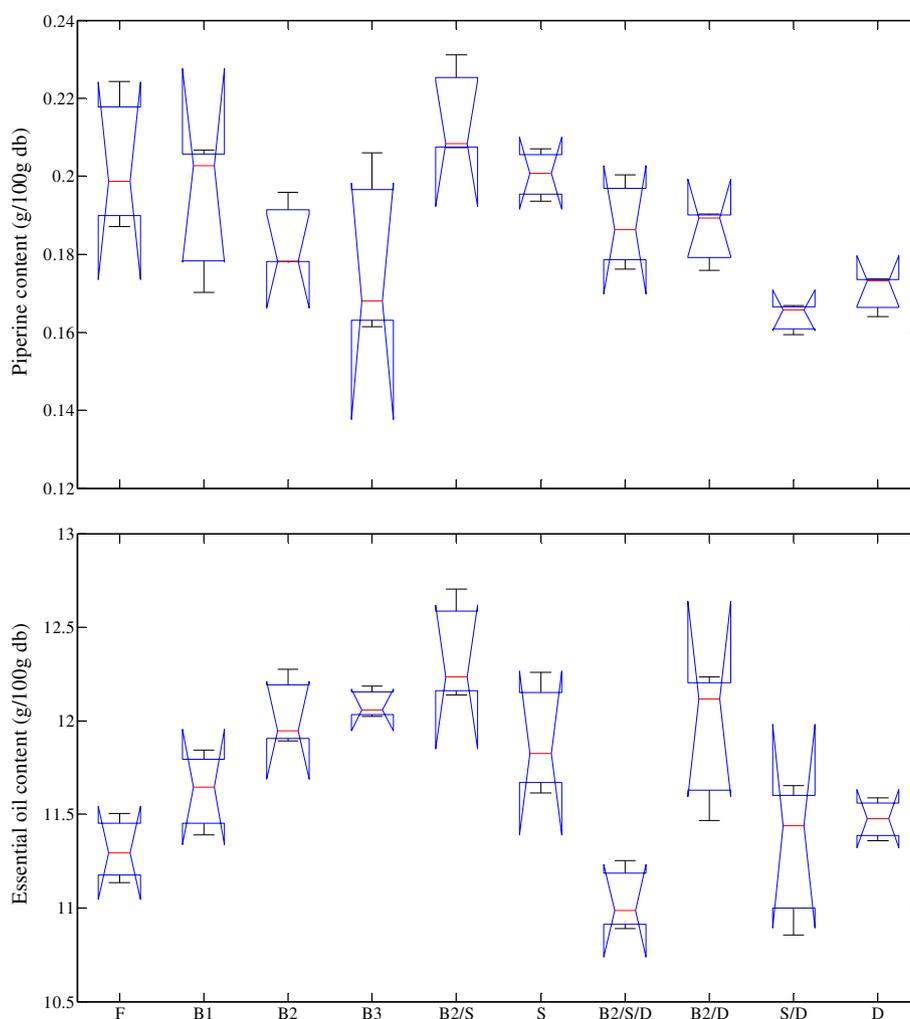
The essential oil content, expressed on a dry basis, was determined using a method adapted from the standard ISO 6571 (International Standard Organization., 2008). One modification in the applied method was the elimination of xylene. The mean relative deviation of repeatability was  $\pm 2.2\%$  ( $n = 3$ ).

#### 2.4.4. Color measurements

Color measurements (CIE  $L^*$ ,  $a^*$  and  $b^*$  values, representing lightness, redness and yellowness, respectively) were made on whole peppercorns using a Minolta CR 400 and utility software. Ten measurements were made on each sample of peppercorns spread in a 1-cm layer in an uncovered Petri dish. The mean relative deviation of repeatability was 1.2%, 2.3% and 3.6% respectively for  $L^*$ ,  $a^*$ ,  $b^*$  ( $n = 10$ ).

#### 2.4.5. Identification and quantification of essential oil compounds

**2.4.5.1. Separation on a polar column.** Volatile compounds were analyzed on a GC (HP 6890), equipped with a Supelco-Wax polar column (Supelco  $-60\text{ m} \times 320\text{ }\mu\text{m} \times 0.25\text{ }\mu\text{m}$ ) coupled to a MS detector. Aliquots ( $0.1\text{ }\mu\text{L}$ ) of concentrated essential oil (obtained as described in Section 2.4.3. above) were injected into the GC-MS in split mode (1:30). The injector’s temperature was  $250\text{ }^{\circ}\text{C}$ .



**Fig. 2.** Impact of processing on piperine and essential oil contents F: fresh pepper; B: blanched (B1: 60 °C/30 s; B2: 75 °C/180 s; B3: 100 °C/300 s); S: sweated (35 °C, 99% RH, 24 h); D: dried (60 °C, 20% RH, 39 h). Boxplots provide a statistic test of group medians: the line in the middle of each box is the sample median ( $n = 3$ ); the tops and bottoms of each box are the 25th and 75th percentiles of the samples.

The temperature of the transfer line was 250 °C and the flow rate of the gas carrier (Helium) was 0.8 mL/min. The temperature program was as follows: initial temperature 60 °C, heating rate of 4 °C/min until a final temperature of 230 °C was reached and maintained constant for 20 min. The molecules were identified using a GC/MS (HP 6890) which functions in electron impact (70 eV) mode. The mass range was between 25 and 350  $m/z$ .

**2.4.5.2. Separation on a non-polar column.** Volatile compounds were analyzed with a GC (HP 6890), equipped with a SPB-5 non-polar column (Supelco  $-60\text{ m} \times 320\text{ }\mu\text{m} \times 0.25\text{ }\mu\text{m}$ ) coupled to a MS detector. Aliquots (0.2  $\mu\text{L}$ ) of concentrated essential oil (obtained as described in Section 2.4.3 above) were injected into the GC-MS in split mode (1:50). The injector's temperature was 250 °C. The temperature of the transfer line was 250 °C and the flow rate of the gas carrier (Helium) was 0.7 mL/min. The temperature program was as follows: initial temperature 60 °C, heating rate of 4 °C/min until final temperature of 250 °C was reached then maintained constant for 50 min. The molecules were identified using a GC/MS (HP 6890) which functions in electron impact (70 eV) mode. The mass range was between 20 and 400  $m/z$ .

**2.4.5.3. Identification.** The aromatic compounds separated on the two columns, were identified by comparing their mass spectrum

to those available in commercial libraries (NIST02, WILEY) or constituted under our care and by comparison of their retention indexes calculated relative to those available in the literature (Adams, 1995; Jennings & Shibamoto, 1980; Kondjoyan & Berdagué, 1996) and Internet databases. (2014).

**2.4.5.4. Quantification on non-polar column.** The aromatic compounds were quantified by a GC (HP 5890), equipped with a SPB-5 non-polar column (Supelco  $-60\text{ m} \times 320\text{ }\mu\text{m} \times 0.25\text{ }\mu\text{m}$ ) coupled to a FID detector. Aliquots (0.3  $\mu\text{L}$ ) of a mixture of concentrated essential oil (obtained as described in Section 2.4.3 above) and internal standard terpinolene (20:2; v/v) were injected into the GC-FID in split mode (1:33). The injector's temperature was 250 °C. The flow rate of the gas carrier (Helium) was 0.7 mL/min. The oven temperature program was as follows: initial temperature 60 °C, rate of 4 °C/min until a final temperature of 250 °C was reached then maintained constant for 20 min. The mean relative deviation of repeatability was  $\pm 2.5\%$  ( $n = 3$ ).

## 2.5. Statistical analysis

Differences in the mean values of piperine content, essential oil content, essential oil composition and  $L^*$ ,  $a^*$  and  $b^*$  values were tested by analysis of variance (ANOVA); the significance of

differences between samples was determined using Fisher's test. The level of significance was  $P < 0.05$ .

### 3. Results

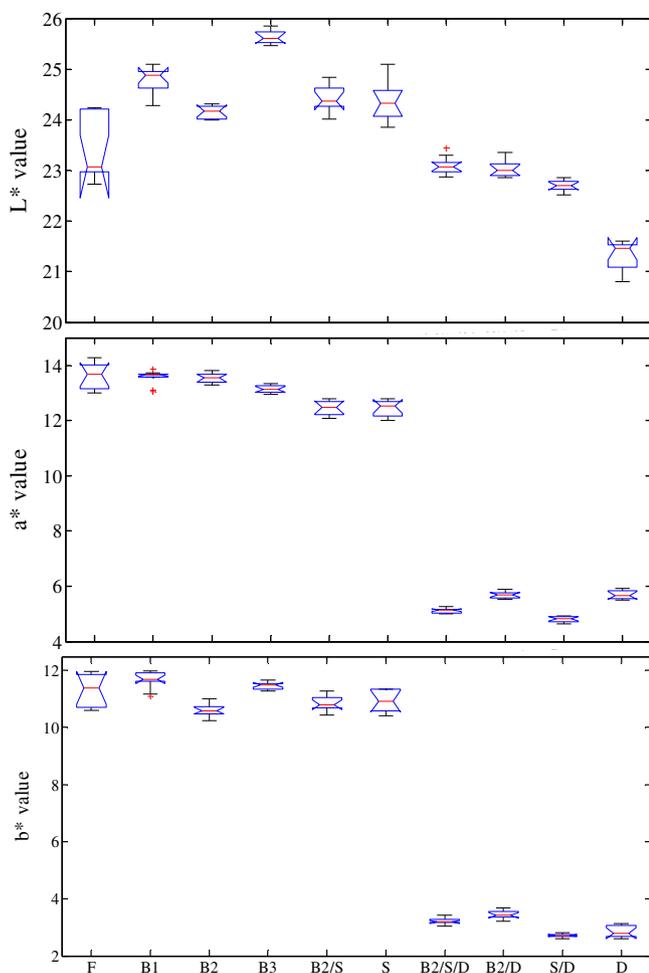
Two levels of observation were considered: unit operations and full processes. In this paper, we considered four full processes: three "wet processes" including blanching and/or sweating and drying and a "dry process" consisting in one drying single operation.

#### 3.1. Impacts of the unit processing operations

Here we describe the impacts of blanching, sweating and drying operations (Fig. 1) on piperine and essential oil contents (Fig. 2), color (Fig. 3) and on drying kinetics (Fig. 4).

##### 3.1.1. Impact of blanching

Blanching had no impact on piperine content (Fig. 2). There was no significant difference in the results obtained (ranging from 0.18% to 0.20%, dry basis) in samples F, B1, B2, and B3. Blanching had no impact on essential oil content (Fig. 2) as evidenced by the absence of a significant difference in the results obtained for essential oil (ranging from 11.31% to 12.09%, dry basis) in the same samples.



**Fig. 3.** Impact of processes on  $L^*$ ,  $a^*$  and  $b^*$  chromatic values F: fresh pepper; B: blanched (B1: 60 °C/30 s; B2: 75 °C/180 s; B3: 100 °C/300 s); S: sweated (35 °C, 99% RH, 24 h); D: dried (60 °C, 20% RH, 39 h). Boxplots provide a statistic test of group medians: the line in the middle of each box is the sample median ( $n = 10$ ); tops and bottoms of each box are the 25th and 75th percentiles of the samples.

Blanching did have a slight impact on color (Fig. 3) as some slight yet significant differences were observed. The  $L^*$  value of sample B3, which was subjected to the most drastic treatment, differed from all the other samples: +2.1 units compared to sample F. The biggest differences in  $a^*$  and  $b^*$  values were respectively lower than 0.5 and 1 in samples F, B1, B2, and B3. Fig. 4 shows the impact of blanching on the drying curves. Two hours were required to obtain a 50% reduction in the initial water content of fresh pepper and 1 h20 min for blanched pepper. The comparison of the drying curves showed a much higher initial drying rate ( $1.44 \pm 0.10 \text{ kg} \cdot \text{kg}^{-1} \cdot \text{h}^{-1}$ ) in blanched pepper than in fresh pepper ( $0.84 \pm 0.02 \text{ kg} \cdot \text{kg}^{-1} \cdot \text{h}^{-1}$ ). The differences between the drying rates were no longer significant when the water content was below  $0.5 \text{ kg} \cdot \text{kg}^{-1} \cdot \text{h}^{-1}$ . This water content was obtained after 4 h in fresh pepper and 3 h15 min in blanched pepper. Blanching greatly increased the drying rate and consequently reduced total drying time.

##### 3.1.2. Impact of sweating

Sweating had no impact on piperine content (Fig. 2). There was no significant difference between sample B2 and sample B2/S on the one hand, and between sample F and sample S on the other hand. Sweating had no impact on essential oil content (Fig. 2) as there was no significant difference between the same samples. However, sweating did have an impact on color (Fig. 3) as sample B2 differed (+1 unit) from sample B2/S in  $a^*$  value, and sample F differed from sample S in  $L^*$  (-0.9 units) and  $a^*$  (+1.2 units) values. Fig. 4 shows the impact of sweating after blanching on the drying curves. One hour twenty minutes was required to obtain a 50% reduction in the initial pepper water content of blanched pepper and 1 h15 min for "blanched plus sweated" pepper. Comparison of the drying curves revealed very similar drying rates in blanched plus sweated pepper ( $1.47 \pm 0.16 \text{ kg} \cdot \text{kg}^{-1} \cdot \text{h}^{-1}$ ) and in blanched pepper ( $1.44 \pm 0.10 \text{ kg} \cdot \text{kg}^{-1} \cdot \text{h}^{-1}$ ). Sweating after blanching did not increase the drying rate; consequently the combined operation did not significantly reduce drying time compared to blanching alone.

##### 3.1.3. Impact of drying

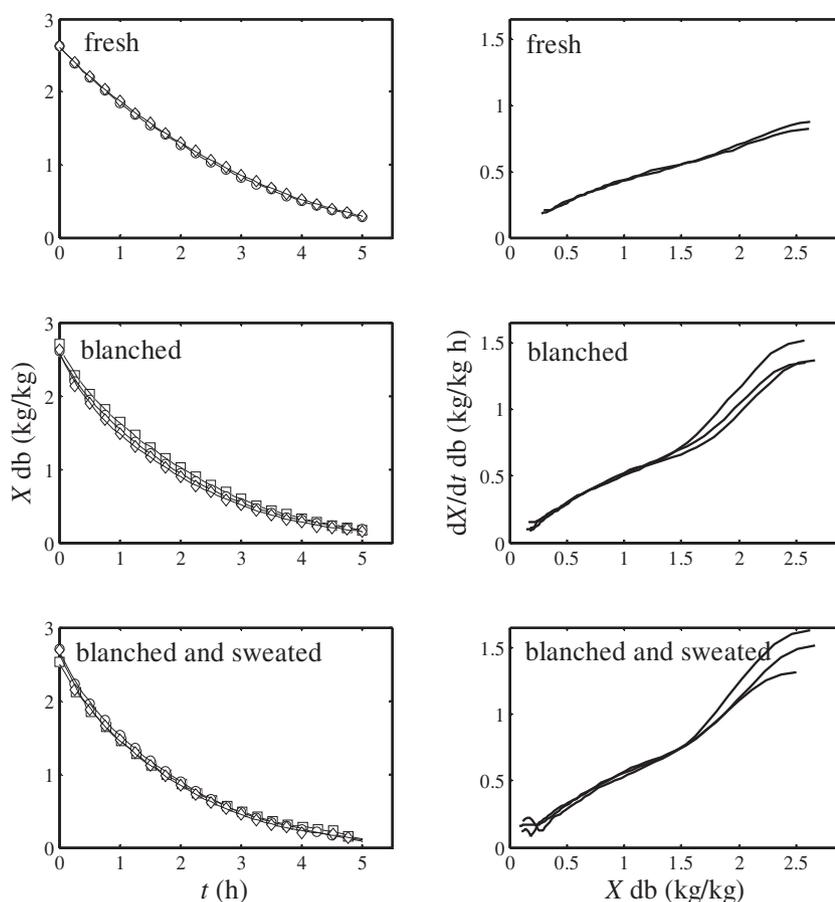
Drying had no impact on piperine content (Fig. 2), as there was no significant difference between samples B2/S and B2/S/D, B2 and B2/D, S and S/D, F and D considered in pairs. Drying had no impact on essential oil content (Fig. 2) as no significant visible difference was observed between the different samples except a slight difference between sample B2/S and sample B2/S/D (relative loss of 11%). Drying did have a marked impact on color (Fig. 3) as there were significant differences in all values ( $L^*$ ,  $a^*$ ,  $b^*$ ) in all dried samples. The greatest differences were observed between samples F and D (Fig. 5) with reductions of 2.2, 7.9 and 8.4 units for  $L^*$ ,  $a^*$  and  $b^*$  values respectively (Fig. 3).

#### 3.2. Impacts of "full" processes

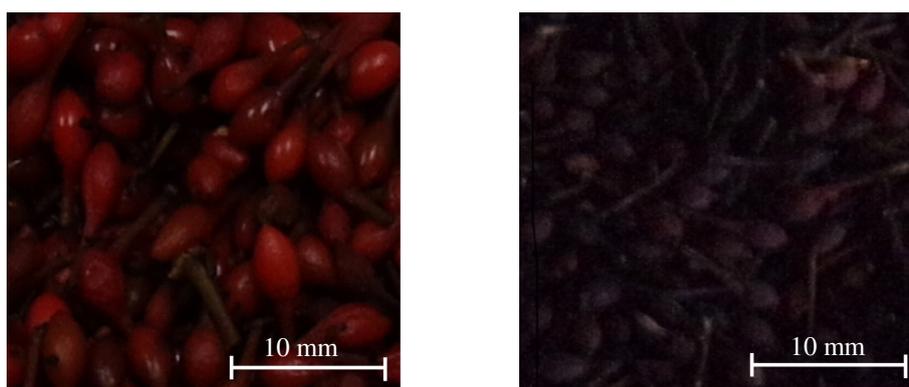
In this study a 'full' process referred either to drying alone (dry process) or a succession of processing operations (wet processes) including blanching and/or sweating before drying (Fig. 1). Here we describe the impacts of these 'full' processes on piperine and essential oil contents (Fig. 2), essential oil composition (Table 1 and Fig. 6), and pepper color (Figs. 3 and 5).

##### 3.2.1. Impact of the "full" processes on piperine and essential oil contents, and on color

None of the "full" processes had an impact on piperine content or on essential oil content (Fig. 2). There was no significant difference between samples F, B2/S/D, B2/D, S/D and D. However, "full" processes did have a marked impact on color, confirming the major impact of the drying operation on all chromatic values (Fig. 3). The



**Fig. 4.** Drying curves of fresh, blanched and blanched/sweated pepper. Water content ( $X$ ) on a dry basis as a function of time ( $t$ ) and drying rate ( $dX/dt$ ) as a function of  $X$ . Drying curves of a “single thin non-compact layer” recorded by an air dryer ( $60\text{ }^{\circ}\text{C}$ , RH 20%, air velocity  $2.7 \pm 0.1\text{ ms}^{-1}$ ). Blanching ( $75\text{ }^{\circ}\text{C}/180\text{ s}$ ); Sweating ( $35\text{ }^{\circ}\text{C}$ , 99% RH, 24 h). The different symbols on curves correspond to different trials.



**Fig. 5.** Impact of drying on the color of the pepper Sample F (fresh pepper); Sample D (dried pepper). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

chromatic dimensions  $L^*$ ,  $a^*$  and  $b^*$  were impacted irrespective of the operation considered concerned, as clearly shown by comparing the values obtained for fresh pepper (sample F) to values obtained for processed peppers (samples B2/S/D, B2/D, S/D and D) or by comparing the values obtained for processed-peppers among themselves. When drying was preceded by blanching and/or sweating, the  $L^*$  value decreased less than during drying alone ( $\leq 0.8$  compared to 2.2 units). The greatest differences (detailed in Section 3.1.3.), mostly due to drying, were observed between sample F and sample D (Fig. 5) and between F and S/D while small

but significant differences (all  $< 1$  unit) were observed for  $a^*$  and  $b^*$  values between samples B2/S/D, B2/D, S/D and D.

### 3.2.2. Impact of the “full” processes on aromatic composition

Taking sample F (fresh pepper) as a reference, we were able to identify 25 aromatic compounds representing 97% (m/m) of the total essential oil. Among these, 15 major compounds, all of which were present at a rate of more than 1%, represented 93% (Table 1) of the total essential oil. The monoterpene family represented 55% of the total aromatic compounds. Limonene (27% of the total),

**Table 1**  
Major volatile compounds in *Piper borbonense* fresh pepper (F) essential oil.

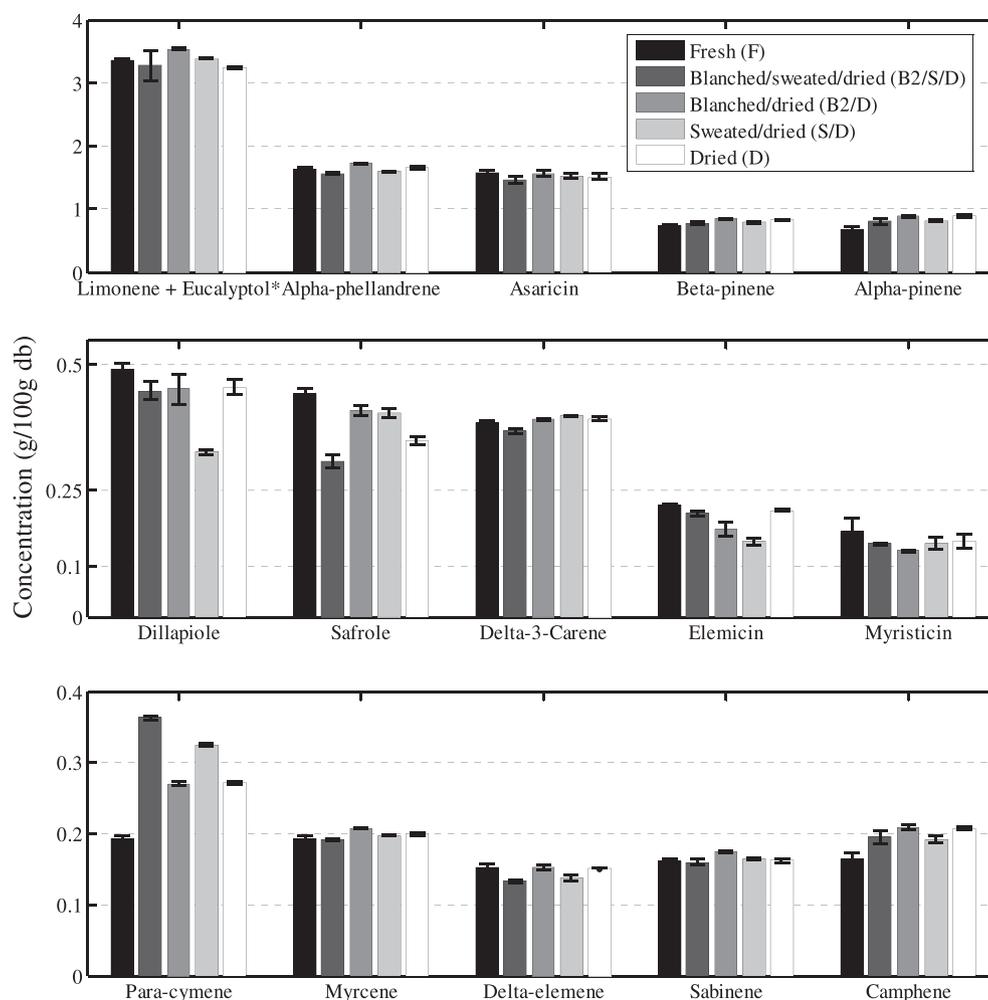
Aromatic compounds	Concentrations (g/100 g)
Limonene + Eucalyptol*	29.54 ± 0.05
Alpha-phellandrene	14.38 ± 0.09
Asaricin	13.94 ± 0.33
Beta-pinene	6.46 ± 0.17
Alpha-pinene	6.00 ± 0.36
Dillapiole	4.32 ± 0.13
Safrole	3.89 ± 0.09
Delta-3-Carene	3.39 ± 0.03
Elemicin	1.95 ± 0.05
Myristicin	1.49 ± 0.24
para-cymene	1.70 ± 0.01
Myrcene	1.70 ± 0.01
Delta-elemene	1.35 ± 0.04
Sabinene	1.41 ± 0.03
Camphene	1.45 ± 0.07
Total	92.98 ± 0.12

Mean values ( $n = 3$ ).

\* Eucalyptol represents around 2.5 g/100 g of essential oil.

was the most abundant compound followed by alpha phellandrene and asaricin (14% each). As the amounts of the compounds in sample F (fresh pepper) were very close to the amounts of compounds in samples B2/S/D, B2/D, S/D or D (processed peppers), we can conclude that globally, the composition of the flavor of the pepper was

little affected by any of the processes (Fig. 6). Nevertheless, two monoterpenes (para-cymene and camphene) were found at higher concentrations in processed peppers (samples B2/S/D, B2/D, S/D and D) than in fresh pepper (sample F). The most remarkable increase (89%) was obtained for para-cymene between samples F and B2/S/D. Conversely, the concentration of safrole, a non-monoterpenic compound, was lower in processed peppers (samples B2/S/D, B2/D, S/D and D) than in fresh pepper (sample F). The most remarkable decrease (33%) for safrole was observed in sample F versus sample B2/S/D. The least affected compounds were delta 3-carene, myrcene and sabinene; no differences of more than 9% in these compounds were found between fresh (sample F) and processed peppers (samples B2/S/D, B2/D, S/D and D). The impacts of single processing operations could be deduced from “full” processes comparison. For example, the concentrations of some monoterpenes (alpha phellandrene, beta pinene and delta-elemene) were reduced by sweating, as shown by the values obtained for samples B2/D and B2/S/D (this sample including sweating) on the one hand and for samples D and S/D (this sample including sweating) on the other hand. The most remarkable significant difference (a drop of 13%) was observed for delta-elemene between samples B2/D and B2/S/D. The aromatic profiles of sample B2/S/D, which included blanching, sweating and drying (full wet process), and sample D (dry process), which only underwent single drying, were very similar. No significant differences were found in 13 out of 15 compounds, while significant



**Fig. 6.** Composition of essential oil of fresh and processed pepper corns. The process conditions were: blanching (75 °C/3 min), sweating (35 °C, 90% RH, 24 h) and drying (60 °C, 20% RH, 39 h). The error bars represent the standard error ( $n = 3$ ). \*Eucalyptol represents 0.2 to 0.3 g/100 g (db) of pepper.

differences were found for two compounds, alpha phellandrene and para-cymene, which was 25% higher in sample B2/S/D than in sample D.

## 4. Discussion

### 4.1. Originality of *Piper borbonense* composition

The 11.3% (db) rate of essential oil in fresh *Piper borbonense* is more than 5 times higher than the 2% content recommended by standard ISO 959-1 (International Standard Organization, 1998) for black pepper. It is also very close to the highest concentration of 13.1% (db) found in the richest Malagasy wild pepper species, local name Tsiperifery (Weil et al., 2014). The rate of 0.20% (db) piperine obtained in fresh *Piper borbonense* is 20 times lower than the value of 4% indicated by the same standard. It is also 2.5 times lower than the lowest concentration of 0.5% found in Malagasy wild pepper species. These low pungency and high aroma values give *Piper borbonense* its originality compared to cultivated black pepper (*Piper nigrum*) and Malagasy wild peppers.

### 4.2. Impact of the processes on piperine and essential oil contents, essential oil composition and color

The different unit operations and 'full' processes tested had no impact on piperine, nor on essential oil contents of the pepper. The results we obtained for piperine are in agreement with those of Nisha, Singhal, and Pandit (2009) but not with those reported by Suresh, Manjunatha, and Srinivasan (2007). Nisha et al. (2009) reported piperine to be stable to heat processing, with only 2.5% loss after 20 min at 100 °C, whereas Suresh et al. (2007) reported 28% losses of piperine in black pepper in the same conditions. Our results concerning essential oil differ from those of Nisha et al. (2009) who reported a 38% reduction in the essential oil content after 20 min at 100 °C, and from those of Schweiggert, Mix, Schieber, and Carle (2005) who reported a 75% loss after 10 min at 90 °C. These differences could be explained by the fact that both Nisha and Suresh used ground pepper and applied blanching conditions that were more drastic than ours. The fact that we observed no drop in piperine and essential oil contents in our study, even after 5 min at 100 °C, could be because the pericarp of the peppercorn acts as a barrier against mass transfer.

In our study, Limonene, alpha phellandrene and asaricin were shown to be the most abundant compounds in *Piper borbonense*. These compounds are also present in cultivated black pepper and play a role in the appreciation of its quality. According to Schulz, Baranska, Quilitzsch, Schutze, and Losing (2005) who worked on black pepper, optimum pepper aroma ("top-peppery-note") is obtained if monoterpene (excluding alpha- and beta-pinene) content is high but at the same time, the pinene content is low. As the essential oil analyzed in our study contained 55% of monoterpenoids excluding pinenes, which represented only 13% of the total, we can conclude that the aroma of wild pepper *Piper borbonense* is of good quality and is preserved even after heat treatment. According to Jirovetz, Buchbauer, Ngassoum, and Geissler (2002), limonene,  $\beta$ -pinene,  $\alpha$ -phellandrene,  $\delta$ -carène, asaricin and elemicin give black pepper its characteristic aroma. In our study, each of these compounds represented more than 1% of the total essential oil; together they represented over 65% of the essential oil of wild pepper *Piper borbonense*. According to Jagella and Grosch (1999),  $\alpha$ -pinene,  $\alpha$ -phellandrene, myrcene, and limonene are key odorants in *Piper nigrum*. These four compounds represented 48% of essential oil in *Piper borbonense* in our study. The composition of essential oil as expressed by the relative amounts of the 15 major compounds in the different samples was almost

not affected by any of the processing operations tested. The biggest decrease (33%) we observed for saffrole after the 'full' wet process is in agreement with the results of Farag and Abo-Zeid (1997) who reported a loss of more than 90% after boiling the seeds for 30 min or drying the seeds for 30 min at 70 °C. Saffrol, which is known to be carcinogenic, could have been degraded during blanching by hydroxylation of the dioxolane ring. Our results which unexpectedly showed that, the volatile compounds remained stable during processing, differ from those of Asekun, Grierson, and Afolayan (2007) who analyzed essential oil extracted from *Mentha longifolia* leaves. These authors observed significant differences in the chemical composition of the essential oils obtained using different drying methods. Four monoterpene compounds (alpha pinene, beta pinene, limonene, 1,8-cineole), stable in our study, were all widely affected (increased or reduced) by the three drying methods tested by these authors. In their case, the fact that the essential oil is known to be stored on or near the surface of the leaf may explain this sensitivity. Argyropoulos and Muller (2014), who studied lemon balm (*Melissa Officinalis* L.) reported pronounced changes in the relative composition of essential oil when the leaves were dried at 60 °C. Most of these changes occurred during the initial period of drying. Apart from the sensitivity of the essential oil constituents to temperature, these authors also attributed these losses to the structure of the leaves. In our case the structure of the peppercorn, and in particular the pericarp, may protect the volatile compounds from transfer during processing.

The three different unit operations tested, especially drying, impacted pepper color, reducing, in particular,  $a^*$  and  $b^*$  chromatic values leading to browning that was visible to the naked eye. Even blanching, which should reduce enzyme activity and hence limit browning, had a slight but significant impact on color. As suggested by Gu et al. (2013), aside from enzymatic browning described by several authors (Dhas & Korikanthimath, 2003; Mangalakumari, Sreedharan, & Mathew, 1983; Variyar, Pendharkar, Banerjee, & Bandyopadhyay, 1988), other browning mechanisms may be involved in the change in the color of the pepper.

### 4.3. Role and interest of each unit processing operation

Blanching significantly increased the drying rate of the pepper. By itself, this result, which can be explained by the partial destruction of the pepper cell walls, thus facilitating water transfer (Kaymak-Ertekin, 2002), justifies this step. Indeed, a blanching step, which reduces drying time, could save energy or limit climate-dependence in the case of sun drying. Blanching is also useful as it removes any dust from the pepper. Dhas and Korikanthimath (2003), suggested that moderate blanching could contribute to uniform browning by promoting the oxidation of phenols by phenolase enzymes. Depending on the length and temperature applied, as a thermal treatment, blanching could be a critical sanitary step as it reduces the microbial load as well as the saffrole content of the pepper. Sweating affected the color of the pepper, systematically reducing the  $a^*$  value, which corresponds to red, validating the hypothesis that the conditions (24 h at 35 °C in a water saturated atmosphere) used for sweating favor enzymatic browning, as described by Mangalakumari et al. (1983). The favorable humidity and temperature conditions could also stimulate the growth of microorganisms. Considering these results, we question the interest of this operation, which is used in Madagascar (Weil et al., 2014) for wild pepper species that are close to our *Piper borbonense*, as well as for *Piper nigrum*. Drying is crucial; it not only stabilizes the product but is also critical for the sanitary and sensorial quality of the pepper. Drying had a major influence on color as all values ( $L^*$ ,  $a^*$ ,  $b^*$ ) were affected, and the color turned

from red to brown. Referring to Agudelo-Laverde, Schebor, and del Pilar Buera (2013) who demonstrated that browning increased with an increase in water content on strawberry slices, we hypothesize that accelerating drying, by rapidly reducing water activity, could help preserve the red color.

## 5. Conclusion

The originality of dry *Piper borbonense* is based on its high aromatic potential, low pungency and red color. Separate or combined, the blanching, sweating and drying operations had no impact on piperine and essential oil content and only a slight impact on essential oil composition. The three unit operations influenced color, drying having the most impact. To preserve the color, sweating should be avoided, while blanching and drying could be optimized. Indeed, as demonstrated in this study, right blanching parameters could reduce drying time as well as limit enzymatic browning. Enhancing and innovating drying conditions would also help reduce both enzymatic and non-enzymatic oxidative reactions.

## Conflict of interest

The authors of this article certify that they have no affiliations with or involvement in any organization or entity with any financial interest or non-financial interest in the subject matter or materials discussed in this manuscript.

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## References

- Adams, R. P. (1995). *Identification of essential oil components by chromatography/quadrupole mass spectrometry*. Illinois, USA: Allured Publishing Corporation.
- Agudelo-Laverde, L. M., Schebor, C., & del Pilar Buera, M. (2013). Water content effect on the chromatic attributes of dehydrated strawberries during storage, as evaluated by image analysis. *LWT Food Science and Technology*, 52(2), 157–162. <http://dx.doi.org/10.1016/j.lwt.2012.06.022>.
- Ahmad, N., Fazal, H., Abbasi, B. H., Farooq, S., Ali, M., & Khan, M. A. (2012). Biological role of *Piper nigrum* L. (Black pepper): A review. [Article]. *Asian Pacific Journal of Tropical Biomedicine*, 2(Suppl. 3), S1945–S1953.
- Argyropoulos, D., & Muller, J. (2014). Changes of essential oil content and composition during convective drying of lemon balm (*Melissa officinalis* L.). *Industrial Crops and Products*, 52, 118–124. <http://dx.doi.org/10.1016/j.indcrop.2013.10.020>.
- Asekun, O. T., Grierson, D. S., & Afolayan, A. J. (2007). Effects of drying methods on the quality and quantity of the essential oil of *Mentha longifolia* L. subsp. *Capensis*. *Food Chemistry*, 101(3), 995–998. <http://dx.doi.org/10.1016/j.foodchem.2006.02.052>.
- Dhas, P., & Korikanthimath, V. (2003). Processing and quality of black pepper – A review. *Journal of Spices and Aromatic Crops*, 12(1), 1–13.
- FAO Statistics Division. (2015) Retrieved 1st October 2015, from <http://faostat3.fao.org/download/Q/QC/E>.
- Farag, S. E. A., & Abo-Zeid, M. (1997). Degradation of the natural mutagenic compound safrole in spices by cooking and irradiation. *Food/Nahrung*, 41(6), 359–361.
- Gu, F., Tan, L., Wu, H., Fang, Y., & Wang, Q. (2013). Analysis of the blackening of green pepper (*Piper nigrum* Linnaeus) berries. *Food Chemistry*, 138(2–3), 797–801. <http://dx.doi.org/10.1016/j.foodchem.2012.11.033>.
- International Standard Organization. (1982). Black pepper and white pepper, whole or ground – Determination of piperine content, ISO 5564.
- International Standard Organization. (1998). Piper (*Piper nigrum* L.), whole or ground – specification, ISO 959–1.
- International Standard Organization. (2008). Spices, condiments and herbs - Determination of volatile oil content (hydro distillation method), ISO 6571.
- Internet databases. (2014) Retrieved 12th October 2014, from <http://www.flavornet.org>, <http://www.pherobase.com>.
- Jagella, T., & Grosch, W. (1999). Flavour and off-flavour compounds of black and white pepper (*Piper nigrum* L.) III. Desirable and undesirable odorants of white pepper. *European Food Research and Technology*, 209(1), 27–31.
- Jennings, W., & Shibamoto, T. (1980). *Qualitative analysis of flavor and fragrance volatiles by glass capillary gas chromatography*. London, United Kingdom: Academic Press.
- Jirovetz, L., Buchbauer, G., Ngassoum, M. B., & Geissler, M. (2002). Aroma compound analysis of *Piper nigrum* and *Piper guineense* essential oils from Cameroon using solid-phase microextraction-gas chromatography, solid-phase microextraction-gas chromatography-mass spectrometry and olfactometry. *Journal of Chromatography A*, 976(1–2), 265–275. [http://dx.doi.org/10.1016/S0021-9673\(02\)00376-x](http://dx.doi.org/10.1016/S0021-9673(02)00376-x).
- Kaymak-Ertekin, F. (2002). Drying and rehydrating kinetics of green and red peppers. *Journal of Food Science*, 67(1), 168–175. <http://dx.doi.org/10.1111/j.1365-2621.2002.tb11378.x>.
- Kondjoyan, N., & Berdagué, J. L. (1996). *A compilation of relative retention indices for the analysis of aromatic compounds*. INRA de Theix, France: Laboratoire Flaveur.
- Mangalakumari, C. K., Sreedharan, V. P., & Mathew, A. G. (1983). Studies on blackening of pepper (*Piper nigrum*, Linn) during dehydration. *Journal of Food Science*, 48(2), 604–606. <http://dx.doi.org/10.1111/j.1365-2621.1983.tb10799.x>.
- Nisha, P., Singhal, R. S., & Pandit, A. B. (2009). The degradation kinetics of flavor in black pepper (*Piper nigrum* L.). *Journal of Food Engineering*, 92(1), 44–49. <http://dx.doi.org/10.1016/j.jfoodeng.2008.10.018>.
- Schulz, H., Baranska, M., Quilitzsch, R., Schutze, W., & Losing, G. (2005). Characterization of peppercorn, pepper oil, and pepper oleoresin by vibrational spectroscopy methods. *Journal of Agricultural and Food Chemistry*, 53(9), 3358–3363. <http://dx.doi.org/10.1021/jf048137m>.
- Schweiggert, U., Mix, K., Schieber, A., & Carle, R. (2005). An innovative process for the production of spices through immediate thermal treatment of the plant material. *Innovative Food Science & Emerging Technologies*, 6(2), 143–153. <http://dx.doi.org/10.1016/j.ifset.2005.11.006>.
- Sumathykutty, M. A., Rao, J. M., Padmakumari, K. P., & Narayanan, C. S. (1999). Essential oil constituents of some *Piper* species. *Flavour and Fragrance Journal*, 14(5), 279–282. [http://dx.doi.org/10.1002/\(sici\)1099-1026\(199909\)10:5<279::aid-fj821>3.0.co;2-0](http://dx.doi.org/10.1002/(sici)1099-1026(199909)10:5<279::aid-fj821>3.0.co;2-0).
- Suresh, D., Manjunatha, H., & Srinivasan, K. (2007). Effect of heat processing of spices on the concentrations of their bioactive principles: Turmeric (*Curcuma longa*), red pepper (*Capsicum annum*) and black pepper (*Piper nigrum*). *Journal of Food Composition and Analysis*, 20(3–4), 346–351. <http://dx.doi.org/10.1016/j.jfca.2006.10.002>.
- Variyar, P. S., Pendharkar, M. B., Banerjee, A., & Bandyopadhyay, C. (1988). Blackening in green pepper berries. *Phytochemistry*, 27(3), 715–717. [http://dx.doi.org/10.1016/0031-9422\(88\)84081-0](http://dx.doi.org/10.1016/0031-9422(88)84081-0).
- Weil, M., Descroix, F., Sing, A. S. C., Boulanger, R., Hoarau, M., Levesque, A., & Bohuon, P. (2014). Postharvest treatments of wild pepper (*Piper* spp.) in Madagascar. *Fruits*, 69(5), 371–380. <http://dx.doi.org/10.1051/fruits/2014025>.