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

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A B S T R A C T

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 of food. The quality of pepper as a spice is measured throughout pun- peptide, 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

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 Reu- nion Island. In Madagascar, wild peppers are processed according to the “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.
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 °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 °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). Blanching consisted in soaking the peppercorns in a hot water bath (Memmert Gmbh type WB 22 Schwabach, Germany) at a ratio of 1:36 peppercorns to water in three different conditions: at 60 °C for 30 s; 75 °C/180 s; and 100 °C/300 s. Sweating consisted of storing the peppercorns in a climatic chamber (BIA Climatic – Type CL 125, Conflans Sainte Honorine, France) at 35 °C and 99% RH for 24 h. Drying was performed by placing aluminum trays (300 cm²) containing 250 g of peppercorns arranged in a compact 1 cm thick layer for 39 h at 60 °C ± 1 °C, RH 20% ± 2% in the same climatic chamber. Hot air (60 °C, RH 20 ± 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 m long × 0.25 m wide × 0.92 m high), 150 g of peppercorns were placed on a sieve (0.25 × 0.25 m²) in a single thin non-compact layer. Hot air (60 ± 1 °C, RH 20 ± 2%) was circulated downwards at 2.7 ± 0.1 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® 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 °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, Memmert Gmbh, Germany) at 105 °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 ±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 α v4.60 (Thermo Fisher Scientific, USA). The mean relative deviation of repeatability was ±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 ±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 m × 320 μm × 0.25 μm) coupled to a MS detector. Aliquots (0.1 μL) 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 °C.
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 m x 320 μm x 0.25 μm) coupled to a MS detector. Aliquots (0.2 μL) 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 flow rate of the gas carrier (Helium) was 0.7 mL/min. The oven temperature program was as follows: initial temperature 60 °C, heating rate of 4 °C/min until a final temperature of 250 °C was reached then 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 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 & Berdagüé, 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 m x 320 μm x 0.25 μm) coupled to a FID detector. Aliquots (0.3 μL) 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 ±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.

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 h 20 min for blanched pepper. The comparison of the drying curves showed a much higher initial drying rate ($1.44 \pm 0.10$ kg, kg$^{-1}$ h$^{-1}$) in blanched pepper than in fresh pepper ($0.84 \pm 0.02$ kg, kg$^{-1}$ h$^{-1}$). The differences between the drying rates were no longer significant when the water content was below 0.5 kg of kg$^{-1}$. This water content was obtained after 4 h in fresh pepper and 3 h 15 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 h 15 min for “blanched plus sweated” pepper. Comparison of the drying curves revealed very similar drying rates in blanched plus sweated pepper ($1.47 \pm 0.16$ kg, kg$^{-1}$ h$^{-1}$) and in blanched pepper ($1.44 \pm 0.10$ kg, kg$^{-1}$ 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).
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 monoterpenoid family represented 55% of the total aromatic compounds. Limonene (27% of the total),
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 monoterpene 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

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

Mean values (n = 3).

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

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 9591-1 (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 Tsiperfery (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 at 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 pepper corn 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, Quilitzh, Schutze, and Losing (2005) who worked on black pepper, optimum pepper aroma (“top-peppery-note”) is obtained if monoterpenoids (excluding alpha- and beta-pinene) content is high but at the same time, the pinene content is low. As suggested by Gu et al. (2013), aside from enzymatic browning described by several authors (Dhas & Korikanthimath, 2003; Mangalakumari, Sreedharan, & Mathew, 1983; Varijar, 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 safrole 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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