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#### REVIEW

### The impact of roasting on cocoa quality parameters

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#### ABSTRACT

Roasting is an essential process in cocoa industry involving high temperatures that causes several physicochemical and microstructural changes in cocoa beans that ensure their quality and further processability. The versatility in roasting temperatures  $(100-150\,^{\circ}C)$  has attracted the attention of researchers toward the exploration of the effects of different roasting conditions on the color, proximal composition, cocoa butter quality, concentration of thermolabile compounds, formation of odor-active volatile organic compounds, generation of melanoidins, production of thermal processes contaminants in cocoa nibs, among others. Some researchers have drowned in exploring new roasting parameters (e.g., the concentration of water steam in the roasting chamber), whilst others have adapted novel heat-transfer techniques to cocoa nibs (e.g., fluidized bed roasting and microwaves). A detailed investigation of the physicochemical phenomena occurring under different cocoa roasting scenarios is lacking. Therefore, this review provides a comprehensive analysis of the state of art of cocoa roasting, identifies weak and mistaken points, presents research gaps, and gives recommendations to be considered for future cocoa studies.

#### **KEYWORDS**

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Proximal composition; Maillard reaction; melanoidins; polyphenols; volatile aroma compounds; cocoa butter

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#### Introduction

Several steps are required in processing cocoa beans (fermented and dried seeds of *Theobroma cacao* L.) before they are consumed or used as ingredients. Roasting particularly stands out due to the generation of authentic chocolate flavor via Maillard reaction (MR) (Minifie 1999). Other essential and desirable transformations that occur during roasting are the proper dehydration of the cocoa nibs, the significant reduction in microbial load, and the decrease in acidity, bitterness, and astringency (Gutiérrez 2017). Comprehensive reviews and books covering the whole cocoa processing chain are available (Rojas et al. 2022; Gutiérrez 2017; Beckett 2008). However, a detailed analysis of the physicochemical phenomena occurring during cocoa roasting process is missing.

Most of cocoa manufacturers roast cocoa beans in large rotating drum equipment, also known as Sirocco-type batch roasters, through which hot air of 110 to 150 °C is blown for 20 min to 2h until the moisture content and water activity of cocoa beans decrease to about 1% and 0.2, respectively (Rojas et al. 2022). After that, the cocoa bean's testas (the coats of the seeds, commonly named shells or husks) are removed by winnowing (Gutiérrez 2017; Beckett 2008). Some continuous roasters are provided with a pre-heating chamber that removes the shells before roasting (Varnam and Sutherland 1994). In that unit, cocoa beans are exposed to a high temperature produced by infrared radiant heat for a short time. Consequently, water from the shells and the edges of the cotyledons (also known as cocoa nibs) quickly evaporates and presses the shells outwards; thus, shells detach from the nibs and can be easily separated (Gutiérrez 2017). Pictures and synonyms of these cocoa beans' fragments are shown in Figure 1.

Essential physical, chemical, and organoleptic changes occur in cocoa nibs during roasting, for example: (i) Micropores are formed, leading to reduced hardness (Abo-Bakr and Shekib 1987; Hoskin and Dimick 1980); thus facilitating grinding processes. (ii) The concentrations of some macro (Oracz and Nebesny 2019; Zzaman et al. 2017; Redgwell, Trovato, and Curti 2003) and micronutrients (Oracz and Nebesny 2019; de Taeye et al. 2017) decrease due to volatilization or to their involvement in thermo-activated chemical reactions like Maillard reaction. (iii) High molecular weight compounds such as melanoidins (Quiroz-Reyes and Fogliano 2018; Sacchetti et al. 2016; Oracz and Nebesny 2019), and low molecular weight compounds with odor activity (Tan and Kerr 2018; Mohamadi Alasti et al. 2019; Zzaman et al. 2017; Diab et al. 2014; Huang and Barringer 2011) are formed via MR. (iv) Thermal process contaminants, such as 5-hydroxymethylfurfural (HMF) (Quiroz-Reyes and Fogliano 2018; Sacchetti et al. 2016) and acrylamide (Żyżelewicz et al.

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Figure 1. Main components of cocoa beans arising from their process.

2017; Granvogl and Schieberle 2007) are formed. Finally, (v) the acidity in cocoa butter, which is the most abundant and expensive cocoa derivative (International Cocoa Organization 2021), is reduced.

Those valuable findings needed to be collated and compared to provide a holistic approach of the physicochemical changes of cocoa upon roasting. Thus, in this review, the current state of knowledge on the effect of different roasting conditions on microstructure, color, proximate composition, polyphenols content, formation of melanoidins, generation of thermal processes contaminants, and production of volatile organic compounds in cocoa nibs; as well as the quality of cocoa butter, are presented. Roasting alternatives and research gaps are also discussed.

#### Literature search

The following databases were used: WUR library search, Web of Science, Google books, Google Patents, and Google Scholar. For each section of this review, studies performed with various roasting conditions (e.g., different roasting temperatures) were highly preferred over those holding only one. Recent studies (last 10 years) were also prioritized over the older ones; nonetheless, we extended our search up to 50 years in some sections due to the above-mentioned filter (more than one roasting condition).

## Effect of roasting on the microstructure and texture of cocoa nibs

Typical cocoa-roasting temperatures trigger the evaporation of water and low-boiling-point organic compounds present in the food matrix. This physical phenomenon implicates the formation of large amounts of gases that generate high internal pressure. This in turn causes noticeable changes in the structure, such as breakdown of the cell walls, microporosity formation, increase in volume, and reduction in density (Massini et al. 1990) and hardness (Zzaman and Yang 2013).

A couple of papers have explored the microstructure of cocoa nibs before and after roasting. Observations of stained

samples through polarized light microscopy demonstrated that drying and roasting processes cause the disruption of parenchymal cells (de Brito et al. 2000). Abo-Bakr and Shekib (1987) and Hoskin and Dimick (1980), who made a structural and topographical examination of cocoa nibs through scanning electron microscopy, found that more pores and pits were present on the surface of the cotyledons of roasted cocoa than on unroasted nib's surface. The typical nib brittleness after roasting was attributed to those microscopic modifications.

Despite those insights, there is still a lack of knowledge on how the main roasting parameters (i.e., temperature and time) affect the microstructure of cocoa nibs. Further studies might go beyond superficial observations by providing quantitative data on porosity and correlating it with the fat bloom phenomenon. Micro X-ray tomography technique may assist this gap since it can reconstruct the three-dimensional density distribution within an object, providing qualitative and quantitative information about the solid and the empty (or airy) sections (Maire and Withers 2014). The potential of micro X-ray tomography is illustrated in Figure 2, which shows the bidimensional projections of cocoa nibs roasted in different conditions (authors' unpublished data).

#### Effect of roasting on the color of cocoa nibs

The initial color of cocoa nibs before roasting is already deep brown. Most of their pigments are formed during fermentation and drying via complexation of amino acids and/or proteins with quinines (an enzymatic oxidation product of polyphenols) or with condensed tannins (high molecular weight product of flavonoid polymerization) via hydrogen bonding (Misnawi et al. 2003). This naturally-dyed matrix hinders visual-color analysis (Peña-Correa et al. 2022). Moreover, the water content changes upon roasting. It is well known that water affects the color perception of materials by reducing light absorption; as a consequence, materials look darker when they are wet. So, finding the sole effect of roasting on the formation of non-enzymatic brown chromophores in cocoa is challenging.



Figure 2. 2-D projections of virtual cross-sections of the solid fraction obtained by X-ray tomography from (a) unroasted cocoa nibs, (b) cocoa nibs roasted in a conventional oven at 120 °C, and (c) cocoa nibs roasted in a fluidized bed roaster at 120 °C.

Nevertheless, numerous studies have investigated the effect of different roasting conditions on color changes of cocoa nibs. The color measurement has been done directly on the surface of the nibs (Zzaman and Yang 2013) and in different processed forms, for example: ground cocoa nibs by Żyżelewicz, Budryn, et al. (2014) and Sacchetti et al. (2016); cocoa liquor (cocoa nibs sufficiently minced into a smoothy pulpy mass -See Figure 1-) by Nurhayati et al. (2019); cocoa powder (defatted cocoa liquor - See Figure 1-) by Quiroz-Reyes and Fogliano (2018), and high molecular weight water-soluble extracts (HMW-WSE) by Quiroz-Reyes and Fogliano (2018), Oracz and Nebesny (2019), and Sacchetti et al. (2016). Most researchers used colorimeters to obtain the CIE L\*a\*b\* color attributes regardless of the aliquot shape. Some other studies have drawn on UV/VIS-spectrophotometry or fluorescence techniques. In our recent study, we showed that computer vision-based image analysis was an effective technique for measuring color changes of cocoa nibs (Peña-Correa et al. 2022). Table 1 summarizes the findings of the mentioned studies.

The majority of the studies quoted in Table 1 agree that roasting increases a<sup>\*</sup> and b<sup>\*</sup> values, especially the latter. Their findings on L<sup>\*</sup> value are diverse; they are influenced by the roasting temperature and sample shape: When the typical cocoa-roasting temperatures  $(110 - 150 \,^{\circ}\text{C})$  were applied, L<sup>\*</sup> values of cocoa nibs' surface did not significantly change upon roasting. However, a darkening effect was evident when roasting between  $150 \,^{\circ}\text{C} - 250 \,^{\circ}\text{C}$ , or when processing the nibs into cocoa powder, cocoa liquor, or HMW-WSE. High roasting temperatures certainly remove significant amounts of water, so the interference of water with L<sup>\*</sup> is mitigated. On top of that, overroasting occurs above 150 °C in cocoa nibs; as a consequence, deep-brown and black compounds are generated.

Neither the efforts in transforming cocoa nibs into other forms nor the use of a robust method like computer vision-based picture analysis seem to sharpen the color analysis of cocoa roasted under regular roasting temperatures. The native pigments seem to hinder any color change. The development of a robust sample preparation would be of great assistance.

#### Chemical changes and neo-formed compounds

During heating treatments of food matrixes, the MR occurs with the formation of various products of diverse molecular weights. The reactants are mainly reactive carbonyl compounds (e.g., monosaccharides and lipid-oxidation products) and amino acids. Figure 3 represents the global dynamics of the main chemical reactions involved during the roasting process of cocoa. The entire pathways of the MR have not been unraveled at all. However, due to its great importance on the quality and final organoleptic characteristics of diverse thermally processed food, it has been the subject of investigation, as reviewed Ruan et al. (2018), Somoza and Fogliano (2013), Echavarría, Pagán, and Ibarz (2012), and Martins, Jongen, and van Boekel (2000).

In contrast to other typically thermally-processed foods like coffee and bread, cocoa beans are particularly rich in fat and phenolic compounds, and contain little quantities of sugars and water. Moreover, the cocoa beans' roasting temperature range is not as high as for coffee roasting  $(180 - 280 \,^{\circ}\text{C})$  or dough baking  $(180 - 250 \,^{\circ}\text{C})$ . Nonetheless, the MR occurs in cocoa nibs and cocoa model systems (Oliviero et al. 2009), as addressed in the coming subsections.

Table 1. Changes in color attributes in cocoa beans upon roasting.

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\*95% confidence interval



Figure 3. Classification and transformation of the main cocoa polyphenols (green box) during fermentation and roasting. Formation of aroma precursors (carbonyl compounds and amino compounds) during fermentation and roasting (blue box) and its interaction with phenolic compounds via Maillard reaction (black box) toward the formation of Maillard reaction (MR) products such as desirable volatile organic compounds (pyrazines and Strecker aldehydes) and Melanoidins (brown box).

### The effect of roasting on the proximate composition of cocoa

The roasting process leads to two critical physical phenomena that might interfere with the interpretation of the chemical analysis if they are not carefully taken into account. They are the loss of water and the migration of lipid fractions from cocoa nibs toward the shells. The second one causes losses of as much as 0.5% of cocoa butter during deshelling (Gutiérrez 2017). Both physical phenomena might cause misinterpretations of the results of the proximal composition of cocoa when: (i) the outcomes are not expressed either in dry basis (d.b.) or in dry and defatted basis (cocoa powder); and (ii) the authors do not specify whether they removed or not the shells before analyzing their samples, a fraction that could account from 10 to 17% of the total cocoa bean weight (Rojo-Poveda et al. 2020). Table 2 summarizes the proximal composition of cocoa from various studies and includes the basis of the units according to the roasting conditions. Most of the researchers declared using deshelled beans during sample preparation; however, their results showed typical features of cocoa beans instead of cocoa nibs; thus, the proper names of the various cocoa derivatives, as expressed in Figure 1, need to be constantly adopted by scientific communities.

As summarized in Table 2, more than half of the macronutrients in cocoa nibs is the fat. Proteins are in second place, comprising about one-fourth. The carbohydrate content of cocoa beans is less than 7% (w/w), including a wide variety of monosaccharides, disaccharides, oligosaccharides, and polysaccharides. Sugars account for less than 2% (w/w). The order of predominance of sugars in fermented and

Table	2.	Effect	of	roasting	in	proximate	composition	of	сосоа	nibs.
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<sup>a</sup>There is no information concerning the conversion of the data into dry bases (d.b)

<sup>b</sup>Cocoa nibs roasted at 120 and 140 °C for the time needed to reach a moisture content of about 1%.

unroasted cocoa nibs is not even among studies (Oracz and Nebesny 2019; Redgwell, Trovato, and Curti 2003). It seems not to be a matter of the variety; the heterogeneous fermentative conditions markedly affect the final amount of sugars in cocoa beans (Redgwell, Trovato, and Curti 2003).

When the roasting time and the roasting temperature of cocoa beans increases, the concentration of sugars is significantly reduced. Fructose is the most affected one, followed by glucose, and then sucrose (Zzaman et al. 2017; Oracz and Nebesny 2019). Fructose and glucose are reducing sugars, and they are directly involved in the reaction with an amine source, while sucrose first needs to be hydrolyzed into glucose and fructose (Rufian-Henares 2016), thus explaining the superior reactivity of monosaccharides. The higher decrease in fructose compared to glucose is in line with model systems experiments (Brands 2002). The hydroxyl group orientation at C3 and C4 positions of fructose may also confer such a higher reactivity (van Putten et al. 2013). Concerning the oligosaccharides, they seem to be steady after roasting (Redgwell, Trovato, and Curti 2003).

The crude fiber content increases after roasting cocoa beans (Redgwell, Trovato, and Curti 2003). In many plant-based products, the insoluble material remaining after specific chemical treatments is referred to as crude fiber and is mainly represented by lignin. Some other compounds, such as tannin/protein complexes and HMW Maillard reaction products, formed during roasting may also contribute to that moiety. The higher content of crude fiber in roasted cocoa compared with unroasted might be related to the formation of non-soluble melanoidins (Redgwell, Trovato, and Curti 2003).

Cocoa butter is considered a stable fat due to its fatty acid composition. Nevertheless, transformations may occur to some extent during roasting, as addressed in the section 'Cocoa butter quality.' According to the tolerance limits of some lipid-oxidation products established by Codex Alimentarius (1981), the concentrations of those products in cocoa butter do not represent significant gravimetric amounts under regular processing conditions. Thus, the formation of lipid-oxidation products should not significantly reduce the initial amount of cocoa butter. The authors of this review confirmed it, as observed in Table 2. Considering this rationale, fat losses upon roasting can be hardly attributed to the generation of neoformed lipid-derived products, as Oracz and Nebesny (2019) explained for the 3.8% fat loss. It is unclear whether the study considered the fat migration toward the shells. Model system experiments better deal with mass balance.

Drawbacks also happen when comparing the total protein content before and after roasting. When using nitrogen-determination methods to calculate protein content by using conversion factors (e.g., Kjeldahl and Dumas), the results from unroasted cocoa vs. roasted cocoa should not significantly differ: In principle, roasting makes some nitrogen atoms to move from one place (e.g., proteins) to another (e.g., volatile organic compounds and melanoidins) within the same matrix, so no significant changes in 'total protein content' are obviously expected. The authors of this review confirmed it (unpublished data), as observed in Table 2. Nevertheless, some researchers (Oracz and Nebesny 2019; de Brito et al. 2000) reported protein reductions of 27.1% and 37% upon roasting, respectively. The latter used Bradford's protein assay method, which also lacks specificity.

Investigating the effect of roasting in changing the concentration of specific proteins or amino acids makes more sense than total protein content, as shown in Table 2. The free amino acid content in cocoa significantly decreases by roasting time and temperature (Zzaman et al. 2017; Bonvehí and Coll 2002; de Brito et al. 2000; Reineccius, Keeney, and Weissberger 1972; Rohan and Stewart 1966), most probably due to their involvement in the MR (Figure 3). The percentage of reduction of total amino acids roughly ranged from 25 to 50% (Table 2). However, some amino acids were more affected than others: In the study of Zzaman et al. (2017), the amount of glutamic acid, tyrosine, glycine, and histidine was the most reduced, in contrast to other tested amino acids. According to the data published by de Brito et al. (2000) and Bonvehí and Coll (2002), the most affected amino acids were histidine, cysteine, and methionine.

It is worth to mention that the composition of cocoa nibs fluctuates among cocoa varieties and origins (Febrianto, Wang, and Zhu 2021); furthermore, harvesting activities, fermentation, and drying contribute to those differences.

#### Effect of roasting on polyphenols content in cocoa

The classification of the most abundant polyphenols present in the seeds of *Theobroma cacao* L. and their dynamics during roasting are shown in Figure 3. Monomeric flavan-3ols such as (-)-epicatechin and (+)-catechin, as well as the proanthocyanidins B1, B2, and C1 (abbreviated as P-B1, P-B2, and P-C1, respectively), are the most abundant classes of polyphenols identified in cocoa beans and derivatives (Grassia et al. 2019; Mazor Jolić et al. 2011). With minor participation, phenolic acids such as gallic acid, protocatechuic acid, ellagic acid, caffeic acid, p-coumaric acid, ferulic acid, and sinapic acid, have been identified in cocoa beans (Oracz and Nebesny 2019).

The concentration of polyphenols may differ among cocoa varieties, being epicatechin the most predominant one, followed by P-B2, which is made of two epicatechin units. Both account for about half of the total polyphenols, according to the data supplied by Oracz and Nebesny (2019) and Quiroz-Reyes and Fogliano (2018). Lower but significant concentrations of P-B1 (made of one unit of epicatechin and one unit of catechin) and P-C1 (comprising three units of epicatechin) in cocoa beans have been reported (Oracz and Nebesny 2019)

The concentration of phytochemicals in cocoa seeds is exceptionally high: they account for 15 to 20% of the dried fat-free mass of fresh cocoa seeds. Their content usually drops to about 5% upon fermentation and drying, as they oxidize to condensed high molecular compounds, mostly insoluble tannins (Wollgast and Anklam 2000). When this reduction is above 10%, it is considered a bad fermentation (Wollgast and Anklam 2000) due to the excess of bitterness and astringency that may persist in cocoa derivatives (Stark, Bareuther, and Hofmann 2005). About 50% of the remaining polyphenols are lost upon roasting (Wollgast and Anklam 2000) as they become part of the melanoidins' polymeric structure (Quiroz-Reyes and Fogliano 2018; Oracz, Nebesny, and Żyżelewicz 2019), as shown in Figure 3. These enzymatic and non-enzymatic transformations of polyphenols have been negatively perceived due to the imminent reduction in antioxidant capacity of cocoa (Urbańska and Kowalska 2019); however, the organoleptic characteristics are considerably improved (Stark, Bareuther, and Hofmann 2005). Various researchers have evaluated or reviewed the changes in concentration of cocoa polyphenols from postharvest until the preparation of chocolate bars (Gil et al. 2021; Urbańska et al. 2019; Bordiga et al. 2015; Payne et al. 2010; Wollgast and Anklam 2000). So, this section focuses on the specific changes upon different roasting conditions.

During heating treatments, proanthocyanidins experiment depolymerization through the cleavage of the interflavanic  $(4\beta \rightarrow 8)$  linkages; consequently, monomeric-flavan-3-ols are released (Figure 3) (de Taeye et al. 2014). Simultaneously, (-)-epicatechin and (+)-catechin undergo epimerization reactions to produce (-)-catechin and (+)-epicatechin, respectively. The epimerization of (+)-catechin into (+)-epicatechin is usually less favored (de Taeye et al. 2014). These changes mainly result in: (i) the significant reduction of the most predominant phenolic compounds, namely epicatechin and P-B2; and (ii) a significant increase of (-)-catechin. The higher the roasting temperature, the more pronounced the changes (Stanley et al. 2018; Quiroz-Reyes and Fogliano 2018; Żyżelewicz et al. 2016; Kothe, Zimmermann, and Galensa 2013). Payne et al. (2010) proposed calculating the epicatechin/catechin ratio as an indicator for the processing history of cocoa beans. However, other studies have reported other dynamics in catechin upon roasting: it may increase during the first 15 min, and then decrease to original or lower concentrations (Żyżelewicz et al. 2016); or just decrease (Oracz and Nebesny 2019; Ioannone et al. 2015). Changes in concentration of catechin are determined by cocoa variety (de Taeye et al. 2017).

The fate of the missed cocoa polyphenols upon roasting has been scarcely studied. As shown in Figure 3. they are involved in the formation of soluble (Oracz, Nebesny, and Żyżelewicz 2019; Quiroz-Reyes and Fogliano 2018) and insoluble (Fogliano et al. 2011) melanoidins via MR, mitigating so the bitterness and astringency. However, the extent of participation of free phenolic compounds in that chemistry is unknown. More detailed information about the kind of polyphenols found in cocoa melanoidins is given in the following section.

#### Melanoidins in cocoa as an advanced MR product

Cocoa melanoidins are high molecular weight heterogeneous compounds with brown-chromophoric properties that are formed in the advanced stages of the Maillard reaction. As shown in Figure 3, they result from the interaction of the amino groups of peptides and free amino acids with carbonyl compounds and polyphenols. Melanoidins can be either soluble or insoluble. The insoluble fraction has been scarcely studied, while the soluble one has drained most of the attention.

Different techniques have been presented to estimate the formation of soluble cocoa melanoidins. Quiroz-Reyes and Fogliano (2018) and Oracz and Nebesny (2019) weighed the dry fraction of high molecular weight water-soluble extracts (HMW-WSE) obtained by ultrafiltration (Quiroz-Reyes and Fogliano 2018) and dialysis (Oracz and Nebesny 2019). Sacchetti et al. (2016) also used dialysis to separate high molecular weight compounds. Different from Quiroz-Reyes and Fogliano (2018) and Oracz and Nebesny (2019), the extraction was not carried out by using 100% of polar solvent; a mix of solvents with a predominant presence of acetone was used instead; then they analyzed the cocoa melanoidins via fluorescence detection using quinine sulfate as standard. With this in turn, Sacchetti et al. (2016) demonstrated that the formation of melanoidins in cocoa beans depends on the roasting temperature and increases exponentially at temperatures above 135 °C. In contrast, the gravimetric method leads to some divergences between studies: On the one hand, Quiroz-Reyes and Fogliano (2018) found that the dry mass of the HMW-WSE (> 20kDa) of unroasted cocoa was around 6.4-7.7% of the cocoa powder. Roasting significantly increased it to about two folds; however, the roasting temperature did not lead to any trend. On the other hand, Oracz and Nebesny (2019) found 12.9-14.3% w/w of HMW-WSE (> 12.4 kDa) in cocoa powders obtained from unroasted cocoa; their roasting experiments performed at 110-120 °C slightly reduced that yield, while the highest temperatures (135-150 °C) did not significantly change it. Differences between research may be explained by the composition of the samples themselves, as well as the roasting conditions and sample preparations.

Estimating the formation of melanoidins by fluorescent or gravimetric methods is still out of focus. One reason is that fluorescent methods are specific to the standard being used; thus, other neo-formed compounds might be underestimated. Another point to be considered is that gravimetric methods retain everything above the molecular weight cutoff of the membrane used. Consequently, other high molecular weight compounds, such as proteins and tannins, are retained in the HMW fraction, which might lead to overestimating the amount of melanoidins.

The exploration of the physical and chemical properties of cocoa melanoidins, as studied by Oracz and Nebesny (2019), Oracz, Nebesny, and Żyżelewicz (2019), Quiroz-Reyes and Fogliano (2018), and Summa et al. (2008) might assist in designing a more accurate quantification method. These are their laudable contributions:

• The molecular weight distribution of the HMW-WSE above 12.4 kDa obtained from unroasted cocoa differs from that of roasted cocoa: in unroasted cocoa, the weight of these compounds was almost equally distributed between 12.4 and 150 kDa, while in roasted cocoa, there was a Gaussian bell distribution that peaked at about 50 kDa. The peak height increased when the roasting temperature increased; thus, it could probably correspond to the formation of cocoa melanoidins (Oracz and Nebesny 2019). Isolating a narrower fraction of the HMW-WSE, perhaps between 30 and 70 kDa, may sharpen cocoa melanoidins' analysis.

- Aligned results were presented by Quiroz-Reyes and Fogliano (2018). The water-soluble compounds were extracted from unroasted and roasted cocoa by consecutive ultrafiltration through membranes of 20, 10, and 2kDa cutoff. Gravimetric measurement of the fraction indicated that the fraction containing compounds above 20kDa represented about 65 to 85% of the total HMW-WSE, while the fractions of 20 to 10 and 10 to 2kDa hold about 10 to 20% each.
- According to the data presented by Quiroz-Reyes and Fogliano (2018), 50% of the antioxidant activity of the whole water-soluble extract of unroasted cocoa relies on compounds above 20 kDa. In roasted cocoa, this fraction was responsible for 60 to 80% of the antioxidant activity. The evident increment in the antioxidant activity upon roasting suggested the effective incorporation of phenolic compounds in cocoa melanoidins (Quiroz-Reyes and Fogliano 2018).
- The HMW-WSE of unroasted cocoa is mainly composed of proteins (15 to 20% w/w), carbohydrates (8 to 16% w/w), and water (≈5%) (Oracz and Nebesny 2019), and a fraction of at least 0.2% w/w d.b. correspond to phenolic compounds according to the data published by (Oracz, Nebesny, and Żyżelewicz 2019). So far, more than half of the composition of cocoa melanoidins is unknown. Nevertheless, the current information is enough to conclude that cocoa melanoidins may differ from other roasted products like coffee beans: Coffee melanoidins contain more carbohydrates (40 60% w/w) and a wider range of protein content (2.3 28% w/w), according to the data published by Coelho et al. (2014).
- Roasting slightly decreases the protein content in cocoa HMW-WSE, significantly reduces their carbohydrate content (Oracz and Nebesny 2019), and significantly increases the polyphenols content. Interestingly, the increment in bounded phenolic compounds increases when roasting temperature increases (Oracz, Nebesny, and Żyżelewicz 2019). This rise is also aligned with the antioxidant activity mentioned above.
- The aforementioned phenolic compounds present in HMW-WSE of cocoa diverge between cocoa varieties and polyphenols' hydrolysis methods (Oracz, Nebesny, and Żyżelewicz 2019). The acid hydrolysis released more phenolic compounds from Criollo's HMW-WSE than alkaline hydrolysis, while HMW-WSE of Forastero beans showed an inverse trend.
- The most predominant phenolic compound released upon alkaline hydrolysis of Forastero's and Criollo's HMW-WSE was epicatechin (60% approx.), followed by catechin (20% approx.). The acid hydrolysis revealed catechin (45% approx.) as the most abundant polyphenol bound to HMW-WSE, followed by epicatechin (20% approx.). The rest of the polyphenols in both hydrolysis methods corresponded to P-B2, gallic acid, ellagic acid, caffeic acid, p-coumaric

acid, ferulic acid, sinapic acid, and protocatechuic acid (Oracz, Nebesny, and Żyżelewicz 2019). Phenolic compounds could be linked to diverse components of the matrix through either ester, ether, or acetal bonds. This diversity makes it difficult the development of one definitive method for hydrolysis (Robbins 2003).

### Thermal processes contaminants formed by the MR during roasting

Heating treatments like roasting have food safety concerns caused by some neoformed compounds like HMF and acrylamide. There are many doubts about HMF's health effects on humans. In rats, the LD50 is between 2.5 and 5.0 g/kg. Those values are away from the estimated dietary HMF intake of 1.6 mg/person, as Capuano and Fogliano (2011) thoroughly reviewed. Nevertheless, the major concern for HMF is related to its in vivo conversion to 5-sulfoxymethyfurfural (Surh et al. 1994). Acrylamide is a well-known carcinogenic and neurotoxic molecule (International Agency for Research on Cancer 1994). Similar to HMF, the toxicological levels of acrylamide estimated in rats and mice ranged far above dietary exposure (World Health Organization 2006). Although the long-term consequences of the dietary intake of those compounds are still under investigation, several strategies for their mitigation have been put forward (Kolek, Simko, and Simon 2006; Claeys, De Vleeschouwer, and Hendrickx 2005).

Both HMF and acrylamide can be formed by MR in foods during thermal treatment. HMF can also be formed via sugar dehydration. The amino acid asparagine forms the backbone of acrylamide. Free asparagine and reducing sugars are needed to form acrylamide via MR. Consequently, HMF and acrylamide mainly occur in carbohydrate-rich products (Capuano and Fogliano 2011). As shown in Table 2, this is not the situation of cocoa nibs; nevertheless, limited formation of these contaminants in cocoa ranging from 15 to 75 µg HMF/g cocoa powder (Quiroz-Reyes and Fogliano 2018), 1.2 to 7.3 nmol HMF/g cocoa beans (Sacchetti et al. 2016), 222 to 922 µg acrylamide/kg cocoa beans (Granvogl and Schieberle 2007), and 20 to 270 µg acrylamide/kg cocoa beans (Żyżelewicz et al. 2017), has been reported. The analysis of acrylamide in various cocoa-based products, including unroasted cocoa beans, roasted cocoa nibs, cocoa butter, cocoa powder, and several end-consumer chocolate products demonstrated that the level of acrylamide is below the general German signal value of 1000 µg/kg (Raters and Matissek 2018). A similar analysis for HMF would provide valuable information for cocoa manufacturers and customers.

HMF formation could vary within cocoa varieties, being Criollo more prone to produce HMF than Forastero (Quiroz-Reyes and Fogliano 2018). The generation of this compound in cocoa increases exponentially at temperatures above 135 °C (Sacchetti et al. 2016); however, at an equal moisture content of 1.9% w/w (almost the end of roasting), the concentration of HMF in cocoa beans roasted at the highest roasting temperatures led to the lowest HMF formation. It means that not only the roasting temperature but also the roasting time plays an essential role in HMF formation, as the lower temperatures demand longer roasting times. Even though acrylamide formation is highly favored under thermal processes, this compound was detected in unfermented and fermented cocoa beans (Granvogl and Schieberle 2007). When roasting both the unfermented and the fermented beans, the acrylamide content significantly rose, especially when roasting the fermented beans. The results suggest that fermentation generates precursors and creates a favored environment for acrylamide formation (Granvogl and Schieberle 2007). Żyżelewicz et al. (2017) did not find acrylamide in unroasted cocoa, but in line with Granvogl and Schieberle (2007), they observed a sharp increase of this compound upon roasting.

#### Volatile organic compounds

The fermentation process holds diverse enzymatic-catalyzed reactions within the freshly harvested seeds of cocoa, such as proteolysis, hydrolysis of carbohydrates (Santander Munoz et al. 2020), and lipolysis (Afoakwa et al. 2014). As a result, amino acids, monosaccharides, and free fatty acids, among other compounds, are produced (Santander Munoz et al. 2020), as briefly shown in Figure 3. For several years, the first two have been referred to as 'aroma precursors' because of their proven participation in the formation of chocolate flavor via MR. Recently, lipid-derived carbonyl compounds were found to participate in that chemistry (Zamora, Lavado-Tena, and Hidalgo 2020; Hidalgo and Zamora 2019); therefore, they also deserve to be called aroma precursors.

More than 600 volatile organic compounds (VOC), including organic acids, alcohols, aldehydes, esters, fatty acids, furans, hydrocarbons, ketones, phenols, pyrazines, pyrroles, and sulfur compounds, have been identified along with cocoa processing. The flavor chemistry in cocoa processing has been reviewed by a number of authors (Rojas et al. 2022; Santander Munoz et al. 2020; Aprotosoaie, Luca, and Miron 2016; Afoakwa et al. 2008).

During cocoa roasting, two relevant phenomena in the matter of VOC profile occur: (i) the amount of acetic acid is significantly reduced. It is the highest odor-active compound in unroasted cocoa beans and is responsible for the typical vinegar-like off-flavor (Michel et al. 2021; ii) Several nitrogen-heterocycles (e.g., pyrazines) and Strecker aldehydes are produced in the intermediate stages of the Maillard reaction. Pyrazines mainly display nutty, earthy, roasty, typical chocolate, potato, and green aromas; and aldehydes sweet and malty notes (Owusu, Petersen, and Heimdal 2012; Michel et al. 2021). Both phenomena lead to improved chocolate flavor.

The formation of pyrazines (the main class of nitrogen-heterocyclics), especially 2-ethyl-5-methylpyrazine, is temperature-dependent under typical roasting temperatures, as demonstrated in our recent study (Peña-Correa et al. 2022). However, when roasting at temperatures above 150 °C, their concentration peaks at intermediate-roasting times, and then drops through the final times of roasting (Huang and Barringer 2011; Zzaman et al. 2017). That decrease might correspond to the contribution of those valuable aroma compounds in forming advanced Maillard reaction products like melanoidins.



**Figure 4.** PCA of the volatile organic compounds grouped by chemical groups present in cocoa beans roasted at four different temperatures (110, 120, 130, and 140 °C) in two different equipment: a fluidized bed roaster (FRB) and a convective oven (CO). The data correspond to the final roasting times. At those points, the water content ranged from 1 to 2% w/w. Source of the data: Peña-Correa et al. (2022).

Different roasting techniques change the VOC profile of the cocoa beans. Our recent study (Peña-Correa et al. 2022) compared the formation of nitrogen-heterocyclics under two roasting techniques, fluidized bed roasting (FBR) and convective oven roasting (CO), at four roasting temperatures (110-140 °C). By using the data of the final roasting time (published in the supplementary material of that study), we elaborated Figure 4. The nitrogen-heterocyclics are positively correlated with FBR cocoa, especially with the highest roasting temperature (FBR-140), meaning that this roasting condition highly favors the formation of pyrazines (Figure 4). The aldehydes were correlated to the highest roasting temperatures of convective-oven-roasted cocoa nibs, while acetic acid was correlated with the lower roasting temperatures of FBR.

The differences between VOC profile of cocoa nibs roasted under different techniques are probably due to the final water activity. The  $a_w$  value of FBR roasted cocoa nibs is close to 0.3, whilst cocoa nibs roasted in CO had an  $a_w$ close to 0.2. The formation of pyrazines peaks at  $a_w$  values nearby 0.3, and is strongly reduced when approaching to 0.2 or 0.38, as confirmed by Scalone et al. (2015) in a model system. It is well known that the performance of chemical reactions depends on the water activity of the matrix, and this factor has been poorly considered in understanding the chemistry behind cocoa roasting.

With these findings, the scientific community and cocoa manufacturers may consider pursuing an  $a_w$  of 0.3 in cocoa nibs to intensify the chocolate aroma by naturally increasing nitrogen heterocyclics without carrying burnt flavors (Peña-Correa et al. 2022).

#### **Cocoa butter quality**

Even though cocoa butter is the largest and the most expensive fraction of cocoa nibs, few studies focus on the effect of roasting on the quality of cocoa butter. It might be because: (i) cocoa butter mainly consists of saturated fatty acids; thus conferring high thermal stability (Żyżelewicz, Budryn, et al. 2014; Oracz, Nebesny, and Żyżelewicz 2014; ii) No much primary-oxidation products (e.g., hydroperoxides) are expected during roasting, as the solubility of oxygen is limited at high temperatures (Velasco and Dobarganes 2002); stable secondary-oxidation products such as polymeric compounds and VOC are favored instead (Velasco and Dobarganes 2002; Tena et al. 2019; Carlin et al. 1986).

The hydrolysis of triacylglycerols into free fatty acids (FFA), diacylglycerols, and monoacylglycerols in cocoa beans mainly occur during postharvest and fermentation via enzymatic catalyzed reactions (Afoakwa et al. 2014). At such low-moderate temperatures, the solubility of oxygen is high; consequently, those hydrolysis products (especially the FAA) easily interact with oxygen to produce hydroperoxides (Velasco and Dobarganes 2002). This phenomenon explains the initial load of FFA and the peroxide value (PV) in fermented and dried cocoa beans. As expected, roasting at conventional temperatures like 120 °C (Afoakwa et al. 2014) and 135°C (Żyżelewicz, Budryn, et al. 2014) leads to a marginal effect on FFA and PV content. However, the stability of cocoa starts to fade at 150 °C (Żyżelewicz, Budryn, et al. 2014); and at 180 °C, the increase in FFA and PV is significantly evident (Djikeng et al. 2018).

The changes in the quality of cocoa butter upon roasting at regular temperatures can be better assessed through the analysis of their VOC. Hashim, Hudiyono, and Chaveron (1997) intentionally oxidized cocoa butter and found relevant changes in the VOC profile: They reported a significant increase in the aldehydes hexanal, heptanal, octanal, nonanal, and decanal. Those aldehydes are oxidative decomposition products formed from free fatty acids (Grebenteuch et al. 2021). An extension of this study involving different roasting temperatures and/or some mathematical modeling might provide valuable tools for cocoa butter producers to understand and predict the limits of this 'stable' fat.

It is worth mentioning that solvent extraction is the most common method used in the lab to obtain cocoa butter; however, this is not the method used in cocoa industry. A horizontal press is mainly used to extract the butter from cocoa liquor. In principle, hot cocoa liquor is introduced into a container where pressing is carried out by applying a sequence of increasing pressures that leaks out the melted fat from the rest of the cocoa (the cocoa cake, see Figure 1) (Gutiérrez 2017). The technological parameters of this treatment may affect the quality of cocoa butter; however, this step has not been deeply considered by scientific community.

#### **Conventional roasting and roasting alternatives**

As mentioned in the introduction of this review, the roasting process in cocoa industry mainly starts from the whole cocoa beans, which are thermally processed in continuous drum roasters for long periods ranging from 20 min to 2 hours (Gutiérrez 2017). Some modifications of the

Table 3.	Investigations	on	roasting	alternatives	for	cocoa
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Entry	Roasting techniques and modifications	Objective	Pros	Cons	References
1	Roasting a thin layer of cocoa liquor under atmospheric or reduced pressure in a conductive-heat transfer cylinder. The roaster is provided with a gaseous flow of air or water.	To improve roasting efficiency. To remove the non-pleasant volatile compounds.	Short roasting time (0.5 to 12min.) This process might reduce or substitute the conching process.	Extra activities required prior roasting: deshelling and grinding.	US Patent number 3904777 (Goerling and Ernst-Zuercher 1975)
2	Roasting a thin layer of cocoa liquor. There are two heating sources: firstly, heat is generated in the paste by friction; then, the paste is lifted out of the layer and sprayed with hot air.	To improve roasting efficiency. To remove the non-pleasant volatile compounds.	Short roasting time (1 to 5 min.).	Extra activities required prior roasting: deshelling and grinding.	US Patent number 4389427 (Schmitt and Birkeneck 1983)
3	Fluidized bed roaster.	To improve roasting efficiency. To improve chocolate flavor.	Roasting time no longer than 4 min (16 times faster than oven roasting). Known as a low-carbon footprint method. Higher production of pyrazines in contrast to oven-roasted cocoa.	Cocoa beans need to be deshelled. Then cocoa nibs have to be cracked into a proper size.	Peña-Correa et al. (2022)
4	A combination of superheated steam with convective oven roasting to raise the chamber's temperature because of the high heat transfer capability of water steam.	To preserve tocopherols, phytosterols, and the antioxidant capacity of the lipid fraction of cocoa beans.	The humid air in the roasting chamber helped to preserve more tocopherols and phytosterols. The loss of cocoa butter migrating toward the shells was reduced.	The roasting time was longer when steam was present in the roasting chamber. Therefore, it might need higher energy.	Oracz, Nebesny, and Żyżelewicz (2014)
5	A combination of superheated steam with convective oven roasting to raise the chamber's temperature because of the high heat transfer capability of water steam.	To test the applicability of the new technology to cocoa beans.	The presence of steam induced similar changes in proximal composition, and similar features in reduction of flavan-3-ols in cocoa beans, as roasting with a dry-atmosphere.	The roasting time was longer when steam was present in the roasting chamber. Therefore, it might need higher energy.	Oracz and Nebesny (2019)
6	Microwaves (MW)	To test the applicability of the new technology to cocoa beans.	<ul><li>Short roasting time (12.5 min). About three times faster than oven roasting.</li><li>Significant reduction of total acidity and volatile acidity in cocoa nibs.</li><li>Fewer losses of cocoa butter toward the shells.</li><li>MW holds the capability of instantaneously applying and removing the heat source.</li></ul>	This technique alone is expensive, and results in the formation of hot spots in some food products. In order to mitigate the costs and the lack of temperature uniformity, this technique might be combined with other conventional heating methods (e.g., hot air, infrared, vacuum).	Krysiak (2011) and Awuah, Ramaswamy, and Tang (2014)

conventional roasting process have been proposed either to improve efficiency or to preserve thermolabile compounds. Table 3 gathers those valuable studies.

Even though it is well known that heat transfer is more efficient when the particle size is reduced to cocoa mass (Goerling and Ernst-Zuercher 1975; Schmitt and Birkeneck 1983) or cocoa nibs (Peña-Correa et al. 2022), many cocoa manufacturers and scientists still practice whole-cocoa bean roasting because it makes shells-winnowing easier without pre-roasting. Large companies deal with tons of cocoa per day; thus, continuous processes are more convenient than batch processes. This reasoning explains why fast roasting processes such as thin-layer roasting (Table 3, entries 1 and 2) and fluidized bed roasting (Table 3, entry 3) are not popular, although they were invented more than 40 years ago. We recently demonstrated that fluidizing bed roaster boosted the formation of pyrazines (chocolate-aroma compounds) in cocoa nibs without carrying the formation of typical over-roasting off-flavors (Peña-Correa et al. 2022); thus, this technique deserves reconsideration.

The injection of superheated steam during convective oven roasting preserves some lipid-soluble antioxidant compounds in cocoa butter like the tocopherol isomers  $\alpha$ ,  $\gamma$ , and  $\delta$ , and the phytosterols  $\beta$ -sitosterol, stigmasterol, campesterol, and  $\Delta^5$ -avenasterol (Table 3, entry 4). The significant losses of those lipophilic phytochemicals were detected from the very mild roasting temperature of 110 °C. The losses were higher when increasing the temperature. The degradation of tocopherols and phytosterols was less advanced when roasting under the relative humidity of 5% than during dry roasting (Oracz, Nebesny, and Żyżelewicz 2014). The researchers claimed that the formation of a steam barrier around the beans protects them from contact with oxygen.

The protective effect of water steam on tocopherols and phytosterols seems not to work for the most appreciated phytochemicals in cocoa, the hydrophilic ones: epicatechin, epigallocatechin, procyanidins B1, B2, B5, and C1 (Table 3, entry 5). In the study presented by Oracz and Nebesny (2019), all the flavan-3-ols decreased significantly after thermal treatment, with or without steam inside the roasting chamber. Catechin was the only flavan-3-ol that increased upon roasting; however, its occurrence was not affected by the hot steam. Furthermore, the presence of steam slowed the roasting process, making this technique less attractive for further industrial scaling.

Infrared radiation is a common alternative in food processing (Aboud et al. 2019). However, its application in cocoa has been limited to the deshelling process. It might be due to the low wavelength of the infrared spectrum, which is not enough to penetrate the center of the kernels; therefore, it only causes vibrating movements of the surface-located water molecules (Aboud et al. 2019). Radiation energy with higher wavelengths, e.g., microwaves (MW), gets better penetration to the inside of solid foods. Only one study was found to use MW in the roasting process of cocoa (Table 3, entry 6). The study of Krysiak (2011) only included one MW roasting condition (700 W, 12.5 min), which resulted in significant reductions of total acidity and volatile acidity in cocoa nibs, and fewer losses of cocoa butter toward the shells, in contrast to convective-roasted cocoa. The FFA, SV, PV, refractive index, and viscosity of the cocoa butter obtained from MW- and convective-roasted beans were not significantly different. Only the IV showed lower values for MW-roasted beans, which was reported to be probably due to the degradation of triacylglycerols.

Krysiak (2011) provided important insights into the use of microwaves in cocoa roasting. However, only one power condition was tested among the broad microwave spectrum. Pramudita et al. (2017) roasted peanuts by MW with different power levels. They found that the moisture loss, the browning, and the formation of micropores on the surface of the peanuts tended to increase when increasing MW-power. In addition, they demonstrated a shorter roasting time compared with drum roasting. A similar study with cocoa beans or nibs would provide valuable information for the use of MW as alternative roasting technique for cocoa.

#### **Recommendations for future research**

There are numerous studies in the literature providing insight into cocoa roasting from different aspects. This review aimed to critically evaluate these studies, draw attention to the important points or limitations, and provide recommendations for future work. The first recommendation would be that scientists should make proper use of the words 'beans' and 'nibs.' Despite the differences between them (as described in this review), there are many imprecisions in the published papers.

To properly compare the lab outcomes to real industrial situations, it is necessary to conduct experiments toward an equal water content of cocoa beans/nibs below 2%. This limit is optimal for further processing, like grinding and fat extraction. Values above 2% are considered incomplete roasting. Water excess in cocoa nibs causes rheological problems in cocoa liquor, affecting its flowability. Moreover, when preparing a chocolate mass, a water content higher than 2% causes difficulties in the dispersion of solid ingredients such as sugar and milk powder into the continuous cocoa butter phase.

A considerable amount of literature has published lab-scale investigations, while few research have reported the results of industrial or pilot-scale experiments. This gap might be due to the need to protect industrial know-how. However, scaling lab results up to industrial levels is very important for properly using the generated knowledge.

As reported in this review, the physicochemical properties of cocoa are more affected by roasting time than by roasting temperature. Novel and alternative cocoa roasting techniques should rather focus on accelerating this process. So far, this goal has been achieved by microwaves, fluidized bed roasting, and thin-layer roasting techniques. Finally, energy efficiency and sustainability should also be considered when a new roasting technique is implemented in cocoa processing.

#### **Author contributions**

Ruth Fabiola Peña Correa designed the format, searched and classified sources of information, drafted the manuscript, and acquired the funding. Burçe Ataç Mogol searched and classified sources of information, supervised the investigation, validated the information, and reviewed and edited the manuscript. Vincenzo Fogliano supervised the investigation, validated the information, and reviewed and edited the manuscript.

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The authors report there are no competing interests to declare.

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