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Coconut sugar (*Cocos nucifera* L.): Production process, chemical characterization, and sensory properties

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ABSTRACT

Due to an increased interest in healthy diets, people try to replace conventionally refined sugar. Consequently, the demand for alternative sweeteners is growing. Coconut blossom sugar benefits from this trend and is conquering European grocery stores and kitchens. While sugar extraction from coconut sap has a long tradition in South and Southeast Asian, not much is known in Europe about its manufacturing process. The purpose of this study was to characterize this product. One-hundred-and-seven coconut blossom sugar samples, purchased in local grocery stores, online or provided by producing companies and traders, were analyzed with regard to sensory properties, $^{13}C/^{12}C$ isotopic ratio, sugar content, gluten level, microscopic characteristics, and lipid profile using well-established methods. The assessment showed that coconut blossom sugar may contain gluten, starch, an addition of sugars from C4 plants, and coconut or palm oil. These additives can have technological reasons such as anti-foaming agents and release agents, or simply serve to increase the yield. Even if it is a natural product with natural variations, the smell and taste of caramel are particularly characteristic.

1. Introduction

Coconut blossom sugar is a sweetener traditionally used in South and Southeast Asian cuisine, e.g., Indonesia, Philippines, India (Levang, 1988). It is produced from the phloem sap of the blossom of the coconut palm tree (*Cocos nucifera* L.) (BAFPS, 2010). Juice collectors climb up the palm trees and cut off the unopened inflorescences with a sickle. The escaping sap is collected in plastic or bamboo containers for 8–12 h. Sometimes lime is added to prevent the sap from fermenting (Hebbar et al., 2015; CRI, 1967 cited by; Dalibard, 1999). The collected sap is then heated over an open fire, stirred constantly until it thickens and crystallizes (Levang, 1988). Due to the manufacturing process, the color of the sugar can vary from light to dark brown. Finally, the sugar is sieved and hand selected to obtain a fine-grained product (PCA, 2015).

Each coconut palm tree produces an average of one inflorescence per month. Each inflorescence gives about 1.5 L of sap a day, which can be harvested twice a day, once in the morning and once in the evening. The fresh coconut sap contains approximately 15 g sugar/100 g, so that after boiling 200 g sugar per day per inflorescence can be produced (Hebbar et al., 2015).

Coconut palm trees can already be used for sap collection at a young age. Each time, tapping and harvesting the phloem sap one to two millimeter of the spadix need to sliced-off. This process can be continued until the spadix is reduced to a stump. This way, a single spadix can be tapped for 40–45 d. A coconut palm tree can be tapped over 20 years (Hebbar et al., 2015; Levang, 1988).

In addition to coconut blossom sugar, many other products can be produced from the sap, e.g., coconut vinegar, coconut toddy, or coconut syrup (Redhead, 1989 cited by; Dalibard, 1999). Just a few years ago, coconut blossom sugar was almost unknown in Europe. Meanwhile, the product is also conquering grocery stores and kitchens there.

Consumers often try to replace refined sugars with alternative sweeteners such as coconut blossom sugar, because of an increased interest in healthy diets and the negative public attention focused on high sugar consumption. Traders advertise the fact that coconut blossom sugar is traditionally produced by small farmers, the palm trees grow organically in mixed cultivation with further crops, and the sugar contains little fructose and has a lower glycemic index than conventional refined cane or beet sugar (CBI, 2016). Consumers are prepared to pay high prices for coconut blossom sugar. The price for a kilogram varies between 15 and 46 €. To compare, a kilogram of conventionally refined sugar only cost 0.70 € in 2017 (CBI, 2016). However, it can also be sold as palm sugar without specification of the palm type (CBI, 2016).

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As coconut blossom sugar is still a niche product in many countries, not much is known about the manufacturing process and the product characteristics themselves. Previous work has focused mainly on the fermentation process of coconut sap, the associated microorganisms (Atputharajah, Widanapathirana, & Samarajeewa, 1986), and the resulting changes in pH, sugar content (Hebbar et al., 2015), and the composition of volatile compounds (Borse, Rao, Ramalakshmi, & Raghavan, 2007). There are only a few works dealing with coconut blossom sugar (Apriyantono, Aristyani, Lidya, Budiyanto, & Soekarto, 2002; Purnomo, 1992).

The aim of the present study was to characterize the product in more detail for drawing conclusions about the manufacturing process, also with regard to possibilities of authentication. Well-established methods such as enzymatic sugar determination, $^{13}C/^{12}C$ isotopic analysis, gluten determination, microscopic analysis, screening of lipid profile using UPLC-QToF-MS, and sensory description, have been used at a large sample set, covering different places of purchase for coconut blossom sugar.

2. Material and methods

2.1. Material

The sample material was purchased in local grocery stores, online and provided by producing companies and traders. In total, 107 coconut blossom sugar samples, in a price range from 9.16 to 59.60 € per kilogram and different appearance, were available for analysis. Of these, 79 samples came from Indonesia, 6 from the Philippines and 22 had no indications of origin.

2.2. Sensory properties

For the description of the sensory properties of coconut blossom sugar a simple descriptive test according to the German official methodology described in § 64 LFGB (German Food and Feed Code) L 00.90–6:2015-06 based on DIN (German Institute for Standardization) 10964:2014-11 (BVL, 2015) was used. For this evaluation, a panel of 18 trained test persons, comprising of 13 females and five males, with an age from 20 to 45 years was selected. The panel was asked to describe appearance, smell, taste, and consistency of the samples with freely selectable attributes.

In order to describe the sensory properties of coconut blossom sugar and to distinguish it from other sweeteners, 24 samples were tested, including three coconut blossom sugars, four coconut blossom syrups, three palm sugars, one cane sugar, one whole cane sugar, one date sugar, one date syrup, one rice syrup, two apple syrups, one pear syrup, one agave syrup, three treacles, one barley malt extract, and one honey.

All samples were presented to the testers in plastic cups with a three-digit random code.

2.3. Microscopic analysis

The Zeiss Primo Star light microscope (Carl Zeiss Microscopy GmbH, Jena, Germany) was used to analyze coconut blossom sugar samples prepared in water, diluted iodine solution, and chloral hydrate solution at 100 to 400 times magnification. The microscope camera Axio Cam ERc 5s (Carl Zeiss Microscopy GmbH, Jena, Germany) was used for documentation.

2.4. Analysis of gluten

Coconut blossom sugar samples were tested in duplicate using the RIDASCREEN® Gliadin kit (R-biopharm AG, Darmstadt, Germany) and a microplate reader (GENios, TECAN Group Ldt., Männedorf, Switzerland). The test is based on a sandwich R5 ELISA (enzyme-linked immunosorbent assay) and has been endorsed by the Codex Alimentarius as a type 1 method (CAC, Adopted in 1979, Amendment in 1983 and 2015, Revision in 2008).

2.5. Moisture determination

The moisture content was determined by drying up to a constant mass at 103 °C (DRY-Line Trockenschrank DL 53, VWR International BVBA, Leuven, Belgium) according to the German official methodology described in § 64 LFGB L 39.00–1:1981-04 (BVL, 1981).

2.6. Enzymatic sugar determination

The content of glucose, fructose, and sucrose was determined with the enzymatic test kits Enzytec™ Liquid D-Glucose, Enzytec™ Liquid Sucrose/D-Glucose, and Enzytec™ Liquid D-Glucose/D-Fructose (R-biopharm AG, Darmstadt, Germany). The assays were carried out according to the manufacturer's instructions using a fully automatic analyzer (ChemWell® 2910, Awareness Technology Inc., Palm City, USA).

2.7. $^{13}C/^{12}C$ isotopic analysis

Coconut blossom sugar samples were sent to a laboratory specialized in isotope analysis (Agroisolab GmbH, Jülich, Germany). The samples were analyzed in duplicate using an elemental analyzer (NA 1500 series II, Carlo Erba Instruments S.r.l., Milano, Italy) interfaced with an isotope ratio mass spectrometer (Horizon, Nu Instruments Ltd, Wrexham, UK) according to methodology described by Winkler and Schmidt (1980).

2.8. Lipid profile using UPLC-QToF-MS

2.8.1. Extraction

Extraction was carried out according to Bligh and Dyer (1959). In brief, 1 g sample was mixed with 10 mL chloroform, 10 mL methanol with 0.03 mol/L formic acid, and 9 mL water with 0.03 mol/L formic acid, followed by shaking for 20 min using a shaker (VIBA 330, Collomix GmbH, Gaimersheim, Germany). The remaining suspension was centrifuged at 4816 $\times g$ at room temperature for 5 min (Heraeus Multifuge X3, Thermo Fisher Scientific Inc., Waltham, USA). After centrifugation, the organic layer was filtered using a regenerated cellulose syringe filter (CHROMAFIL® Xtra RC-20/25, MACHEREY-NAGEL GmbH & Co. KG, Düren, Germany) and used for analysis.

2.8.2. UPLC-QToF-MS analysis

Chromatographic separation was conducted using an UPLC (ultra-high performance liquid chromatography) system with a 150 mm × 2.1 mm i.d., 1.7 µm, UPLC BEH C18 column coupled with a 5 mm × 2.1 mm i.d. guard column of the same material (ACQUITY, Waters Corp, Milford, USA) at 55 °C, and a flow rate of 0.3 mL/min. Mobile phase A was water and B was isopropanol/acetonitrile (3:1 v/v), both containing 0.01 mol/L ammonium formate and 0.03 mol/L formic acid. The elution started with 70% B for 3 min, linearly increased to 100% in 15 min and kept constant for 5 min. In 1 min it was brought back to 70% B, followed by 3 min of re-equilibration. The injection volume was 1 µL for all coconut blossom sugar samples; 0.1 µL were injected from reference oils and fats.

For the detection, a QToF-MS (quadrupole time-of-flight tandem mass spectrometer) with an ESI (electrospray ionization) source (VION, Waters Corp., Milford, USA), operating in positive ion mode in the mass range of 100–1000 m/z with a scan time of 0.150 s was used. Mass spectrometry conditions were set as follows: capillary voltage = 3.50 kV; source temperature = 120 °C; desolvation temperature = 550 °C; cone gas = 50 L/h, desolvation gas = 1000 L/h; low collision energy = 6 eV. The acquisition of fragment spectra was carried out using a high collision energy ramp from 10–60 eV in high definition MS^E (MS with elevated

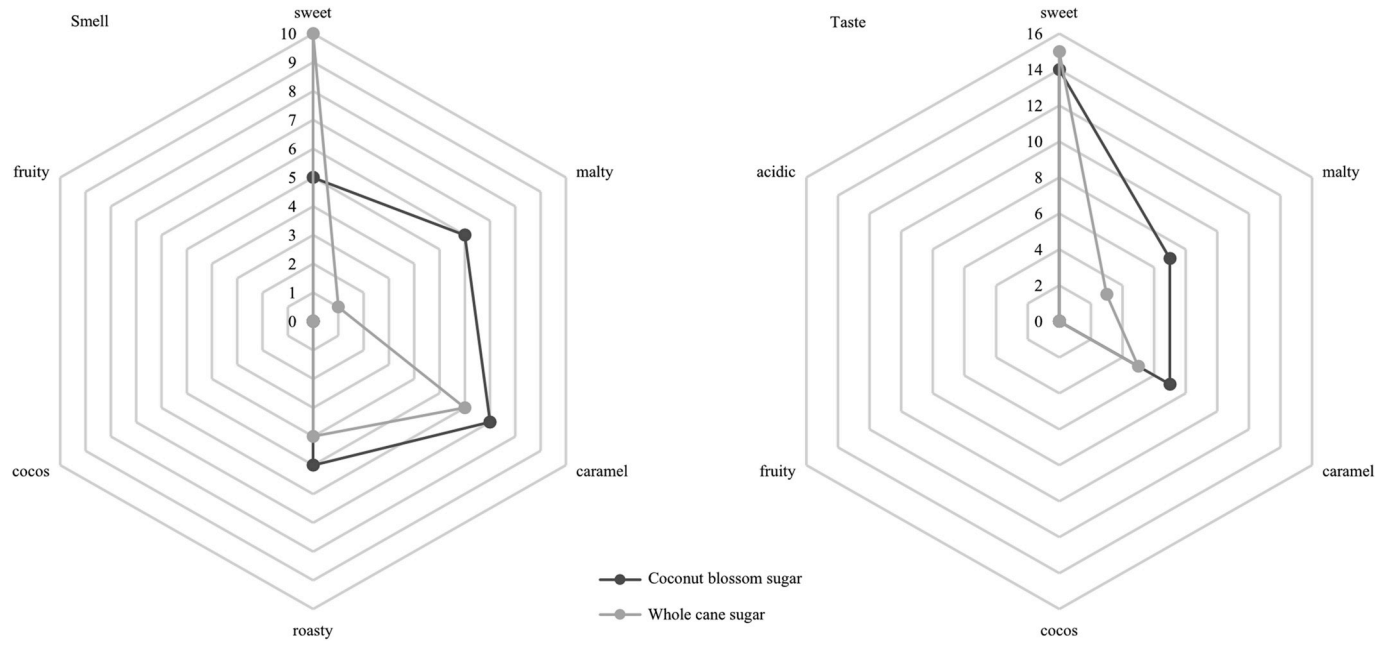

Fig. 1. Taste and smell of coconut blossom sugar compared to whole cane sugar.

collision energy ramp) mode.

Calibration in the respective mass range was conducted with the Major Mix IMS/Tof Calibration Kit (Waters Corp., Milford, USA). Additionally, a lock mass calibration was applied using leucine-enkephalin (Waters Corp., Milford, USA) at a concentration of 0.1 µg/mL dissolved in acetonitrile/water (1:1 v/v), containing 0.03 mol/L formic acid. For the assessment of instrument stability and analytic reproducibility, a pooled sample as QC (quality control) sample as well as a blank sample (acetonitrile) were included in the sample list every 6 injections. Furthermore, the QC and blank sample were injected at least 10 times before initiating the run, in order to equilibrate the column. Samples were injected in random order. The autosampler was set to 10 °C.

Data acquisition, processing, and visualization were carried out using UNIFI Scientific Information System (Waters Corp., Milford, USA).

3. Results

3.1. Sensory properties

Coconut blossom sugar samples were usually brown sugar, whose appearance can vary depending on the price. While more expensive products were light brown and fine powdery, inexpensive products were described by the testers as brown to medium brown and coarse-grained. In addition, expensive products were characterized by a higher pourability. As can be seen in Fig. 1, the smell of coconut blossom sugar was described with the attributes caramel, malty, sweet, and roasty. While the sweet scent predominated in cheap products, the caramel scent was particularly dominant in expensive products. The taste of coconut blossom sugar was mainly described as sweet. For expensive products, the attributes caramel and malty were added to the description. Contrary to the expectations of the testers, coconut blossom sugar had neither a smell nor a taste of cocos.

Apple and pear syrup were described mainly by the attributes acidic, fruity and sweet; treacle and barley malt were dominated especially by a bitter sensation. A strong caramel smell and taste was clearly characteristic for coconut blossom sugar. It differed from whole cane sugar particularly in its malty character. Coconut blossom sugar showed most similarities with palm sugar.

3.2. Analysis of gluten and microscopic analysis

The *Codex Alimentarius Commission* defines gluten levels for gluten-free, gluten-reduced, and gluten-containing foods. Accordingly, gluten-free foods must not exceed a gluten content of 20 mg/kg. A level above 20 up to 100 mg/kg is considered gluten-reduced and a level above 100 mg/kg is considered gluten-containing (CAC, Adopted in 1979, Amendment in 1983 and 2015, Revision in 2008).

Gluten is a protein fraction naturally found in wheat, rye, barley, oats, or their crossbred varieties and derivatives (CAC, Adopted in 1979, Amendment in 1983 and 2015, Revision in 2008). Coconut blossom sugar is naturally gluten-free. As evident from Table 1, 92% of the samples tested had a gluten level less than 20 mg/kg 3% of the samples had a gluten level above 20 up to 100 mg/kg and 5% had a level above 100 mg/kg.

The microscopic images of coconut blossom sugar samples with a gluten level below the quantification limit of 5 mg/kg did not show any special characteristics. As shown in Fig. 2, iodine preparations of the samples tested positive for gluten showed blue stained of 15–40 µm large, roundish to lenticular and 2–10 µm small, isodiametric to round starch granules which are characteristic for wheat. There was a positive correlation between the number of starch granules and the level of gluten.

Table 1
Summary findings of the gluten level of the tested coconut blossom sugar samples.

Category	0 to < 5 mg/kg	≥ 5 to < 20 mg/kg	≥ 20 to < 100 mg/kg	≥ 100 mg/kg
	Below determination limit	Gluten-free	Gluten-reduced	Gluten-containing
n = 36	29 (81%)	4 (11%)	1 (3%)	2 (5%)

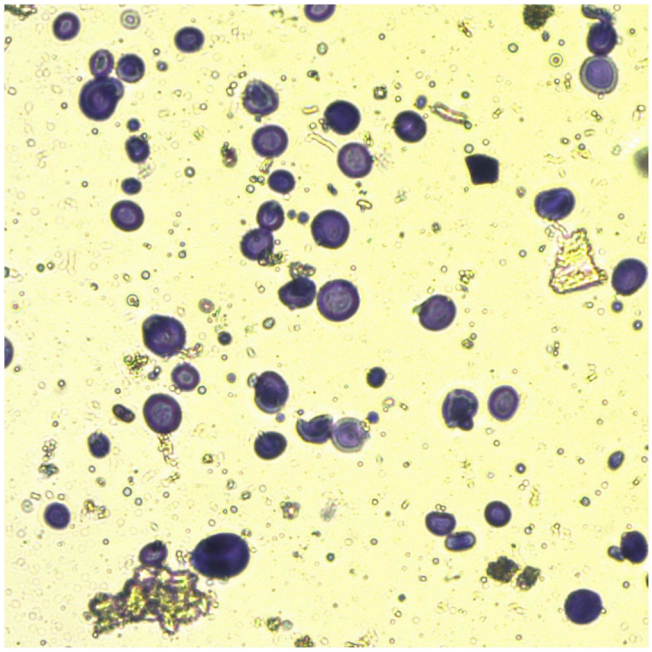

Fig. 2. Microscopic image of the iodine preparation of a coconut blossom sugar with ≥100 mg/kg gluten and an estimated starch content of 0.7 g/100 g. Starch granules are colored in blue. View at 100 times magnification. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

3.3. $^{13}C/^{12}C$ isotopic analysis

$^{13}C/^{12}C$ isotopic analysis can be used to distinguish between different plant groups, the so-called C3 and C4 plants. C3 and C4 plants differ in the photosynthetic process of CO_2 assimilation. C3 plants, which also include coconut palms, fix CO_2 with the participation of the enzyme Rubisco (ribulose-1,5-bisphosphate carboxylase/oxygenase). A C3 body, the so called ribulose-1,5-bisphosphate, is formed. In contrast, C4 plants form a C4 body, oxaloacetate, under the influence of the enzyme PEP carboxylase (phosphoenolpyruvate carboxylase). Rubisco prefers the "lighter" $^{12}CO_2$. Due to this isotopic discrimination, C3 plants have a lower $^{13}C/^{12}C$ isotope ratio than C4 plants. C3 plants are in the $\delta^{13}C$ range from −24 down to −32‰ referred to PDB (Pee Dee Belemnite). C4 plants cover a $\delta^{13}C$ range from −10 to −16‰ (PDB). This difference helps to identify other sources of sugar (e.g., cane sugar) in coconut blossom sugar (Winkler & Schmidt, 1980).

As evident from Table 2, 83% of the samples tested showed $^{13}C/^{12}C$ isotope ratios, which were within the $\delta^{13}C$ range of C3 plants. 7% of the samples showed a relatively enriched $^{13}C/^{12}C$ isotope ratio, which is more likely for C4 sugar addition of about 10–20 g/100 g. 7% showed a significantly enriched $^{13}C/^{12}C$ isotope ratio, being explainable for coconut blossom sugar with C4 sugar addition of 20–50 g/100 g and 3% showed ratios, which are more likely for ≥ 50 g/100 g addition of C4 sugar.

3.4. Moisture determination and enzymatic sugar determination

An evaluation of moisture and sugar content for coconut blossom sugar was carried out on 10 samples. According to these tests, 100 g coconut blossom sugar consists of 81.6–91.1 g sucrose, 1.18–2.39 g moisture, 0.66–2.32 g fructose, and 0.48–2.28 g glucose. As can be seen from Fig. 3, a correlation between C4 addition and glucose-fructose ratio could be observed. Coconut blossom sugar samples, which are in the $\delta^{13}C$ range of C3 plants, have a lower percentage of glucose and therefore, a lower glucose-fructose ratio than samples with C4 addition. For samples with C4 addition < 10 g/100 g, the ratio varied from 0.5 to 0.7, while samples with C4 addition ≥ 20 g/100 g had glucose-fructose ratios from 1.0 to 1.2.

3.5. Lipid profile using UPLC-QToF-MS

In a non-targeted approach, the lipid profile of the 107 coconut blossom sugar samples was analyzed by UPLC-QToF-MS.

As shown in Fig. 4, 3 different lipid profiles could be observed. A dependence between the geographical origin of the samples and the lipid profiles could be determined.

Six coconut blossom sugar samples were declared with "Philippines" as an indication of the geographical origin. Those samples did not show any significant peak in their lipid profile and therefore contained no lipids. 93% of the samples with known origin were labeled with "Indonesia" as geographical origin. Those samples could be divided into two groups: One group showed very intensive peaks in a retention time window from 12 to 18 min. They came from the area around *Yogyakarta*. The samples from the other group came from the area around *Purwokerto* and had intensive peaks between 17 and 18 min.

An unprocessed coconut palm sap from Indonesia did not show any significant peak in the lipid profile. Consequently, lipids are added during the cooking process. By comparison with reference oils and fats, the lipid profiles could be assigned to coconut and palm oil. While coconut oil (intensive peaks in a retention time window from 12 to 18 min) was characteristic for samples from *Yogyakarta*, samples from *Purwokerto* contained palm oil (intensive peaks in a retention time window between 17 and 18 min).

4. Discussion

According to the *Philippine National Standard*, coconut blossom sugar is defined as a sweetener obtained from pure fresh coconut sap and consisting of 78–89 g/100 g sucrose, 1–4 g/100 g fructose, 2–3 g/100 g glucose, 0.5–0.8 g/100 g moisture, and ≤ 2.4 g/100 g ash (BAFPS, 2010). The present study showed that coconut blossom sugar may also contain gluten, starch, an addition of sugars from C4 plants, coconut or palm oil. These additives can have technological reasons. Levang (1988) already mentioned that coconut quarters were added to the sap to prevent it from overboiling during the manufacturing process. Coconuts usually contain 36.5 g/100 g oil (Souci, Fachmann, & Kraut, 2011). It is known from other branches of food production that oil is added to prevent overboiling. This is particularly the case if surface-active substances are naturally contained in the suspension which tend to foam. For example, in beet or cane sugar production, proteins form a foam stabilized by sugar. An overfoaming of the suspension while boiling would lead to a considerable loss of yield. Also a physical removal of the foam would lead to a diminished yield. For that reason, antifoaming agents are necessary additives in sugar production

Table 2
Summary findings of the C4 sugar addition of the tested coconut blossom sugar samples.

Category	0 to < 10 g/100 g[a]	≥ 10 to < 20 g/100 g	≥ 20 to < 50 g/100 g	≥ 50 g/100 g
n = 59	49 (83%)	4 (7%)	4 (7%)	2 (3%)

[a] Within the statistical range of C3 plants. No significant C4 sugar addition (≥10 g/100 g) detectable.

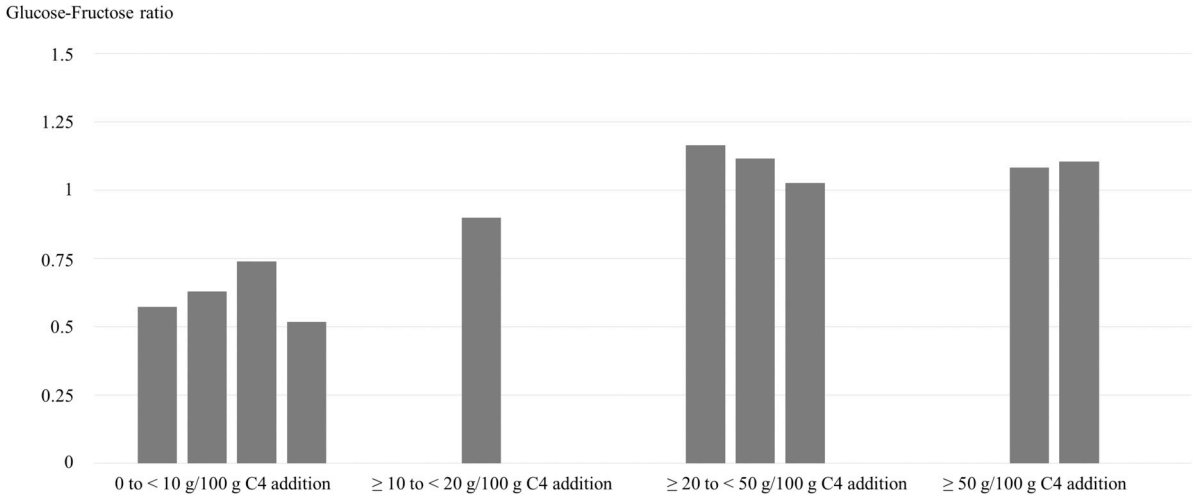

Fig. 3. Correlation between C4 addition and the glucose-fructose ratio for 10 coconut blossom sugar samples.

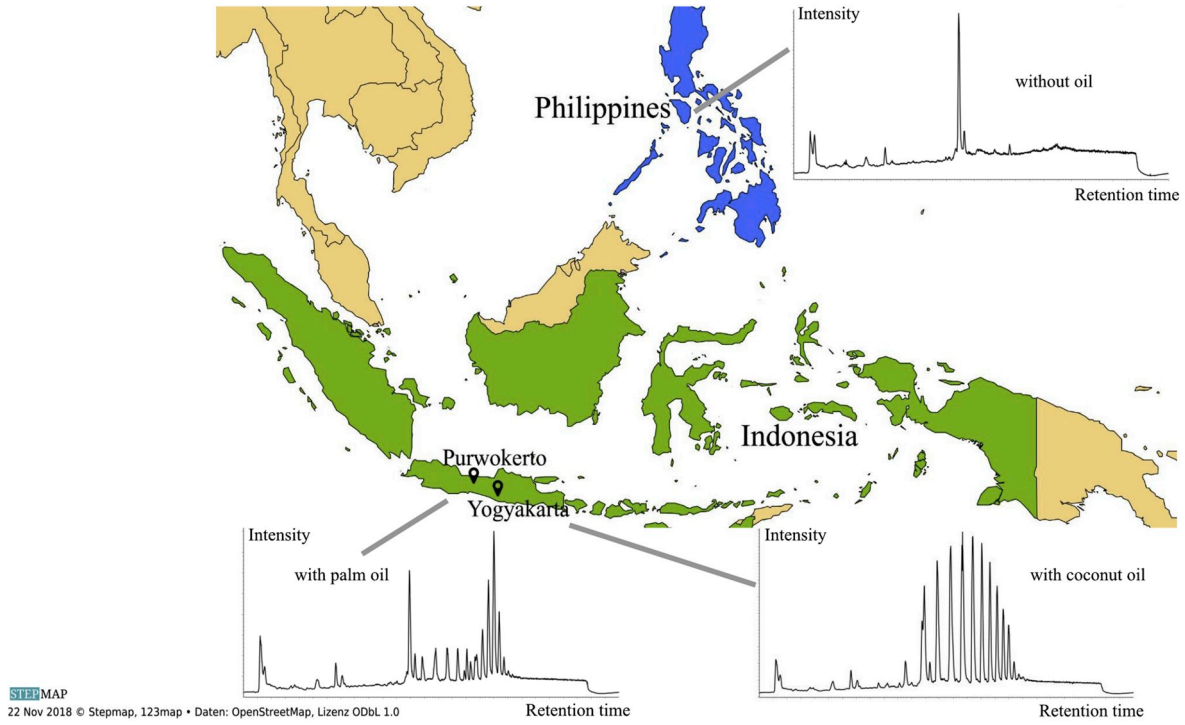

Fig. 4. Lipid profiles of coconut blossom sugar samples according to their geographical origin.

(Schmid, Meffert, Schenker, & Asbeck, 1990). However, common practice seems to differ depending on production country/site: From requests to traders, who produce their own coconut blossom sugar in the Philippines, it is known that no antifoaming agents are added to the coconut sap during the manufacturing process. On the opposite, audits in Indonesia confirmed the use of oil for foam regulation.

The addition of starch can help to maintain pourability. From other areas of food production, it is known that powdery products that tend to absorb moisture from the environment, are added so-called release agents.

Coconut blossom sugar also tends to absorb moisture from the ambient air and to agglutinate. Clumped coconut blossom sugar is difficult to dose. Added starch also absorbs moisture, keeps the sugar dry and prevents it from agglutination. By adding wheat starch, gluten is also added to the product. This can cause problems for consumers who are sensitive to gluten (e.g., as in celiac disease).

For crystallization of the sugar during the boiling process, it is important that a supersaturated solution is present and that it is continuously stirred at a moderate, uniform speed. It is known from cane and beet sugar production that, in order to save time and achieve uniform crystal sizes, the crystallization of the sugar-containing suspensions is promoted with seed crystals (Hoffmann, Mauch, & Untze, 1985). This can explain the addition of sugar from C4 plants, detected in some of the coconut blossom sugar samples. However, high levels of C4 addition (≥ 20%) are hypothesized to indicate food stretching. Coconut blossom sugar is a high priced product. Consumers are prepared to pay 20 to 65 times more money than for conventional refined sugar (CBI, 2016). Unfortunately, high priced products are most prone to fraud. In times of globalization and complex supply chains, food fraud is becoming an increasing problem all over the world and the substitution of sugar rich products with cheaper sugar sources without correct labeling is not untypical (Moore, Spink, & Lipp, 2012). For example, starch-based sugar syrups, high-fructose corn syrup, glucose syrup, and sucrose syrups, which are produced from sugar beet or cane

sugar, are used for adulterating honey. The addition of other sugar sources can cause changes of the ratios of glucose, fructose, and sucrose and it can change the $^{13}C/^{12}C$ isotope ratio. While the addition of cane sugar (C4 plant) and high-fructose corn syrup (C4) can be detected with $^{13}C/^{12}C$ isotope analysis, the addition of beet sugar (C3) is difficult to identify and poses a major problem (Tosun, 2013; Winkler & Schmidt, 1980).

Nevertheless, it must be taken into account that coconut sap is a natural product whose composition is subject to genotype (coconut variety) and ecophysiological factors such as season, weather, soil, plant nutrition. Further, the manufacturing process is still carried out according to indigenous knowledge and tradition in many places (Dalibard, 1999; Pethiyagoda, 1978, pp. 86–91 cited by; Dalibard, 1999). This can also explain variation in smell, taste, and appearance of the different coconut blossom sugar samples. However, taste and smell of caramel are particularly characteristic of coconut blossom sugar. In its sensory properties most similar to coconut blossom sugar is palm sugar. Palm sugar can be made from different palm species, *Borassus flabellifer* L., *Nypa fruticans* Wurmb and *Arenga pinnata* (Wurmb) Merr. Coconut blossom sugar can also be sold as palm sugar. This makes a distinction between the two products very difficult, especially as both products are produced very similarly (CBI, 2016; Dalibard, 1999).

5. Conclusion

The tests carried out on numerous coconut blossom sugar samples have shown that coconut blossom sugar may contain additives of sugars from C4 plants, starch, gluten and oil. For example, 17% of the samples contained a significant addition of C4 sugar ($\geq$ 10 g/100 g) and 8% of the samples contained gluten $\geq$ 20 mg/kg. These additives can have technological reasons as well as serve to increase the yield. When assessing the product, it must always be taken into account that it is a natural product which is manufactured under non-standard conditions. For a clear authentication, the manufacturing processes have to be taken into account.

Author contribution

Jasmin Wrage designed the study and performed the experiments (60%). Stephanie Burmester supervised and evaluated the sensory analysis (20%). Jürgen Kuballa and Sascha Rohn were involved in writing the manuscript (each 10%).

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