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Research Article

Ponkan essential oil from peels and agro-industrial waste of *Citrus reticulata* Blanco

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Abstract

The tangerine juice corresponds to 47 wt.% of the fruits and it is normally extracted by pressing generating agro-industrial waste rich in compounds with market value. Thus, the objective of this study was to obtain the essential oil from *Citrus reticulata* Blanco peels and compare it with those obtained from the waste of the ponkan tangerine juice industry, rich in *d*-limonene. The waste and peels were cut into pieces of approximately 1 cm², dried in an oven with air circulation at 50 °C for 15 h, ground, and classified by sieving with particle size between 20 to 28 *mesh*. The moisture content in dried waste and peels were 8.5 wt.% and 6.0 wt.%, respectively. A particle size was defined for the extractions. The essential oil extractions were performed by hydrodistillation, in Clevenger method, with 50 g of sample, 500 mL of distilled water for 2 h. The essential oil yield was 0.22 wt.% (88.5 % of *d*-limonene) for ponkan waste and 1.65 wt.% of for ponkan peel (89.7 % of *d*-limonene). The results showed that separation of the Ponkan peel before processing the fruit enables more efficient extraction of the compounds of interest.

1. Introduction

Ponkan peels essential oil has the same characteristics as the essential oils of citrus, which present high antioxidant, fungicidal, and mainly bactericidal capacity [1–3]. This is characterized, as green oil, yellow oil, and red oil, according to the degree of maturity of the fruit and the extraction method used [4]. Furthermore, these oils are applied in several areas, such as the perfumery, food and beverage, cleaning materials and pharmaceutical industries [5]. Kwangjai *et al.* [6] reported that Ponkan peel essential oil contains anxiolytic compounds that modify brain waves (95.7% *d*-limonene), contributing to the sleep phase of increased physical energy recovery occurring more often and for longer periods. Similar properties to the essential oil of tangerine or mandarin (*Citrus reticulata*) and bergamot (*Citrus bergamia*), which have antidepressant, tranquilizing, calming and sedative properties [7]. Ferreira, Silva and Nunes [8] concluded that *Citrus reticulata Blanco* peel extract has antioxidant properties due to the high *d*-limonene content. Dosoky and Setzer [9] reported protective activity against pulmonary fibrosis (74.2% *d*-limonene). Oliveira *et al.* [10] showed the efficacy of Ponkan peel essential oil against *Leishmania amazonensis*, and Zhang *et al.* [11] proved the bactericidal activity of this same essential oil.

The chemical composition of essential oils is genetically determined, but can be altered due to environmental stimuli, causing selectivity or the biosynthesis of other compounds.

Table 1. Chemical composition of the essential oil from *citrus reticulata* Blanco

Majority components, 5.1 to 96.0%	Intermediate components, 1.0 to 5.0%	Minority components, 0.0 to 0.9%	Country of study	References
d-Limonene, γ-Terpinene	myrcene	α-tujene, α-pinene, sabiene, trans-β cymene, citronellal, geraniol, geranial, α -terpineol, limonene oxide	Brazil	[16]
d-Limonene	γ-terpinene	-	India	[15]
d-Limonene	β-pinene, -3-carene	α-pinene, β-fellandrene	Thailand	[6]
d-Limonene	β-pinene, γ-terpinene	α-tujene, α-pinene, sabienol, myrcene, *p*-cymene, terpinolene, linalool, *n*-decanal	Brazil	[5]
d-Limoneno, γ-Terpinene	α-pineno, β-pineno, β-mirceno	Sabieno, *p*-cimeno, *n*-metil antranilato de metilo	Brazil	[17]
d-Limoneno, γ-Terpinene	β-mirceno	α-tujeno, α-pineno, sabieno, β-pineno, α-terpineno, *o*-cimeno, terpinoleno, terpinen-4-ol, α-terpineol, metil éter timol	Brazil	[18]

Among these stimuli are the plant interaction with other living beings, the time of the year and the harvest time. Furthermore, different abiotic stresses, such as drought, concentration of salts and heavy metals in the soil, temperature, and UV radiation can reduce the uptake and diffusion of CO_2 and alter the different biochemical reactions of the plant. Different stimuli affect photosynthesis and essential oil production and consequently its chemical composition [12–14].

The essential oil of *Citrus reticulata* has 70% to 85% *d*-limonene, 5% linanool and 2% furanocoumarins [7], and regarding the essential oil from Ponkan peels, as expected, studies by different authors have shown that *d*-limonene is the main component in the essential oil. According to Kwangjai *et al.* [6] and Singh *et al.* [15] it ranges from 89% to 95% in composition. Table 1 presents the chemical composition in terms of major compounds found in essential oils from Ponkan peel.

Besides *d*-limonene and other terpenes can be identified essential of Ponkan peel, such as *γ*-terpineno, *β*-pinene, *γ*-3-carene, β-myrcene, in amounts ranging from 1% to 5%. Terpenic alcohols (linalool, terpinen-4-ol, *α*-terpineol), esters (*n*-methyl anthranilate), aldehydes (*n*-decanal) and methyl ether phenols (methyl ether thymol) are present in amounts less than 1%.

d-Limonene ($C_{10}H_{16}$), the major component of essential oils from Ponkan peel is an aliphatic hydrocarbon (non-oxygenated monoterpene, cyclic and consisting of two isoprene units), colorless, non-toxic, and identified as the main component of essential oils from different citrus species. Being known for its pleasant citrus fragrance it is commonly used as a flavoring and antioxidant in beverages and foods [15,19].

Because of this, it has applications in numerous industries, such as in perfumes, paints, component of bio-pesticides [20]. Besides the organoleptic and antioxidant properties, *d*-limonene has therapeutic characteristics, being an adrenocortical stimulant, expectorant, anxiolytic, antiviral, digestive, anti-inflammatory and chemo-preventive [7]. As a raw material for the natural production of terpenoid flavors and fragrances, *d*-limonene can be converted into several high value-added compounds, such as *α*-terpineol, menthol, carvone, limonene-1,2-diol, and perillyl alcohol [21–23]. *d*-Limonene can be obtained from fruit peel and citrus waste by extraction techniques such as hydrodistillation [15, 24] and this is the focus of this study.

Hydrodistillation is a traditional essential oil extraction technique that the matrix is immersed in water and the system is heated to evaporation, where the water vapors carry the essential oil particles. The simplicity of the equipment and its high selectivity are positive aspects of this approach. With the use of the Clevenger-type apparatus it is possible to extract and separate volatile and non-volatile compounds present in different plant species [25–28]. However, there is a lack of information on the extraction of Ponkan essential oil from agroindustrial waste in the literature. Thus, the present work aims to compare the yield of essential oil and the compounds extracted of the peels and the waste of Ponkan.

2. Materials and methods

The waste of ponkan (PW) was purchased in the region of Cerro Azul, state of Paraná, Brazil. This raw material consisted of peel, seeds and pulp and was cut into pieces of approximately 1 cm^2. *Citrus reticulata* Blanco fruits were purchased in the same region as PW. The peels were manually separated from the fruit and cut into pieces of approximately 1 cm^2, thus this raw material (named PP) consisted only of peels. The raw materials were dried in an oven with air circulation at 50 °C for 15 hours until they obtained moisture below 10 wt% [29], ground in a Willey-type knife mill and classified into two fractions (Tyler between 20 to 28 and 28 to 35 *mesh*). Moisture was determined according to Sluiter *et al.* [30] in a Shimadzu infrared balance (Model ID200). The analyses were performed in triplicate, using approximately 1.0 g of sample. The essential oil was extracted by hydrodistillation in Clevenger apparatus, for 2 h, using 50 g of dry solid (m_{ss}) and 500 mL of distilled water (V_w) [2]. PW extractions were performed for two fractions with different particle diameter to verify the influence of surface area on extraction yield, while PP extractions used only 20 to 28 mesh. The amount of essential oil obtained (m_E) was determined in a Radwag AS C/220/2 analytical balance and then dried with $NaSO_4$ and stored in amber flasks at - 4 °C. Fig. 1 shows the Clevenger used (A) and the oil obtained (B).

EO yield (η_{EO}) of the extractions was obtained by the ratio between the mass of EO (m_{EO}), and the mass of dry solid (m_{DS}) on wet basis (WB) and dry basis (DB), in which the moisture (x_w) was considered, according to Equations 1 and 2.

$$\eta_{EO} = \frac{m_{EO}}{m_{DS}} \times 100 \quad (1)$$

$$\eta_{EO} = \frac{m_{EO}}{m_{DS} x_W} \times 100 \quad (2)$$

The essential oil was analyzed by gas chromatography mass spectroscopy (GC-MS), after dilution in chromatographic grade hexane (Panreac - UV-IR-HPLC). Chromatographic analyses were

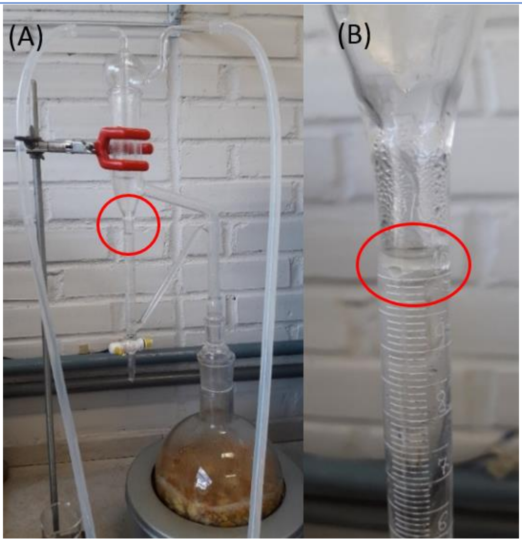

Figure 1. Hydrodistillation (**a**): Clevenger apparatus, (**b**): Essential oil (detail) and water in the decanter.

performed on a Shimadzu TQ8040 chromatograph with a ZB-5MS column (30 m x 0.25 mm x 0.25 μm) following a method proposed by Ozturk, Winterburn and Gonzalez-Miquel [31] for identification and quantification of d-limonene. The NIST (National Institute of Standards and Technology) mass spectrometry library was adopted for the identification of the other compounds present. The 30 largest peaks were considered for quantification, with a minimum area of 100.000 ua and the mass fraction of each compound was calculated from the area of each peak using Postrun GC-MS software.

The *d*-limonene mass (m_L), in g, was calculated with the mass fraction of *d*-limonene (m_L) present in the EO (m_{EO}) as shown in Equation (3).

$$m_L = x_L m_{EO} \quad (3)$$

The *d*-limonene selectivity was calculated according to Equation 4.

$$S_L = \frac{m_L}{m_{EO} - m_L} \quad (4)$$

To verify the possible profitability of the process, a simplified calculation was performed for the production of essential oil (P_{EO}), according to Equation 5.

$$P_{EO} = m_P (1 - x_J)(1 - x_W) \frac{\eta_{EO}}{100} \quad (5)$$

In which:

m_p= annual Ponkan production (tons)

x_j = juice mass fraction in fruits

x_w = moisture mass fraction of waste

η_{EO} = essential oil mass fraction yield

3. Results and discussion

3.1 Characterization of raw material

The characteristics shown of PW and PP are in Table 2. It can be observed that the final moisture content of PW was higher than PP due to the initial characteristics of this raw material that contains much higher water content (77.20 ± 0.60%), since it is obtained after the juice extraction process.

Table 2. Characteristics of the raw materials used to obtain the essential oil.

Characteristic	Agroindustrial waste (PW)	Fruit Peel (PP)
Color	Orange	Orange
Aroma	Citric	Citric
moisture raw material (wt%)	77.20 ± 0.60	64.17 ± 0.02
Moisture dry material (wt%)	8.5 ± 0.4	6.01 0.06

3.2 Essential oil extraction

The essential oil of *Citrus reticulata* Blanco obtained from PW and PP for the particles sorted between 20 and 28 *mesh* was extracted by hydrodistillation. Due to the small volume (0.2 mL) of essential oil obtained from PW, because of the mixed composition of PW, there was difficulty in handling the essential oil.

According to Farrer-Halls [19] the essential oil of *Citrus reticulata* Rutaceae, commonly known as tangerine, has an orange-yellow color with sweet and citrusy top notes - delicate but intense. The essential oil obtained from *Citrus reticulata* Blanco (Ponkan), for both PW and PP, had yellow-orange color and citrus aroma and are like that obtained from mandarin. After 2 hours of hydrodistillation, an average of 1.5 mL of essential oil was obtained from the peels (related to 50.24 g of peel) and 0.2 mL from the agro-industrial waste (related to 50.08 g of solids). $NaSO_4$ was used to remove the water entrained during the process. The removal of moisture was necessary so as not to interfere with the composition of the essential oil obtained. The yields obtained for the two raw materials used in this study are presented in Table 3.

Although several studies have been developed for the valorization of *Citrus* industrial waste, there are few studies on obtaining essential *Citrus reticulata* Blanco oil from peels and waste by hydrodistillation [32–34]. Essential oil yield of 3.186% (WB) was obtained for PP, a value 7.3 times higher than that obtained for the waste- approximately 0.44% (DB). Higher yield for PP was expected, because PW is composed of peel (50 wt%), pulp (45 wt%), and seed (5 wt%), thus the yield of essential oil in PP will be higher for the same mass of dry solid extract.

According to Sharma and Vashist [32], the essential oil yield of *Citrus reticulata* Blanco peels was 1.5%$_{wt}$ (WB), and according to Hou *et al.* [2] the essential oil yield is 3.1%$_{wt}$ (DB). Those authors performed the extractions for 2 hours, however, the first study used powdered peels and the second used particles between 20 and 30 *mesh*. In this study for 20 to 28 *mesh* it was obtained approximately 3.0%$_{wt}$ (DB), a similar value to that obtained by Hou *et al.* [2].

According to Fetzer *et al.* [35] and Xiong and Chen [36] particle diameter interferes with the mass transfer rates and diffusion of the extraction. Thus, it is inferred that smaller particle size could enhance the mass transfer and improve the extraction yield of the essential oil obtained from PW. To evaluate this possibility, hydrodistillation of PW was performed for particles with diameters of 28 to 32 *mesh*. The results compared to those obtained for 20 to 28 *mesh* are presented in Table 4.

As can be seen in Table 4, there was no change in the essential oil yield with the variation of the particle size range. Ranges below 20 to 28 *mesh* were not evaluated, because during milling there is agglomeration of fines making separation difficult and with a probable loss of essential oil by friction with the equipment. This could justify the low yield obtained by Sharma and Vashist [32] that used peel powder for extractions.

3.3 Chemical profile

The Ponkan essential oils obtained for PW and PP (20 to 28 *mesh*) were analyzed by GC-MS. The semi-quantitative method was adopted to quantify the chemical composition of the essential oil, so it was possible to compare the fraction of *d*-limonene in the

Table 3. Results of the extractions of Ponkan (PW) waste and peel (PP) by hydrodistillation (20 to 28 *mesh)*

Sample	m_S (g)	m_{EO} (g)	V_{EO} (mL)	d_{EO} (g/mL)	η_{EO} (%WB)	d_{EO} (%DB)
PW	50.08 ± 0.07	0.11 ± 0.01	0.20 ± 0.01	0.55 ± 0.02	0.399 ± 0.002	0.436 ± 0.002
PP	50.24 ± 0.34	0.83 ± 0.01	1.50 ± 0.01	0.56 ± 0.01	2.970 ± 0.200	3.186 ± 0.013

m_S: solid mass, m_{EO}: essential oil mass, V_{EO}: essential oil volume, η_{EO}: essential oil yield, d_{EO}: density.

Table 4. Extraction results of Ponkan (PW) waste by hydrodistillation for 28 mesh and 32 mesh

Sample	m_S (g)	m_{EO} (g)	V_{EO} (mL)	η_{EO} (BU) (%)
20 to 28 *mesh*	50.08 ± 0.07	0.1100 ± 0.0001	0.20 ± 0.01	0.3990 ± 0.0005
28 to 32 *mesh*	50.14 ± 0.20	0.1100 ± 0.0001	0.20 ± 0.01	0.3990 ± 0.0020

m_S: solid mass, m_{EO}: essential oil mass, V_{EO}: essential oil volume, η_{EO}: essential oil yield.

Table 5. Chemical composition of the essential oil obtained

Compound	Essential oil composition (%) of Ponkan (PW)	Essential oil composition (%) of peel (PP)
γ-Terpineol	0.20	-
β-Pinene	-	0.42
β-Myrcene	-	1.91
Carene	-	0.24
Decanal	0.57	0.21
d-Limonene	88.50	89.72
γ -Terpinene	8.98	7.01
Hexanal	0.08	-
Linalool	1.67	0.34
o-Cymene	-	0.15
d-Limonene Selectivity	7.70	8.73

essential oil in the different raw materials used. Table 5 shows the main components identified.

It can be observed that the Ponkan peel essential oil obtained from PP contains in its chemical composition more terpene compounds and a higher amount of *d*-limonene (approx. 90%). This result was expected since the essential oil glands are in the fruit peel. However, the amount of *d*-limonene present in PW was high (88.5%) and shows a good quality of the essential oils obtained. It is worth noting that the amount of linanool was higher in PW, probably due to the high content of water in the original waste (77.20 ± 0.60%) favored the formation of this alcohol, and probably its decomposition into γ-terpineol, according to Filly *et al.* [37] in the presence of water occurs the formation of linanlool and terpi-4-ol. The juice processing may also have favored the transformation of *β*-myrcene and *β*-pinene into other terpenes such as *γ*-terpinene. However, these hypotheses need to be confirmed in future studies.

The Ponkan essential oil also showed the presence of decanal, less than 1%, i.e., 0.57% and 0.21% in PW and PP, respectively. Value in agreement with that obtained by Simas *et al.* [5] who obtained 0.28%. According to Baudoux [7] esters and aldehydes are responsible for the sedative and calming properties of essential oils, thus the presence of decanal in the chemical composition of the obtained Ponkan essential oil is an indication that it has these therapeutic properties that may be beneficial for aromatherapy purposes.

The essential oil from *Citrus* peel is recognized as safe by the FDA – Food and Drug Administration [38], but the ISO 3528:2012 is broader and also includes pulp from the fresh fruit of *Citrus reticulata* Blanco [39]. Thus, the essential oil obtained from the waste can be used in the food industry or the industries of perfumery, personal care products, and cleaning materials. It was also observed that the composition of the waste (peels, pulp and seeds) practically did not influence the composition of the Ponkan essential oil, especially for *d*-limonene, with similar fractions for PW and PP, 88% and 90%, respectively, and the selectivity in the same order of magnitude, that show the potential of the essential oil obtained from agroindustrial waste.

Another alternative would be to use the essential oil from Ponkan waste to perform different chemical syntheses that use *d*-limonene as a chemical route. As reported by Becerra, González and Villa [23], Bicas *et al.* [22] and Rottava *et al.* [21], it is possible to produce *α*-terpineol, menthol, carvone, limonene-1,2-diol and perillyl alcohol from *d*-limonene. For this purpose, other extraction methods and more selective solvents can be used to improve the *d*-limonene yield in

Ponkan essential oil as suggested by Batista [34]. If the separation of *d*-limonene from the other terpenoids present in the Ponkan essential oil is necessary, it can be done by deterpenation [40–42].

The profitability of the extraction of essential oil from Ponkan waste (PW) was verified. For the calculation was considered the production of 2022[2] - 81,000 tons of Ponkan per year - the fraction of juice (47%), the waste (PW) moisture (77%) and the essential oils yield (0.22%). For these conditions, the annual production of essential oils from Ponkan waste would be approximately 21.7 tons/year, which has a market value of approximately US$ 900,00/kg [43,44]. This process could add value to agroindustrial waste before destined for composting, animal feed, or landfill [45].

The best option according to the perspective from a circular economy of ponkan waste is to extract products with high-value compounds leading to more profitable valorization [45]. Therefore, the previous separation of the peels, before obtaining the juice, makes it possible to obtain 163.0 tons/year of Ponkan essential oil[1]. This increases by more than 7 times the production of essential oils and reduces the energy expenditure for the hydrodistillation process. But to guarantee the sustainability of the process, an economic analysis is necessary.

4. Conclusions

The ponkan essential oil obtained from peels and the juice industry waste presented similar mass fraction of *d*-limonene and both were approximately 90%. Thus, the Ponkan essential oil obtained from agro-industrial waste can be used in different types of industries, such as food or personal care and cleaning products, allowing the use of a natural additive for various products of interest to society. The essential oil from waste Ponkan had a low yield from hydrodistillation compared to that obtained from the extraction of peels. Thus, this study also showed that the composition of the waste is a determining factor in obtaining essential oil from Ponkan fruits and that the separation of the peels, before processing the fruits to obtain juice, can be an economically advantageous step.

Footnotes

[1]Production of Ponkans in the Vale da Ribeira – Brazil.
[2]For the same mass fed to the extractor

Authors' contributions

Conceptualization, M.B.K. and M.L.C.; Methodology, M.G.F.B. and J.M.F.; Investigation, M.G.F.B.; Resources, M.L. C.; Data Curation, M.G.F.B.; Writing–original draft preparation, M.G.F.B; Writing–review & editing, M.B.K., F.A.P.V. and M. L.C.; Supervision, F. A. P. V. and M.B.K.; Funding acquisition, M.L.C.

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Conflicts of interest

The authors declare no conflict of interest.

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