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Yield of essential oil from varieties of *Citrus sinensis* (L.) Osbeck

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Abstract: Residues are generated after the processing of citrus fruits and can be used for essential oil extraction. Thus, the objective of this work was to evaluate the composition, yield, and quality of the proven oil of 30 orange varieties, as an alternative for the better use of its residues. The peels were crushed and subjected to hydrodistillation in a Clevenger-type. The samples were submitted to an Entirely Randomized Design (DIC) and the results were analyzed in Analysis of Variance, Principal Component Analysis, and Hierarchical Cluster Analysis. The greatest gains in essential oil were yielded by the varieties Valencia IAC (5.57%), Pera DP 06 (4.75%), Hamlin 20 (4.58%), and Pera DP 25 (4.28%). Valencia IAC has the potential to extract up to 7.24 kg of essential oil per ton of orange. Limonene represents 93.18 to 99.61% of the oil components, being the major compound among all varieties. Some varieties did not describe secondary compounds or more than one. These components are highly antioxidant and of great importance in the production of cosmetics, personal care products, homeopathy, help gastrointestinal diseases, among other uses. It is possible to conclude that the varieties have different compounds and oils, even when submitted to the same drying and oil extraction process, being the intrinsic characteristics of each variety.

Index Terms: Chemical composition, oranges, limonene, processing.

Rendimento do óleo essencial de variedades de *Citrus sinensis* (L.) Osbeck

Resumo: Após o processamento dos citros, são gerados resíduos que podem ser utilizados na extração do óleo essencial. Assim, o objetivo deste trabalho foi avaliar a composição, o rendimento e a qualidade do óleo comprovado de 30 variedades de laranja, como alternativa para o melhor aproveitamento dos resíduos. As cascas foram trituradas e submetidas à hidrodestilação em máquina tipo Clevenger. As amostras foram submetidas a um Delineamento Inteiramente Casualizado (DIC), e os resultados foram analisados em Análise de Variância, Análise de Componentes Principais e Análise de Agrupamento Hierárquico. As variedades Valência IAC (5,57%), Pera DP 06

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(4,75%), Hamlin 20 (4,58%) e Pera DP 25 (4,28%) apresentaram os maiores ganhos em óleo essencial. Valência IAC tem potencial para extrair até 7,24 kg de óleo essencial por tonelada de laranja. O limoneno representa entre 93,18 e 99,61% do óleo, sendo o composto majoritário entre as variedades. Algumas variedades não descrevem compostos secundários, ou mais de um composto secundário. Esses componentes são altamente antioxidantes e de grande importância na produção de cosméticos, produtos de higiene pessoal, homeopatia, uso em doenças gastrointestinais, entre outros. É possível concluir que as variedades possuem diferentes compostos e óleos, mesmo quando submetidas ao mesmo processo de secagem e de extração do óleo, sendo as características intrínsecas de cada variedade.

Termos para indexação: Composição química, laranjas, limoneno, processamento.

Introduction

Orange (*Citrus* ssp.) is one of the relevant fruit trees in the horticulture industry providing financial gain to the farmers and nutritional benefits to consumers (OTIENO, 2020). Global orange production for 2021/2022 is estimated at 48.8 million tons, 1.4 million tons more than in 2020. Most of the production goes to processing (Brazil, Mexico, and Turkey) (USDA, 2022).

Brazilian industries that produce natural and concentrated juices, sweets, pulps, or extracts use the orange pulp and discard –in most cases– seeds and peel, which are rich in essential oils that could also be used as products, minimizing waste. Brazil stands out by being one of the first essential oil producers due to the high extraction of vegetable oils from citrus. The orange peel is one of the primary plant materials used to extract essential oil from the fruit's pericarp; is a by-product of the juice industry, but with low yield (KOBORI; JORGE, 2005; BIZZO et al., 2009).

Oil extraction can be carried out by steam distillation or cold press, which are the most used methods in the industry. The extraction process by steam distillation uses a still (a container usually made of stainless steel), a condenser, and a bottle to collect the oil. The polar particles are separated from the non-polar ones, so the oil moves away from the water and is removed. The cold press extraction uses a hydraulic press, where the fruit is squeezed in its entirety, and the juice and oil will be available, which will then be separated by distillation because the last can influence the juice's flavor (SILVEIRA et al., 2012).

Essential oils are secondary metabolites produced by plants. Its production, quantity, and quality can be influenced by environmental factors that cause biochemical and physiological changes, such as altering the product's flavor. Essential oils can have several components, with limonene being the main component of oils from citrus fruits. These oils are unstable, especially when exposed to the open air, the presence of metals, humidity, light, or heat (FERRONATTO; ROSSI, 2018).

The orange essential oil is extracted from the pericarp of the fruit and is a by-product of the juice industry. In addition, this essential oil is a product of great importance for the pharmaceutical industry, as it may be used in the production of perfume fixatives, soaps, flavorings, cleaning products, in the food industry, among others (BIZZO et al., 2009).

Due to the great use of essential oils from the residue of orange peels and the increase of *Citrus sinensis* varieties in Brazilian orchards, there is a need to evaluate the content and yield of essential oil of new orange varieties. We hope that this work will contribute to finding varieties that effectively yield more quantities of essential oil and have a variety of substances beneficial to the body, such as phenolic compounds and terpenes that are antioxidants and beneficial to health. Thus, the objective of this work was to evaluate the composition, yield, and quality of the oil of thirty orange varieties, as a suggestion for better use of waste generated in the juice processing industry.

Materials and methods

We evaluated of the content and composition of the essential oil from the fruit peel of 30 varieties of orange (*C. sinensis*) harvested in 2018. The varieties are Bahia 101, Bahia Baianinha, Biondo, Cara-cara, Hamlin 02, Hamlin 20, Jafa, João Nunes, Kona, Lima Score, Midsweet, Natal 112, Natal IAC, Pearson Brown, Pera 2000, Pera DP 06, Pera DP 12, Pera DP 21, Pera DP 25, Pera IAC, Pinneaple, Rubi, Salustiana, Shamouti, Sunstar, Toregosa, Valência IAC, Valência Monte Morelos, Valência Tuxpan, and Westin.

The orchard was planted in October 2009, with ten plants for each cultivar. All plants were grafted with Rangpur lime (*Citrus limonia*, Osbeck). The varieties were from the citrus collection of the Agência Goiana de Assistência Técnica, Extensão Rural e Pesquisa Agropecuária (Agency for Technical Assistance, Rural Extension, and Agricultural Research of Goías State), located in the municipality of Anápolis, Goiás. The original sample consisted of 15 fruits of each variety.

The fruits were peeled by a peeling machine suitable for oranges, thus obtaining only the epicarp (yellow peel) homogeneously. The peel was separated and placed on absorbent paper. The peels were dried at room temperature (25ºC) and in the shade for 48 to 60 hours, then frozen and stored at -4ºC.

We used only dried peels for the analysis. The peels were crushed and subjected to hydrodistillation in a Clevenger-type apparatus for two hours (RAO et al., 2011). Clevenger was wrapped with aluminum foil to prevent the oil from losing its chemical properties. The essential oil obtained was desiccated with Na_2SO_4, placed in an impurity-free container, hermetically closed, and stored at -4ºC for composition analysis (RAO et al., 2011; ROMANO et al., 2020).

The essential oil obtained was submitted to chromatographic analysis, in triplicate for each sample, in gas phase coupled to mass spectrometry (GC/MS), in a Shimadzu GC-MSQP5050A device, with SBD-5 silica capillary column (30 m × 0.25 mm ID, 0.25 m film thickness) (composed of 5% phenyl-methylpolysiloxane), and temperature-programmed as follows: 60-240°C at 3°C / minutes; after, 280°C at 10°C / minutes, ending with 10 minutes at 280°C. The carrier gas was set to a 1 mL/minute flow rate and split mode at 1:20 ratio. The injection port was set at 225°C (ROMANO et al., 2020).

The operational parameters of the significant quadrupole mass spectrometer were set at an interface temperature of 240°C, electron impact ionization at 70 eV with a mass range of 40-350 m/z, and the sampling rate of 1 scan/s. Chemical components of the essential oil were identified by comparing the mass spectra and retention indices with those reported in the literature. Retention indices were calculated by co-injection of a mixture of hydrocarbons, C8 – C32, and using the Van Den Dool and Kratz equation (VAN DEN DOOL; KRATZ, 1963; ADAMS, 2007).

The samples were submitted to Completely Randomized Design (DIC), and the chromatographic results to Analysis of Variance (ANOVA), with means compared by the Scott-Knott test using SISVAR 5.7 software (FERREIRA, 2014). The data were submitted to multivariate analysis of Principal Component Analysis (PCA) and Hierarchical Cluster Analysis (HCA), with Euclidean distance dissimilarity coefficient, using Unweighted Paired Group Method with Arithmetic Averages (UPGMA) in PAST software, version 4.01. Multivariate analyses complement the conclusion of the results, providing a statistically privileged view of the data set (MOITA NETO, 2004; DAWYNDT et al., 2006).

Results and discussion

The varieties presented oil yield values ranging from 0.23 to 5.57% (Table 1); all of them were exposed to the same extraction method. Farahmandfar et al. (2020), studying Navel Thomson orange, found oil yields ranging from 1.20 to 6.90%, depending on the method used to extract the oil, in which the extraction of the fresh fruit represented a lower yield and the dry peel higher.

Valência IAC variety obtained 44.66% more oil yield than Pera IAC. These two are the best-known varieties. All extractions were carried out with the peel of the dried fruits and the orange has 13% of dry mass. The conversion of fresh fruits to dry mass is, for each ton of fresh orange fruit, 130 kg is dry mass (SIQUEIRA; SALOMÃO, 2017).

Table 1 - Oil mass values proportion to the average fruit mass and essential oil yield (%).

VARIETY	OIL (g)	Mass (g)	Yield (%)
BAHIA 101	6.24	288.44	2.16
BIONDO	3.74	189.86	1.97
CARA-CARA	6.00	209.09	2.87
HAMLIN 02	2.53	126.77	2.00
HAMLIN 20	7.67	167.38	4.58
JAFA	5.48	227.13	2.41
JOÃO NUNES	7.56	204.15	3.71
KONA	4.17	186.8	2.23
MIDSWEET	4.25	208.22	2.04
NATAL 112	6.39	173.58	3.68
NATAL IAC	6.74	178.19	3.78
PEARSON BROWN	1.61	194.97	0.83
PERA 2000	3.91	225.56	1.73
PERA DP 12	3.43	143.61	2.39
PERA DP 21	6.23	162.57	3.83
PERA DP 25	8.18	190.99	4.28
PERA IAC	6.90	179.31	3.85
PINEAPPLE	6.05	172.74	3.50
RUBI	0.83	189.31	0.44
SALUSTIANA	5.21	184.3	2.83
SHAMOUTI	5.09	200.49	2.54
SUNSTAR	3.92	234.07	1.67
TOREGOSA	3.50	187.16	1.87
VALÊNCIA IAC	10.57	190.00	5.57
VAL MONTE MORELOS	7.06	210.95	3.35
VALÊNCIA TUXPAN	3.89	175.16	2.22
WESTIN	3.51	169.68	2.07
BAHIA BAIANINHA	0.46	201.03	0.23
LIMA SCORE	2.31	166.53	1.39
PERA DP 06	9.84	206.89	4.75

A maximum of 0.4% of essential oil is extracted from citrus fruit of commercial orchards that follow the production of juices in industrialization yields. Thus, at the end of processing, a ton of fruit generates 4.0 kg of oil by pressing the whole fruit. Hydrodistillation produces a transparent, clear oil, characteristic citrus aroma, low water solubility, photosensitive, and higher yield. In the current work, hydrodistillation allowed all the varieties analyzed in this study to exceed the yield described by Bizzo et al. (2009), of 0.4% for citrus, except for the Bahia Baianinha variety (0.23%) (BIZZO et al., 2009; MEDEIROS, 2014).

Valência IAC was the most representative variety in the study, with a 5.57% oil yield on dry mass (Table 1). Therefore, a ton of fresh fruits has 130 kg of dry mass, 5.57% containing essential oil. Thus, Valência IAC has the potential to extract up to 7.24 kg of essential oil per ton of orange, about 80.50% more than that found by Bizzo et al. (2009), given the high conversion of the fruit in essential oil contents.

Orange peel essential oil is a bioactive component composed of limonene and myrcene as the main components. The chemical composition of citrus, in general, is divided between hydrocarbon terpenes and terpenoids, mainly linalool, nerol, neral, linalin acetate. Limonene is the most common terpene hydrocarbon. Among the varieties, four substances were found in different concentrations: limonene, myrcene, alpha-pinene, and sabinene. These compounds are responsible for the known benefits of essential oils: antimicrobial, and therapeutic. It has been gaining ground in the food, cosmetic and pharmaceutical industries. In the case of citrus, another positive point is its use for food, reducing residues (ARAÚJO et al., 2016; FERRONATO; ROSSI, 2018).

Among the main compounds of the essential oil, limonene represented between 93.18 and 99.61% of the oil (Table 2). Pera DP 06 variety showed the highest percentage of limonene, and almost all the extracted oil was composed of limonene. The varieties with the highest percentages of limonene were: Pera DP 06 (99.61% limonene and 4.75% oil yield), Kona (99.54% limonene and 2.23% oil yield), Valência IAC (99.50% limonene and 5.57% oil yield) and João Nunes (99.50% limonene and 3.71% oil yield).

Limonene, or D-limonene, is the main found compound in other studies of compositions of essential oils extracted from citrus peel. Ferronato and Rossi (2018) found an average of 91.40% limonene, Medeiros (2014) up to 98.91%, and Velázquez-Nuñes et al. (2013) found 96.90% of limonene in orange essential oil samples.

The variety Bahia 101 had the highest percentage of myrcene (1.18%), followed by Ruby (0.86%) and Biondo (0.85%) varieties. Some varieties showed no secondary compounds or more than one secondary compound. Cara-cara presented only limonene, and Salustiana had limonene (96.72%) and myrcene (0.49%). Pera DP 06 and Bahia baianinha presented limonene (99.61% and 95.66%, respectively) and α-pinene (0.39% and 0.33%, respectively).

Only five varieties had sabinene in their composition, and the highest concentration found was 0.37% in the Jafa variety. Nineteen varieties showed compounds not identified by chromatography, with low representation

Table 2 - Percentage (%) of substances detected and identified in the chromatographic analysis of the essential oil composition of the 30 orange peel varieties.

VARIETY	limonene	myrcene	α-pinene	sabinene	CNI*
IR-CALCULATED	1015-1020	973-976	917-921	957-958	-
BAHIA 101	98.29 a	1.18 a	0.44 a	0.09 b	0.00 b
BIONDO	96.13 b	0.85 a	0.34 a	0.00 b	2.68 a
CARA-CARA	98.38 a	0.00 b	0.00 c	0.00 b	1.62 b
HAMLIN 02	94.88 b	0.74 a	0.24 a	0.05 b	4.08 a
HAMLIN 20	97.08 a	0.76 a	0.26 a	0.00 b	1.90 b
JAFA	99.06 a	0.24 b	0.33 a	0.37 a	0.00 b
JOÃO NUNES	99.50 a	0.20 b	0.31 a	0.00 b	0.00 b
KONA	99.54 a	0.18 b	0.28 a	0.00 b	0.00 b
MIDSWEET	97.40 a	0.47 b	0.14 b	0.00 b	1.98 b
NATAL 112	98.04 a	0.37 b	0.14 b	0.00 b	1.45 b
NATAL IAC	98.40 a	0.34 b	0.13 a	0.00 b	1.13 b
PEARSON BROWN	99.35 a	0.34 b	0.31 a	0.00 b	0.00 b
PERA 2000	98.84 a	0.76 a	0.40 a	0.00 b	0.00 b
PERA DP 12	98.42 a	0.00 b	0.08 c	0.00 b	1.50 b
PERA DP 21	97.43 a	0.66 a	0.30 a	0.00 b	1.62 b
PERA DP 25	96.58 b	0.79 a	0.33 a	0.00 b	2.30 a
PERA IAC	99.41 a	0.34 b	0.25 a	0.00 b	0.00 b
PINEAPPLE	95.61 b	0.74 a	0.32 a	0.05 b	3.27 a
RUBI	95.61 b	0.86 a	0.35a	0.00 b	3.18 a
SALUSTIANA	96.72 b	0.49 b	0.00 c	0.00 b	2.79 a
SHAMOUTI	95.65 b	0.35 b	0.25 a	0.00 b	3.74 a
SUNSTAR	95.31 b	0.68 a	0.24 a	0.00 b	3.77 a
TOREGOSA	93.18 b	0.84 a	0.31 a	0.00 b	5.68 a
VALÊNCIA IAC	99.50 a	0.19 b	0.13 b	0.00 b	0.00 b
V MONT MORELOS	97.52 a	0.38 b	0.18 b	0.00 b	1.92 b
VALÊNCIA TUXPAN	94.02 b	0.18 b	0.07 c	0.00 b	5.73 a
WESTIN	99.10 a	0.57 a	0.33 a	0.00 b	0.00 b
BAHIA BAIANINHA	95.66 b	0.00 b	0.33a	0.00 b	4.00 a
LIMA SCORE	99.43 a	0.22 b	0.26 a	0.09 b	0.00 b
PERA DP 06	99.61 a	0.00 b	0.39 a	0.00 b	0.00 b
Fisher' f	2.94	5.92	7.07	7.15	3.12
Standard Error	1.07	0.13	0.04	0.03	1.00
CV (%)	1.90	48.72	29.05	209.04	95.76

*CNI = Unidentified Compounds.

in the total oil volume. The Valencia Tuxpan variety had the highest percentage of unidentified substances representing 5.73% of its essential oil.

Secondary compounds, such as octanal, β-Phellandrene, 1-octanol, linalool, decanal, citral, and butylated hydroxytoluene, among others, can be found in lower concentrations but are not irrelevant. Together with limonene, they act strongly in microbial, antifungal, and antioxidant activities. Up to 70 components can be found in essential oils from oranges when extracting oils from dry peels, by pressing the whole fruit, or even other methods of extracting fresh peels, which are more efficient, and minimize the losses of highly volatile components that manage to remain in the oil after separation (ESPINA et al., 2011; FERRONATO; ROSSI, 2018).

Limonene, myrcene, alpha-pinene, and sabinene found in orange essential oil samples can be used in cosmetics, pharmaceuticals, natural products for pest and disease control, as bioactive ingredients, with antifungals activities, and antibiotics. These compounds are not lost when the skin is dried prior to oil extraction. However, other compounds such as nerol, geraniol, isoneral, terpineol, 1-nonanol, undecanal, and other volatile compounds can dissipate, depending on the drying method (FARAHMANDFAR et al., 2020).

Orange essential oils have been used in homeopathy and aromatherapy for years, with increasing consumption of adepts of these practices, cause as inhalation through aromatherapy does not cause toxicity to the human body. The most common forms of use are ingestion in teas and drinks, applying a drop under the tongue, inhalation, or topical use. Among the main health benefits are: anticarcinogenic and antitumor activities, inhibiting cell death; feeling of relaxation; anxiolytic, antidepressant effect; anti-inflammatories helping to relieve muscle pain and antibiotic effect, and can be used in the treatment of gastrointestinal diseases (LEHRNER et al., 2000; BERGONZELLI et al., 2003; GOUGEON et al., 2005; YIP; TAM, 2008; MORAES et al., 2009; FATURI, et al., 2010; CHIDAMBARA et al., 2010; VIANA et al., 2016; MAGALHÃES, 2019; KOYAMA; HEINBOCKEL, 2020)

In general, orange and citrus essential oils are considered great antioxidants and can be widely used in cosmetics, acting as bioactives, helping to detoxify the body, helping with weight loss, and treating acne. It also inhibits oxidative reactions, being a food preservative, and due to its antifungal and antimicrobial capacity, it can be used in the treatment of fruit peel, prolonging shelf life (HAAZ et al., 2006; CHERNIACK, 2008; MATIZ et al., 2012; KAMAL et al., 2013; VOLPATO et al., 2015; JORGE et al., 2016; TORRES-ALVAREZ et al., 2017).

Essential oils affect the biochemical processes of insects and mites, mainly by smoking or topical action, and can be neurotoxic or growth regulators, acting with acaricidal, insecticidal, larvicidal, and repellent effects. They have antifungal and antibiotic action against various pathogens (KIDANE, 2011; ASSUNÇÃO, 2013; CAMPOLO et al., 2014; CAMARA et al., 2015; MAIA et al., 2015, OBOH et al., 2017, FARIAS et al., 2018).

The multivariate analysis for the substances found in the essential oil of the varieties showed that the first and second PCA components explain 99.75% of the data variance. The first component explained alone, 97.32%, and the second 2.43%. PCA was able to reproduce the results found on the graph. Toregosa and Valência Tuxpan varieties are positioned on the far left, with the lowest concentrations of limonene, thus, being placed in the graph opposite to limonene. On the far right, the varieties with the highest limonene contents, upwards, are Pera DP 06, Kona, Valência IAC, João Nunes, Lima Score, Pera IAC, Pearson Brown, Jafa, Westin, and Pera 2000. Bahia 101 variety, with a high content of limonene and myrcene, is positioned in the second quadrant above the other varieties (Figure 1).

The cluster analysis concerning the substances found (Figure 2) showed a cophenetic correlation of 0.77. It made it possible to group the varieties into four groups, where the

Bahia 101 variety differs from the others as it has the highest concentration of myrcene. In addition, it has 0.44% α-pinene and 0.09% sabinene in its composition. There are three other large groups, the first group formed by Toregosa and Valência Tuxpan, the varieties with the lowest limonene contents and the highest volumes of unidentified compounds in the oil. The second group, composed of Biondo and Rubi varieties, with a

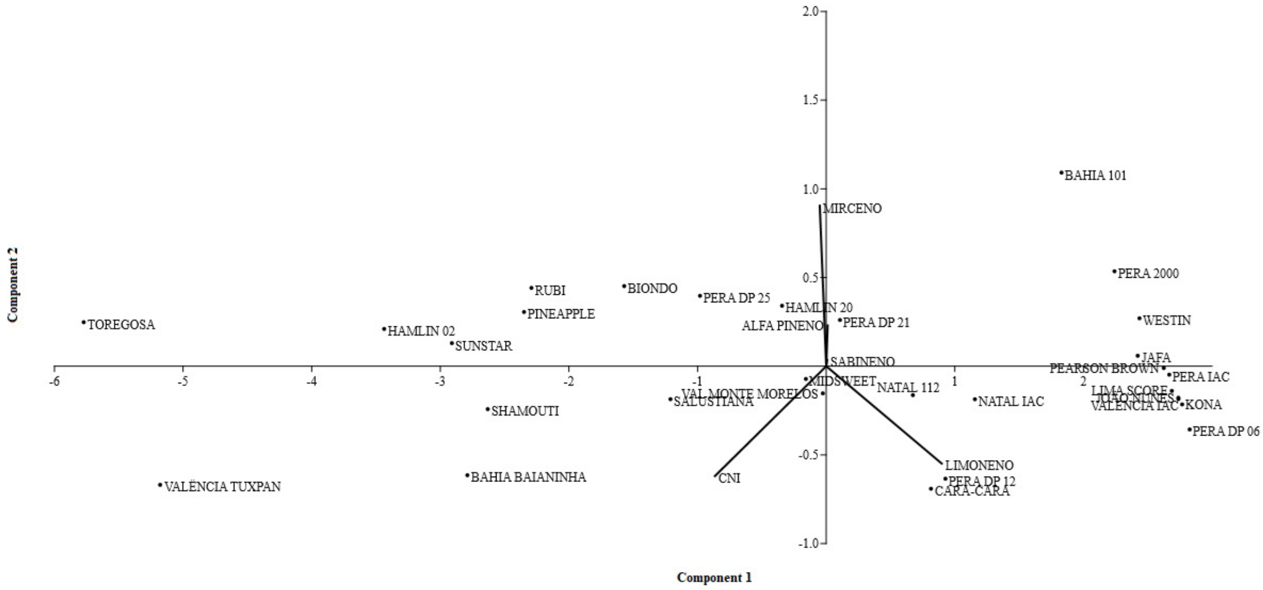

Figure 1 - Biplot of substances found in the essential oil of the orange peel varieties (limonene, myrcene, α-pinene, sabinene, and CNI (Unidentified Compounds).

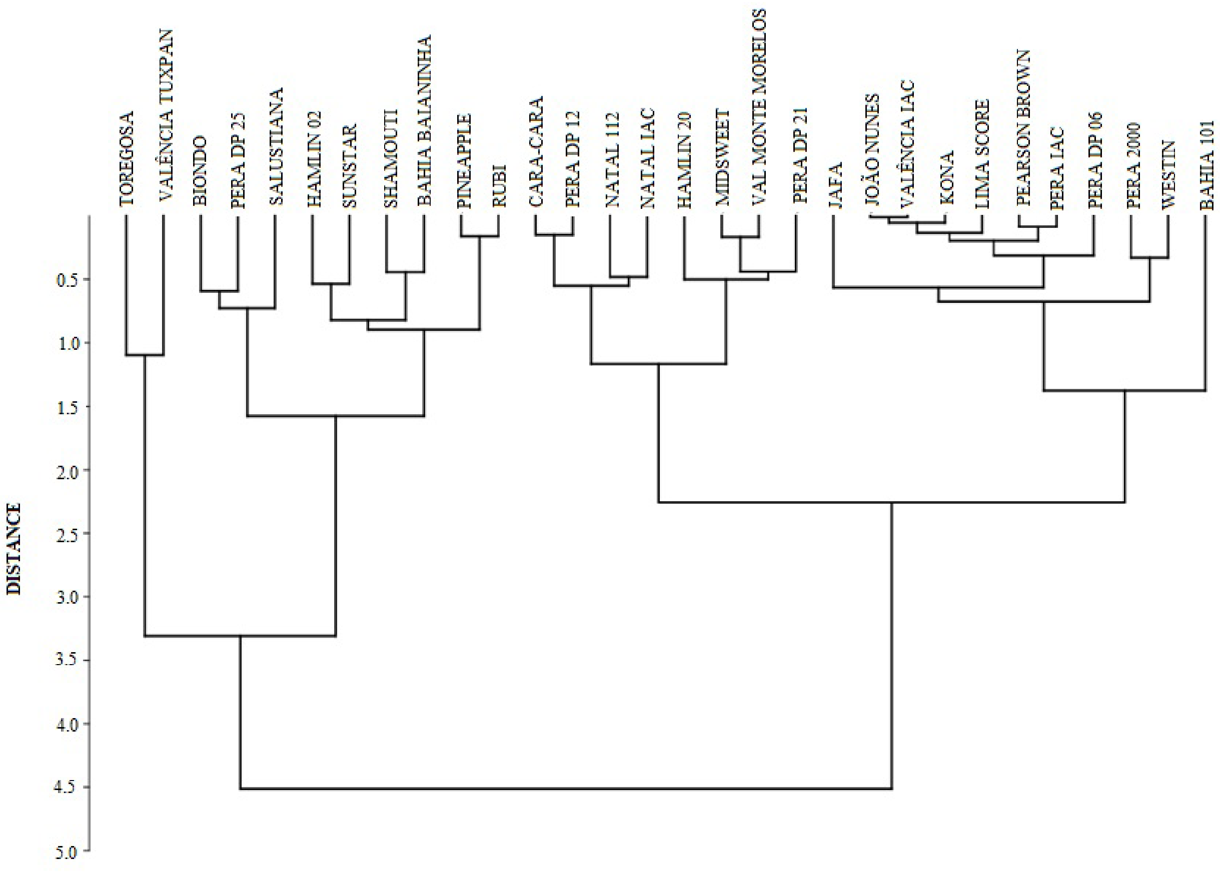

Figure 2 - Dendrogram of Hierarchical Cluster Analysis, by Euclidean Distance, (UPGMA method) among orange varieties concerning raw materials in essential oil.

concentration of unidentified components between 2.68 and 4.00%, the third group, with Cara-cara, Pera DP 21 and others, has fewer unidentified components, between 1.13 at 1.98%. The last group was formed by the 11 varieties that did not present any unidentified compounds in their oil: Jaffa, João Nunes, Valência IAC, Kona, Lima Score, Pearson Brown, Pera IAC, Pera DP 06, Pera 2000, Westin, and Bahia 101.

Studies like these are necessary, mainly due to the multiple applications of essential oils. Thus, it is crucial to understand the composition of substances found in essential oils and the varieties that have greater amounts of beneficial substances, and those that have greater oil yield in their fruit peels. Complementary studies are needed to find the most efficient extraction methods, which lead to lower losses and higher oil yields.

Conclusion

Except for Bahia Baianinha (0.23%), all varieties have an essential oil yield above the yield found in commercially grown oranges, which is 0.4%.

The varieties Valencia IAC (5.57%), Pera DP 06 (4.75%), Hamlin 20 (4.58%), and Pera DP 25 (4.28%) have amounts of essential oil considered satisfactory and can be exploited, being alternative varieties to the ones existing on the market.

Limonene, myrcene, *α-pinene,* and sabinene are compounds that remain in the fruit peel even after the drying process and oil extraction by the hydrodistillation method. They are highly antioxidant compounds, with limonene being the primary compound in the essential oil, regardless of the orange variety.

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