

Phytochemical characterization of lime (*Citrus aurantifolia* Christm Swingle) and orange (*Citrus × sinensis* (L.) Osbeck) rind at Guerrero, Mexico

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Abstract: Phytochemicals, that include both primary and secondary metabolites (SMs) of the plants, are of great interest in a variety of sectors, such as agriculture, pharmaceuticals and cosmetics. Recently, it has been recognized that SMs could be used as a basis to develop biopesticides. Lime and orange peels were collected and dried at room temperature from Guerrero, México. They were ground until obtaining a fine powder in a blender and then the extraction of the compounds was conducted. Characterization of plant extracts was done through gas chromatography-mass spectrometry (GC/MS). The outstanding compounds in the extracts were *d*-limonene and citric acid. *d*-Limonene stood out as one of the predominant ones with a concentration of 95.66 %. The ethanolic extracts presented a higher number of compounds than aqueous extracts. This study represents a significant advance in the characterization and comprehension of orange and lime peel extracts, providing a solid base for future studies and practical applications in various scientific and commercial fields.

Keywords: Extracts, Phytochemical, Metabolites, *d*-limonene.

Introduction

Conventional agriculture (CA) is essential for food production globally since it provides most of the foods that people consume daily. This type of agriculture is characterized by the intensive use of chemical products, such as pesticides and fertilizers, as well as by the use of high-yield varieties, monocrops, genetically modified organisms, heavy machinery, intensive mechanization, and irrigation systems (Willer & Lernoud, 2017; Le et al., 2020). Although this model is efficient in terms of production, it presents important sustainability and environmental impact problems, among which some that stand out are air, soil and water pollution, due to the use of chemical pesticides that can affect local biodiversity, generate human health problems, contribute to climate change, and resistance problems. For example, for the case of weeds, in the year 2022, 350 cases of weed resistance to glyphosate were reported, and in addition multiple resistance was found in 23 species of weeds in 17 countries around the world (Arispe-Vázquez et al., 2023). For the case of insects, in the year 2021 it was reported that *Spodoptera frugiperda* J. E. Smith (Lepidoptera: Noctuidae) was already resistant to 33 active ingredients in different parts of the world (Cerna et al., 2022). There are also monocrops, which in many cases increase the vulnerability of crops in the presence of pests and diseases, with the tendency to generate a continuous dependency on pesticides and fertilizers.

From a perspective of sustainability, CA suggests worries due to its intensive use of non-renewable resources, such as fossil fuels and energy for fertilizer production, while water consumption in irrigation systems can be unsustainable in regions prone to drought. These problems have led to a growing interest in more sustainable alternatives such as organic agriculture, agroecology, and other approaches

that promote agricultural practices that are less dependent on chemical pesticides and more respectful with the environment and human health in the long term.

In the 2023 cycle, the surfaces sown in Mexico for lime (*Citrus × limon* L. Burm. f.) and orange (*Citrus × sinensis* (L.) Osbeck) were 222,643.40 and 353,609.46 ha, with a production of 3,239,914.70 and 4,942,658.65 million t, respectively. The value of lime and orange production was USD 2,704,395.64 in 2023. During that same cycle, the state of Guerrero had a production of Mexican lime and orange of 71,560.04 and 5,987.43 t, respectively (SIAP, 2024). Concerning the state of Guerrero during the same cycle, the specific production was 71,560.04 t of Mexican lime, and these data highlight the significant contribution of the state of Guerrero to the national production of Mexican lime.

The phytochemicals that include both primary and SMs of the plants are of great interest in a variety of sectors, including the agricultural, pharmaceutical and cosmetic (Elshafie et al., 2023; Reshi et al., 2023). Primary metabolites are essential for the growth and development of plants, while secondary metabolites, also known as specialized metabolites, carry out crucial roles in the adaptation of plants to environmental stress and in their defense against predators and pathogens (Kroymann, 2011; Fernie & Pichersky, 2015; Yang et al., 2018; Isah, 2019; Pang, 2021; Fazili et al., 2022; Ochatt et al., 2022; Jeyasri et al., 2023; Salam et al., 2023; Sugiyama, 2023) these SMs are produced in small amounts yet they are extremely important due to their bioactive properties.

The SMs can be classified into several categories, such as: phenols, terpenes, steroids, alkaloids and flavonoids, each with specific properties that can benefit both agriculture and other industries (Guerrieroma et al., 2018; Kessler & Kalske, 2018). The current knowledge about how to obtain plants with high concentrations of these metabolites is vital to advance in agricultural and horticultural techniques that use economic and sustainable biomass (Selwal et al., 2023). In addition to their potential as biopesticides, SMs are also applied in agrochemicals, food additives, and the fragrance industry (Patil, 2020). The production of SMs is influenced by a variety of factors both biotic and abiotic; among the biotic factors there are interactions with other pathogenic organisms, and on the other hand, among abiotic factors there is availability of water, light and temperature. The type of fertilization applied and general agronomic management can also affect the production of SMs, and some studies have proven that certain fertilizers and agronomic practices can alter the concentration and composition of these compounds in plants. As a whole, understanding these factors and their adequate manipulation is essential to improve the production and quality of SMs, opening new opportunities for innovation in various fields. Therefore, the objective of this study was to characterize aqueous and ethanol extracts of lime and orange rind.

Results and Discussion

The results from this study broaden the existing knowledge about the abundant phytochemical composition of aqueous and ethanol extracts of orange and lime. The aqueous extract of orange presented a dark brown color and the presence of mainly 10 chemical compounds, of which *d*-limonene stood out as one of the predominant ones with a concentration of 95.66 %, with a retention time of 4.1 min, followed by citric acid with 1.11 %. In this regard, it is important to highlight that in the aqueous extract of orange, there were other compounds such as: α -terpinene, linalool, palmitic acid, 4-terpineol, oleic acid, β -pinene, 2-methoxy-4-vinylphenol and α -terpinolene at concentrations of \leq 0.74 %, which are classified within organic acids, terpenoid compounds, terpenic alcohols and phenols. Their concentrations and respective retention times and abundance are presented in Table 1. The terpenes correspond to the largest compounds among SMs and have been widely studied due to their potential as antimicrobial, insecticide and weed control agents. The ethanol extract of orange presented a dark yellow color and 14 chemical compounds were observed, of which 6 were equal to those obtained from the aqueous extract of orange, although in higher concentrations, with the exception of *d*-limonene which reduced its concentration to 60.7 % in the ethanol extract, compared to the aqueous extract. In the ethanol extract of orange, 8 new compounds were found among which hexanedioic acid and caryophyllene stand out, which were observed at concentrations of 11.18 % and 1.8 %, respectively. The remaining 6 new compounds observed were present at concentrations of \leq 0.80 % (Table 1).

The aqueous extract of lime presented a reddish-brown color and the presence of 11 chemical compounds was observed, while their concentrations were between 1.75 and 17.77 %. The compounds that presented the highest concentrations were *d*-limonene and palmitic acid with 16.77 and 13.20 %, respectively (Table 1, Figure 1). Meanwhile, the remaining 8 compounds showed a concentration of \leq 9.51 %. The ethanol extract of lime presented a dark orange color, and it was the extract where the highest number of chemical compounds was observed, with 25 from which only 7 were the same as those obtained in the aqueous extract of lime. Its concentration was between 0.15 and 36.94 %. The compounds that presented the highest concentrations were citric acid with 36.94 %, followed by oleic acid with 10.84 % (Table 1). Likewise, it was seen that in the ethanol extract of lime the concentration of oleic acid, citric acid and caryophyllene increased, and the concentration of *d*-limonene, β -bisabolen, 4-terpineol, α -farnesene, and caryophyllene decreased, compared to the aqueous extract. Masakazu et al. (1999) mentioned that *d*-limonene can be present at a concentration close to 95 % in orange rinds, which agrees with what was seen in this study. Several factors, including genotype, climatic conditions and cultural practices can influence the assortment and accumulation of citrus essential oils in citrus rinds (Salvatore et al., 2022). For this reason, it is crucial to continue exploring the phytochemical composition of the rind of different citrus varieties and species. The predominant chemical compounds were organic acids and terpenes, which are very common in natural extracts and essential oils. Similarly, some authors mention factors that affect the percentage of active ingredients, such as geographic distribution, environmental conditions, temperature, rainfall, altitude, and sunlight hours (Hossain et al., 2014). Other authors mention that citrus species have mainly phenolic compounds (flavonoids, phenolic acids and coumarins), terpenoids (limonoids and carotenoids), and pectins (Lado et al., 2018; Cebadera-Miranda et al., 2019; Lu et al., 2021).

Diverse studies have shown that *d*-limonene is a natural antioxidant and anti-inflammatory compound. It has been studied for its potential not just as herbicide and organic insecticide (repellent), but due to its chemical properties against weeds and insects. In this regard, Davidowski & DiMarco (2009) mention that orange oil has a considerable amount of limonene and has numerous applications, including as fuel in motors, a strong degreaser in cleaning applications, and a natural pesticide. Its biodegradable and less toxic properties compared to chemical herbicides makes *d*-limonene an attractive and sustainable alternative (Lin et al., 2024). For example, studies have documented its ability to act as a natural repellent against mosquitos and other flying insects, highlighting its potential in pest control in a more ecological way (Michaelakis et al., 2009; Maia et al., 2011; Pohlit et al., 2011; Giatropoulos et al., 2012).

Table 1. Components and concentration of the extracts under study.

NE	Color	NC	Compound**	Formula	Retention Time (min)	Concentration (%)
1		1	<i>d</i> -Limonene	C ₁₀ H ₁₆	4.147	95.66
		2	Citric acid	C ₆ H ₈ O ₇	3.056	1.11
		3	α-Terpinene	C ₁₀ H ₁₆	6.98	0.74
		4	Linalool	C ₁₀ H ₁₈ O	5.32	0.44
		5	Palmitic acid	C ₁₆ H ₃₂ O ₂	16.36	0.42
		6	4-Terpineol	C ₁₀ H ₁₈ O	6.72	0.41
		7	Oleic acid	C ₁₈ H ₃₄ O ₂	18.02	0.20
		8	β-Pinene	C ₁₀ H ₁₆	2.75	0.19
		9	2-methoxy-4-vinyl phenol	C ₉ H ₁₀ O ₂	8.90	0.13
		10	α-Terpinolene	C ₁₀ H ₁₆	5.15	0.12
2		1	<i>d</i> -Limonene	C ₁₀ H ₁₆	6.59	16.77
		2	Palmitic acid	C ₁₆ H ₃₂ O ₂	23.39	13.20
		3	Camphene	C ₁₀ H ₁₆	12.70	9.51
		4	Oleic acid	C ₁₈ H ₃₄ O ₂	25.05	9.19
		5	β-Bisaboleno	C ₁₅ H ₂₄	18.24	8.28
		6	Citric acid	C ₆ H ₈ O ₇	1.53	5.77
		7	4-Terpineol	C ₁₀ H ₁₈ O	12.31	3.44
		8	α-Farnesene	C ₁₅ H ₂₄	17.24	3.12
		9	Caryophyllene	C ₁₅ H ₂₄	17.06	2.99
		10	Tetradecanoic acid	C ₁₄ H ₂₈ O ₂	23.56	2.38
		11	9-Octadecanoic acid-(z)-methyl E ester	C ₂₀ H ₃₈ O ₂	24.61	1.75
3		1	<i>d</i> -Limonene	C ₁₀ H ₁₆	7.20	57.49
		2	Hexanedioic acid	C ₆ H ₁₀ O ₄	27.23	11.18
		3	Oleic acid	C ₁₈ H ₃₄ O ₂	25.22	6.24
		4	Citric acid	C ₆ H ₈ O ₇	3.97	5.82
		5	Palmitic acid	C ₁₆ H ₃₂ O ₂	23.43	3.21
		6	Caryophyllene	C ₁₅ H ₂₄	18.24	1.80
		7	α-Terpinolene	C ₁₀ H ₁₆	9.95	1.20
		8	β-Pinene	C ₁₀ H ₁₆	5.54	0.82
		9	β- Copaene	C ₁₅ H ₂₄	17.06	0.80
		10	α-Copaene	C ₁₅ H ₂₄	16.84	0.58
		11	9-Octadecanoic acid	C ₁₈ H ₃₄ O ₂	21.25	0.51
		12	δ-Cadinene	C ₁₅ H ₂₄	18.50	0.47
		13	Linoleic acid	C ₁₈ H ₃₂ O ₂	26.36	0.32
		14	β -Farnesene	C ₁₅ H ₂₄	17.54	0.25
4		1	Citric acid	C ₆ H ₈ O ₇	4.50	36.94
		2	<i>d</i> -Limonene	C ₁₀ H ₁₆	5.10	5.16
		3	Gamma-Terpinene	C ₁₀ H ₁₆	5.45	1.35
		4	p-Cymene	C ₁₀ H ₁₄	5.89	0.83
		5	4-Terpineol	C ₁₀ H ₁₈ O	7.15	1.10
		6	α-Terpineol	C ₁₀ H ₁₈ O	7.33	1.45
		7	α-Myrcene	C ₁₀ H ₁₆	9.99	0.41
		8	Caryophyllene	C ₁₅ H ₂₄	10.43	3.69
		9	α-Farnesene	C ₁₅ H ₂₄	10.86	0.84
		10	β-Bisabolene	C ₁₅ H ₂₄	11.34	4.06
		11	Lauric acid	C ₁₂ H ₂₄ O ₂	12.04	1.13
		12	Aromadendrene	C ₁₅ H ₂₄	12.13	0.28
		13	Myristic acid	C ₁₄ H ₂₈ O ₂	14.31	0.71
		14	Methyl-Palmitate	C ₁₇ H ₃₄ O ₂	15.93	0.15
		15	Palmitic acid	C ₁₆ H ₃₂ O ₂	16.54	10.16
		16	Limetin	C ₁₁ H ₁₀ O ₄	16.84	1.54
		17	13-acid octadecanoic, methyl ester	C ₁₉ H ₃₆ O ₂	17.63	0.19
		18	Linoleic acid	C ₁₈ H ₃₂ O ₂	18.20	4.90
		19	Oleic acid	C ₁₈ H ₃₄ O ₂	18.44	10.84
		20	Isopimpinellin	C ₁₃ H ₁₀ O ₅	19.20	0.18
		21	Hexanedioic acid	C ₆ H ₁₀ O ₄	20.25	3.57
		22	6-Octadecenoic acid	C ₁₈ H ₃₄ O ₂	20.73	0.12
		23	Pentacosane	C ₂₅ H ₅₂	20.99	0.09
		24	Eicosane	C ₂₀ H ₄₂	22.47	0.15
		25	1,5,8-p-Mentatriene	C ₁₀ H ₁₄	22.90	0.64

NE: extract number, 1: aqueous orange extract, 2: aqueous lime extract, 3: ethanolic orange extract, 4: ethanolic lime extract, NC: compound number, **: predominant compound in the four extracts.

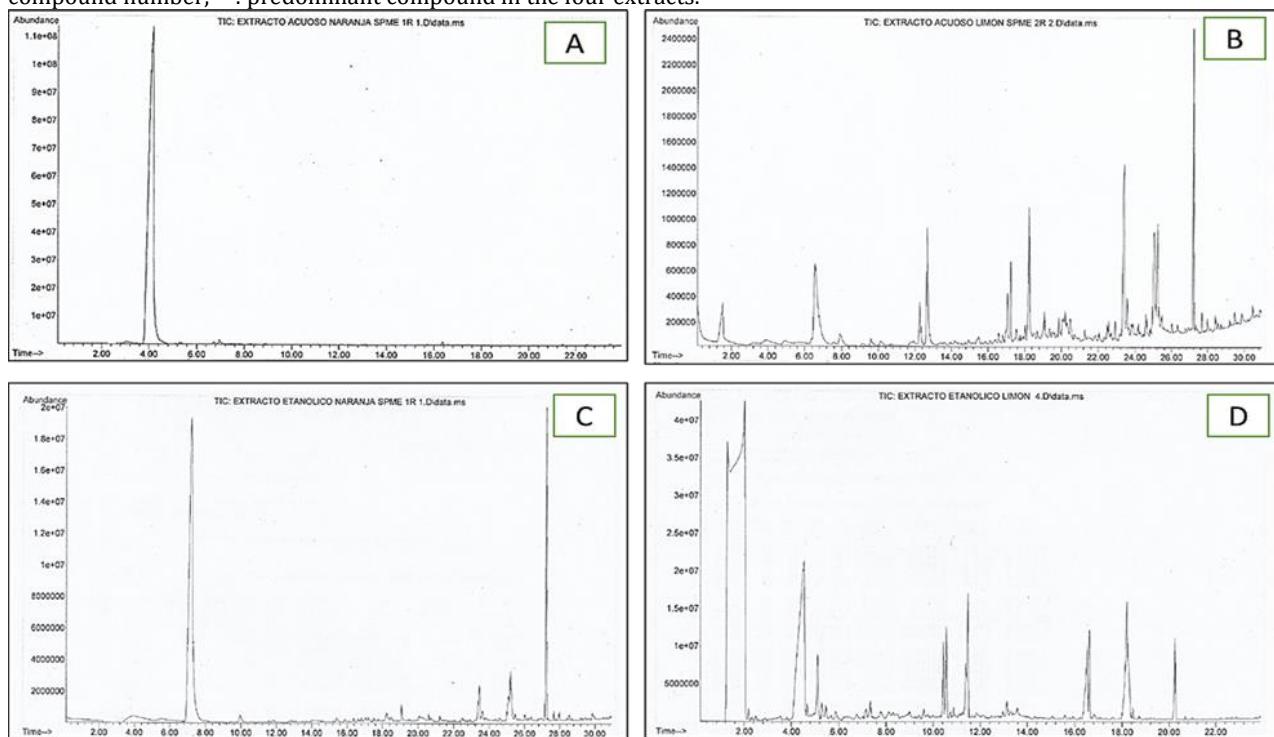


Figure 1. GC Chromatogram of different extracts under study. A: chromatogram of the compounds identified in the aqueous extract of orange, B: chromatogram of the compounds identified in the aqueous extract of lime, C: chromatogram of the compounds identified in the ethanolic extract of lime, D: Chromatogram of the compounds identified in the ethanolic extract of orange. The high peaks represent *d*-limonene.

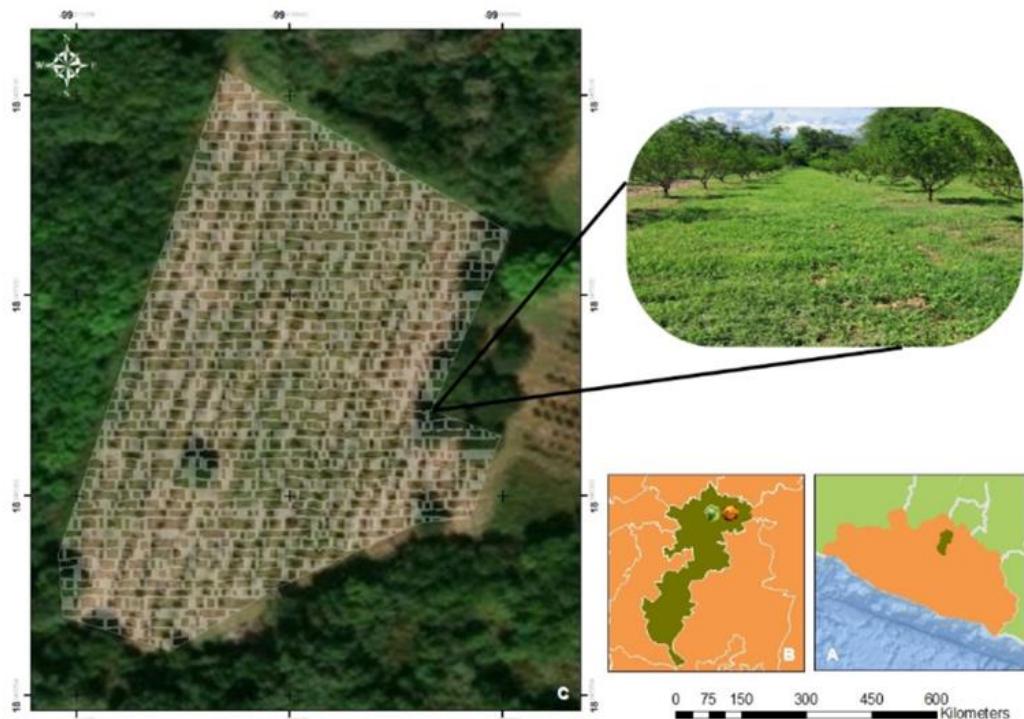


Figure 2. Geographic location of the study area. (A) State at Guerrero (B) Municipality at Iguala de la Independencia, (C) Mexican lime plot.

d-Limonene is recognized for its antioxidant potential, anti-inflammatory properties and anti-cancer properties and many health-promoting effects (Akhavan-Mahdavi et al., 2022). These findings highlight the potential of orange and lime extracts as promising sources of bioactive compounds with applications in different industries such as health and agriculture. For example, the ethanol extract of orange presented great similarity in terms of color and viscosity to a commercial bioherbicide based on mullein, coconut oil, pine resin, puccinia fungus, and papain (as shown in the products label), so it is interesting to continue with the research of these extracts as biopesticides, among them as organic herbicides. Commercial and organic limonene products have been tested, applied in the field for weed management, attaining low but visible control effects on wide and narrow leaves.

Materials and Methods

Study area and collection of plant material

The study was conducted in the Instituto Nacional de Investigaciones Forestales, Agrícolas y Pecuarias (INIFAP)-Iguala Experimental Field (CEIGUA) at Iguala de la Independencia, Guerrero, Mexico. Mexican lime *Citrus aurantifolia* Christm Swingle varietie Colimex was registered in the Servicio Nacional de Inspección y Certificación de Semillas (SNICS) service in Mexico, with registration number LIM-003-230708, was collected from an experimental plot of 2 ha in CEIGUA, which is located on geographic coordinates 18.337614 of latitude and -99.511906 of longitude (Figure 2). The orange rinds were obtained from juice shops in the central zone at Iguala de la Independencia.

Processing of samples

Clean and sterile containers were used for this process. The lime and orange rind were washed with tap water and left untreated. They were ground into a fine powder in a blender and then the extraction of the compounds was conducted using 20 L jugs as containers (only 10 L of solvent and plant material were used). In each jug, 20 g of ground rind was placed and 1 L of tap water or ethanol was added, depending on the type of extraction. For the extraction with water, the jugs were left out for 53 days, while for the extraction with ethanol they were left only for 15 days for the extraction at ambient temperature (25 °C). During the entire time of extraction, regular monitoring was conducted to ensure that the conditions were optimal (in each monitor the gases were released from the fermentation product with the solvent), no signs of fungal or bacterial contamination were observed (Toledo-Aguilar, 2024).

Filtration of the solutions was carried out at the end of the periods specified for each method of extraction. For the case of the extraction of phytochemical compounds through water, high-quality filters were used (WhatmanTM No.1), which separated the rind residues from the liquid solution that contained the bioactive compounds. This step was repeated several times to ensure that no solid particles remained in the final solutions, which could interfere with later analyses. For the extraction through ethanol, a vacuum pump was used (Ulvac® model DTC-22A) with filters (WhatmanTM No.1) to eliminate any residual particle. Then, the ethanol extract was subjected to an evaporation process through a rotary evaporator, which operated at 150 rpm and 90 °C, thus concentrating the compounds extracted. This procedure allowed obtaining a pure and concentrated extract, eliminating the ethanol from the final product. The use of the rotary evaporator (Yamato® model VR300) allowed eliminating the solvent, preserving the integrity of the bioactive compounds extracted and avoiding their degradation from prolonged exposure. The extracts of lime and orange rinds were placed in amber glass containers to protect them from light and were stored in refrigeration at 5 °C.

Qualitative chemical analysis

The characterization of plant extracts was carried out through gas chromatography-mass spectrometry (GC/MS) in an Agilent Technologies 7820A equipment, with mass selective detector (MSD, Agilent Technologies® 5975), operated in the mode of complete radiofrequencies scan (full scan) in splitless mode, with an injection volume of 1 µL of sample. The initial temperature slope was 50 °C, followed by an increase of 10 °C min⁻¹ until 300 °C, where it was kept for 1 min. Then a post-run of 3 min at 300 °C was added. The injection port was kept at 250 °C and the source and quadrupole were at 230 °C and 150 °C, respectively. The column used was HP-5MS 5 % phenyl methyl silox: 30 m x 250 µm x 0.25 µm. High-purity helium was used as carrying gas, with a flux of 1 mL min⁻¹ with electron impact in ionization mode 70 eV. For the analysis of the aqueous extracts, the SPME-ID technique reported by Rivera-Dávila et al. (2022) was used. For the ethanol extracts, they were previously concentrated in a rotary evaporator at 50 °C to a third of their volume. Next, the ethanol extract was passed through micropore filters of 0.22 µm. After filtering, 1 µL of sample was injected for its analysis. The extractions were carried out by triplicate. The compounds were identified based on their retention indices and mass spectrums, using the W9N11.L database and a similarity index higher than 90 %.

Conclusion

The *d*-limonene stood out as one of the predominant ones with a concentration of 95.66 %. The ethanolic extracts could capture the most compounds. This study represents a significant advance in the characterization and comprehension of orange and lime extracts, providing a solid base for future studies and practical applications in various scientific and commercial fields.

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