

Original Article

## Chromatography of *Solanum habrochaites* extracts with the first record of the docosanoate, hexadecanoate and octadecanoate ethyls in this plant and in Solanaceae

Cromatografia de extratos de *Solanum habrochaites* com o primeiro registro de etil docosanoato, hexadecanoato e octadecanoato nesta planta e em Solanaceae

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### Abstract

The increasing need for sustainable alternatives to synthetic insecticides has driven the analysis of extracts from *Solanum habrochaites*, a wild tomato, through fractionated column chromatography. Potential bioactive compounds for pest management, a clean and promising biotechnological solution, have been reported from this plant. The objective is to provide detailed gas chromatography data, including peaks, structural formulas, and retention indices for the extracts of *S. habrochaites* aerial parts. Column chromatographic analysis was conducted with five fractions (F1, F2, F5, F3, and F4) of *S. habrochaites* extracts. Long-chain hydrocarbons such as hexadecanoic acid and docosano were identified in the F1 fraction; fatty acid esters, including hexadecanoate and octadecanoate ethyls in the F2 and methyl ketones, with tridecan-2-one as the major component in the F5, while no identifiable compounds were disclosed in the F3 and F4 fractions. The column chromatography provided valuable insights into compounds in the F1, F2, and F5 fractions of *S. habrochaites* extracts, highlighting fatty acid esters, long-chain hydrocarbons, and methyl ketones. The bioactive compounds, from extracts of this plant, including the first record of the docosanoate, hexadecanoate and octadecanoate ethyls in *S. habrochaites* and Solanaceae, reinforces their promising biological application in different areas of science.

**Keywords:** botanical, bioactive, compound, control, insecticide, pest.

### Resumo

A crescente necessidade por alternativas sustentáveis no manejo de pragas motivou a análise de extratos de plantas do tomate silvestre, *Solanum habrochaites*, por cromatografia em coluna de fração. O objetivo foi detalhar resultados da cromatografia gasosa, incluindo picos, fórmulas estruturais e índices de retenção para compostos de extratos de partes aéreas de *S. habrochaites*. A análise cromatográfica em coluna foi realizada para cinco frações (F1, F2, F5, F3 e F4) dos extratos de *S. habrochaites*. Hidrocarbonetos de cadeia longa, como ácidos hexadecanóico e docosanoato, foram identificados na fração F1; ácidos graxos, incluindo hexadecanoato de etila e octadecanoato de etila na F2 e metilcetonas, com tridecan-2-ona como o composto majoritário, na F5 e nenhum composto nas frações F3 e F4. A análise dos extratos de *S. habrochaites*, por cromatografia em coluna, identificou compostos nas frações F1, F2 e F5, desta planta, destacando ésteres de ácidos graxos, metilcetonas e hidrocarbonetos de cadeia longa e o primeiro relato dos docosanoato, hexadecanoato e octadecanoato nessa planta e em Solanaceae.

**Palavras-chave:** botânico, bioativo, composto, controle, inseticida, praga.

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## 1. Introduction

Chemical compounds from the secondary metabolism of plants are part of their communication, defense, and growth (Uranga et al., 2016). Fraction column chromatography identifies chemical compounds in plant extracts (Prasathkumar et al., 2022), with the potential for cosmetic (Popova et al., 2020), botanical deterrents, insecticides, and repellents (Santos et al., 2023), herbicides (Menezes et al., 2014), and treatment of human diseases (Bahgat et al., 2023).

Problems associated with synthetic pesticides misuse, such as ecological risks, intoxication, and high costs increase the need for alternative strategies in pest management programs (Leach and Mumford, 2008). Plant extracts with insecticidal secondary metabolites are a clean biotechnology approach (Attia et al., 2013). These extracts are biodegradable and pose lower environmental risks and potential insect resistance compared to conventional synthetic insecticides (Petacci et al., 2012).

*Solanum habrochaites* (Solanaceae), a wild tomato, is less preferred by various insect pests (Sánchez-Peña et al., 2006; Santos et al., 2023). The insecticidal potential of compounds from the aerial parts of this plant has been demonstrated (Santos et al., 2023). The tridecan-2-one induces midgut cytochrome P450 activity in insects, which may explain this plant's non-preference (Santos et al., 2023). This makes necessary to identify the chemical composition, gas chromatography peaks, structural formula, and retention indices of molecules from *S. habrochaites* extracts.

The aim of this work was to provide gas chromatography peaks, structural formula, and retention indices of compounds in the extract of aerial parts of *S. habrochaites* to contribute to existing studies on insect-plant interaction, plant communication and ecology, and on the application of its extract in the management of arthropod pests.

## 2. Methods

### 2.1. Experimental site

The experiment was carried out at the Supramolecular and Biomimetic Chemistry Laboratory of the Department of Chemistry at the Federal University of Viçosa (UFV) and at the Insect Biological Control Laboratory (LCBI) of the

Institute of Biotechnology Applied to Agriculture (BIOAGRO) in Viçosa, Minas Gerais state, Brazil.

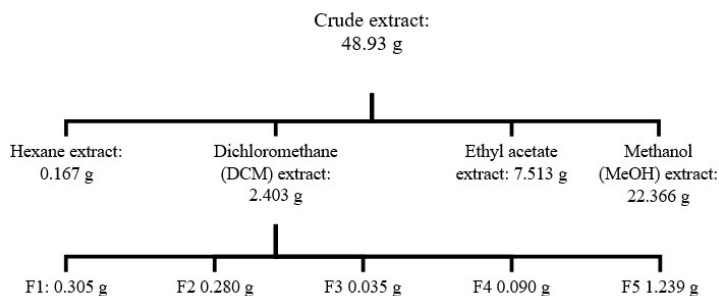
Seeds of *S. habrochaites* were collected in the campus of the UFV and cultivated in a greenhouse using standard practices (Kubond and St. Clair, 2023). The aerial parts (stems and leaves) were harvested from the cultivated plants and processed (Tavares et al., 2013).

### 2.2. Extractions and chromatographic procedures

Extractions and chromatographic procedures used analytical-grade solvents under vacuum and in open columns with silica gel 60 (70-230 mesh) as the stationary stage. Analytical thin-layer chromatography was performed using silica gel 60 G F254 plates (0.25 mm thickness) and revealed under ultraviolet light (254 and 366 nm), followed by immersion in a vanillin acid solution. The gas chromatography analyses were conducted in a Shimadzu GCMS QP-5000A chromatograph, equipped with a DB-5 Supelco capillary column (30 m × 0.25 mm × 0.25 μm) under the following conditions: electron impact method (70 eV); scan mode, 30 to 700 m/z; carrier helium (He) gas flow of 1.6 mL min<sup>-1</sup>; split ratio 1:2. The temperature program was: T1= 40 °C for 2 min, with a gradient of 20 °C min<sup>-1</sup> to T2= 300 °C; injector and detector temperatures were set at 290 °C.

### 2.3. Obtention and fractioning the extracts of the aerial parts

Stems and leaves of *S. habrochaites* were dried in a drying oven at 40 °C, followed by cold maceration extraction in pure ethanol (3 L) for seven days, a process repeated twice. The ethanolic extracts were combined, dried with anhydrous sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>), and the solvent removed using a rotary evaporator, resulting in 58.936 g of crude extract, initially fractionated by vacuum chromatography using silica gel 60 (70-230 mesh) as the stationary phase and 2.5 L of dichloromethane (DCM) as the solvent (Figure 1) (Coll and Bowden, 1986). The DCM extract was fractionated by column chromatography using silica gel 60 (70-230 mesh) as the stationary phase, and elution performed with a hexane-dichloromethane gradient of 1:0 to 0:1 (v v<sup>-1</sup>) to identify the chemical constituents of its fractions. Fractions of 10 mL were collected in test tubes (2 × 12 cm) and grouped based on the analytical change-coupled device (CCD) profile, resulting in five fractions and the crude extract (48.936 g) divided into:



**Figure 1.** Fractioning of the crude extract of *Solanum habrochaites* (Solanaceae) in grams (g) by vacuum chromatography.

hexane-dichloromethane extract 1:0 to 0:1 (v v<sup>-1</sup>) (2.403 g) [with the fractions of F1 (0.305 g), F2 (0.280 g), F3 (0.035 g), F4 (0.090 g), and F5 (1.239 g)], which were analyzed using Gas Chromatography-Mass Spectrometry (GC-MS) besides hexane (0.167 g), ethyl acetate (7.513 g), and methanol (22.366 g) extracts (Figure 1). The extraction process employed ethanol as the solvent to isolate compounds from the aerial parts of *S. habrochaites*. The ethanol esterifies fatty acids under certain conditions, and, for this reason, it is crucial to confirm that the identified esters in the extracts are not artifacts resulting from the esterification of fatty acids during the extraction process (Coll and Bowden, 1986).

Constituents were identified by comparing *S. habrochaites* extract mass spectra to chromatograph library data, with matches confirmed when retention and similarity indices values were over 90%. These indices were calculated using hydrocarbon standards  $LRI = 100 \times \left\{ \left[ \frac{t_c - t_n}{t_{n-1} - t_n} \right] + n \right\}$  (Viegas and Bassoli, 2007) with the equation:  $LRI =$  linear retention index;  $t_c =$  retention time of the compound of interest;  $t_{n+1} =$  retention time of the subsequent hydrocarbon;

$t_n =$  retention time of the preceding hydrocarbon;  $n =$  number of carbon atoms in the preceding hydrocarbon.

### 3. Results

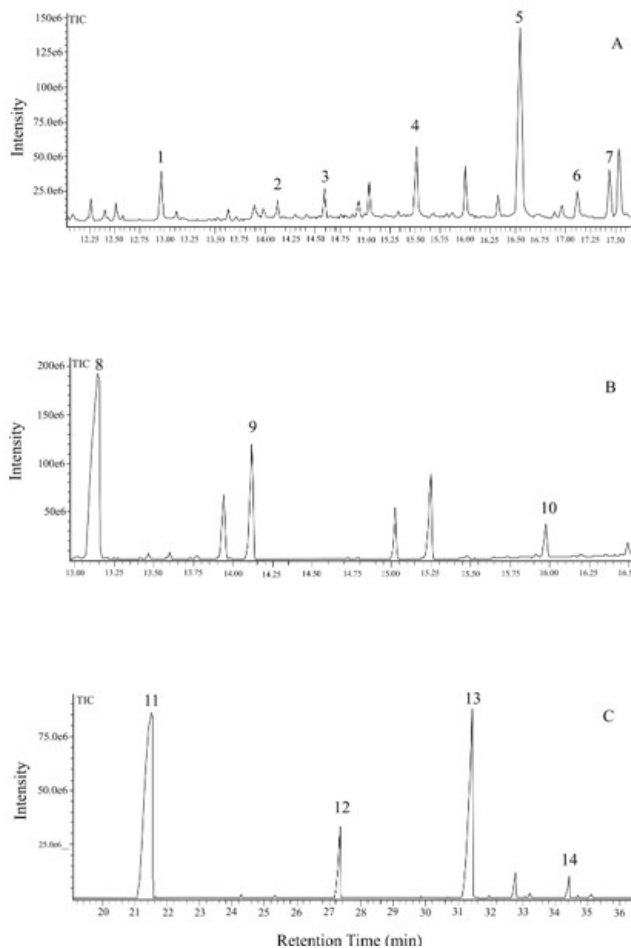
#### 3.1. Chromatography of the F1, F2, F3, F4 and F5 fractions

Fraction F1 from column chromatography identified the long-chain hydrocarbons docosane, heptacosane, hexadecanoic acid, octacosane, pentacosane, squalene, and tricosane (Figures 2, 3, Table 1).

Fraction F2 from column chromatography revealed the long-chain fatty acid esters docosanoate, hexadecanoate, and octadecenoate ethyls (Figure 3, Table 1).

Fractions F3 and F4 from column chromatography did not yield compounds with a similarity index >90% or in sufficient quantity for identification.

Methyl ketones tridecan-2-one, pentadecan-2-one, and 6,10,14-trimethylpentadecan-2-one, and the aldehyde 9,12,15-ocadecatrienal were identified in the F5 fraction of

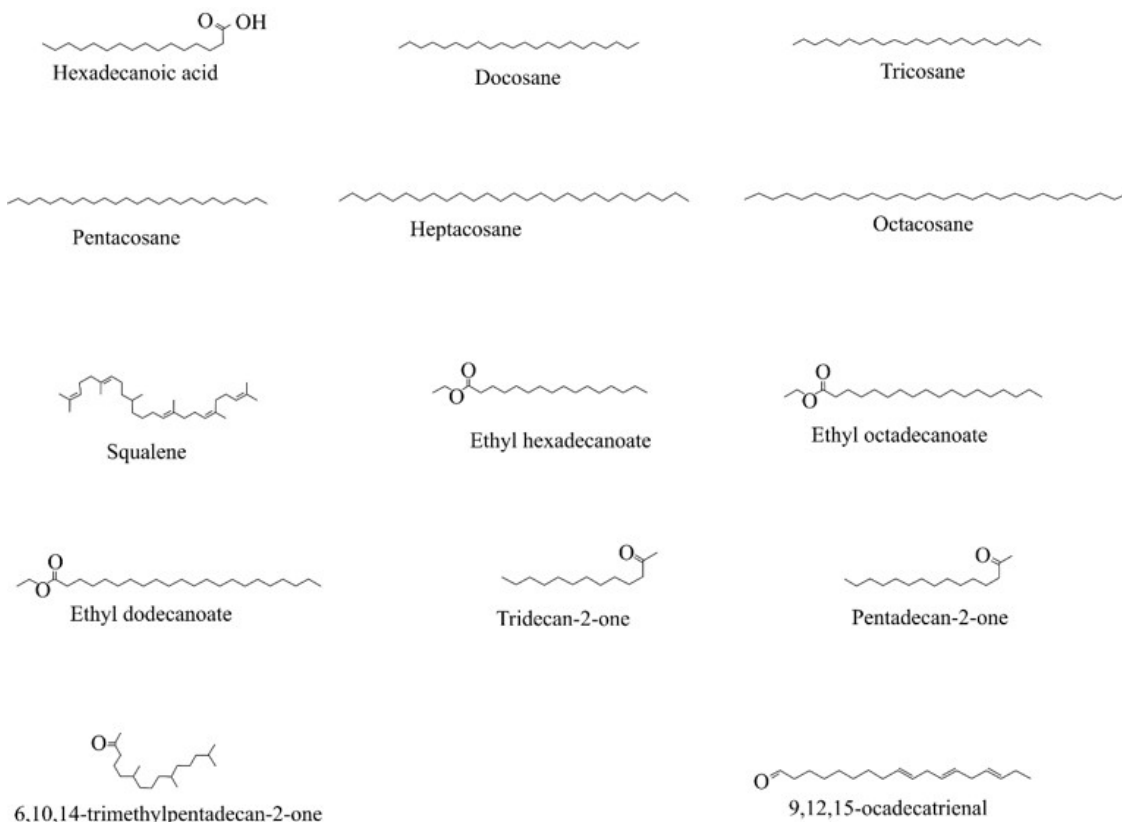


**Figure 2.** Gas chromatogram of total ions retention time and carbon index (TIC) from the F1 (A), F2 (B), and F5 (C) fractions of *Solanum habrochaites* (Solanaceae) extracts. 1- Hexanoic acid, 2- Docosane, 3- Tricosane, 4- Pentacosane, 5- Heptacosane, 6- Octacosane, 7- Squalene, 8- Ethyl hexadecanoate, 9- Ethyl octadecanoate, 10- Ethyl docosanoate, 11- Tridecan-2-one, 12- Pentadecan-2-one, 13- 6,10,14-Trimethylpentadecan-2-one, 14-9,12,15-Ocadecatrienal.

**Table 1.** Chemical composition (Comp.) of the F1, F2, and F5 fractions of *Solanum habrochaites* (Solanaceae) extract in hexane by gas chromatography-mass spectrometry and references (Ref.).

Comp.	Compound	Area	RI	RI*	Ref.
Factions	1 Hexadecanoic acid	2.23	1975	1973	1
	2 Docosane	0.99	2205	2200	2
	3 Tricosane	2.70	2305	2302	1
Fraction F1	4 Pentacosane	5.31	2506	2500	2
	5 Heptacosane	11.54	2712	2700	3
	6 Octacosane	1.58	2806	2800	4
	7 Squalene	2.97	2855	2847	2
	8 Ethyl hexadecanoate	8.91	2010	2013	5
Fraction 2	9 Ethyl octadecanoate	2.71	2204	2207	6
	10 Ethyl docosanoate	0.56	2601	2596	7
	11 Tridecan-2-one	38.02	1512	1512	8
Fraction 5	12 Pentadecan-2-one	4.46	1705	1705	9
	13 6,10,14-Trimethylpentadecan-2-one	20.05	1856	1846	10
	14 9,12,15-Ocadecatrienal	1.18	1973	1979	11

RI= Retention Index.\*reported in literature. 1- Hosseinihashemi et al. (2013), 2- Samejo et al. (2013), 3- Silva et al. (2013), 4- Naquvi et al. (2014), 5- Selli et al. (2006), 6- Ansorena et al. (2000), 7- Heinrich et al. (2002), 8- Owens et al. (1997), 9- Souza et al. (2009), 10- Shi-Dong et al. (2014), 11- Yang et al. (2012).

**Figure 3.** Structural formula of the molecules identified in the F1, F2, and F5 fractions of *Solanum habrochaites* (Solanaceae) extract in dichloromethane. Tridecan-2-one, pentadecan-2-one, 6,10,14-trimethylpentadecan-2-one, and 9,12,15-ocadecatrienal have been reported for this plant (Santos et al., 2023).

the *S. habrochaites* extract (Figure 3, Table 1), with tridecan-2-one constituting 38.02% of its total content (Table 1).

## 4. Discussion

### 4.1. Chromatography of the F1, F2, F3, F4 and F5 fractions

The long-chain hydrocarbons, docosane, heptacosane, hexadecanoic acid, octacosane, pentacosane, squalene acid, and tricosane identified in the chromatography of the F1 fraction from *S. habrochaites* extracts confirm a high diversity of compounds in this plant. This is the first record of docosane in Solanaceae. This compound, from *Sargassum fusiforme* (Sargassaceae), reduced respiratory syncytial virus (RSV) infection, one of the leading causes of bronchiolitis and pneumonia in infants (Chathuranga et al., 2021). The hexadecanoic acid can decrease the volatility of plant defense monoterpenes (Dell and McComb, 1981) and oviposition by *Tetranychus evansi* (Trombidiformes: Tetranychidae) on *Solanum sarrachoides* plants (Murungi et al., 2013). Squalene, also identified in the F1, may have a role in plant chemical defense (Kanmani et al., 2021). This compound, in *Nicotiana tabacum* (Solanaceae) leaf extracts, was linked to the toxicity against *Sitophilus oryzae* (Coleoptera: Curculionidae) (Kanmani et al., 2021). The toxicity of heptacosane and pentacosane to insects needs further study, but these compounds are of pharmaceutical relevance (Radulović et al., 2006; Satyal et al., 2015; Popova et al., 2020). Tricosane is one of the main volatile compounds attracting euglossine bees (Hymenoptera: Apidae) to *Cyphomandra* spp. (Solanaceae) plants (Sazima et al., 1993).

This is the first identification of the ethyl esters docosanoate, hexadecanoate and octadecanoate in a Solanaceae. These compounds are important, particularly, for the pharmaceutical industry (Radulović et al., 2010; Asgarpanah and Haghghat, 2012; Chung et al., 2011). Docosanoate is one of the volatile compounds found in grapes (Vitaceae) (Radulović et al., 2010), while hexadecanoate and octadecanoate ethyls are antioxidants (Chung et al., 2011), and aromatics and pharmacologicals (Asgarpanah and Haghghat, 2012), but they lack documented insecticidal activity.

The lack of identification of compounds in the F3 and F4 fractions of *S. habrochaites* may be due to factors such as inadequate resolution, contamination, or impurities, non-ideal chromatographic and variations in experimental conditions, and the complexity of the sample matrix. The similarity index, usually expressed in percentage, reflects how much the compound retention pattern is similar to a reference standard. Values below 90% indicate differences in retention periods of the unknown compound compared to the reference standard (Li et al., 2022).

The identification of the methyl ketones 6,10,14-trimethylpentadecan-2-one, pentadecan-2-one, tridecan-2-one, along with the aldehyde 9,12,15-ocadecatrienal, expands the known chemical profile of *S. habrochaites*. Further investigation of these compounds is warranted for potential applications, including insect pest management (Sánchez-Peña et al., 2006; Santos et al., 2023).

## 5. Conclusion

Chemical compounds with potential applications, such as the development of cosmetic products, botanical acaricides and insecticides, herbicides, and treatment of human diseases, were identified in the extracts of *S. habrochaites* plants through chromatography. Key compounds identified across fractions of these plants include aldehydes, fatty acid esters and methyl ketones. This is the first report of the hexadecanoate, octadecanoate, and docosanoate ethyls in this plant and in Solanaceae.

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