VALORIZATION OF *Solanum viarum* DUNAL BY EXTRACTING BIOACTIVE COMPOUNDS FROM ROOTS AND FRUITS USING ULTRASOUND AND SUPERCRITICAL CO$_2$

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**Abstract** - *Solanum viarum* Dunal belongs to the Solanaceae family and is considered to be a grazing weed toxic to cattle. In this study, ultrasound-assisted extraction (UAE) and supercritical CO$_2$ extraction (SFE-CO$_2$) were applied to evaluate the extraction yield and chemical composition of fruits and roots matrices of *Solanum viarum* Dunal. A hydroalcoholic solution (60% ethanol/40% water, v/v) was the solvent used for UAE. For comparisons, extractions with Soxhlet and maceration were carried out. For SFE-CO$_2$, the highest yield was obtained at 60°C and 250 bar. For UAE, the highest yield was obtained at an ultrasound intensity of 75.11 W/cm$^2$ and pulse cycle of 0.93. The techniques seem to be efficient for the extraction of chemical compounds, indicating a large number of bioactive compounds. The major compounds are 1,2-benzenedicarboxylic acid, quinic acid, octadecenoic acid, and solasodine. The results highlight the application of UAE to recover compounds from vegetal matrices, since it presented higher yields and more chemical constituents when compared with other techniques.

**Keywords**: *Solanum viarum* Dunal; Ultrasound-assisted extraction; Supercritical fluids; Soxhlet; Chemical composition.

**INTRODUCTION**

*Solanum viarum* Dunal belongs to the Solanaceae family and is a native plant in South America, known as “juá” or “joá-bravo” in Brazil (Braguini et al., 2018). It is described as a broad leaf herb, subshrub or shrub (Kausar and Singh, 2018). Furthermore, it is considered to be grazing weed and highly toxic plant because it can be lethal when ingested by cattle or other herds (Mentz et al., 1997; Milner et al., 2011).

Studies about the toxicity of *S. viarum* leaves are described (Braguini et al., 2018) and report interesting chemical constituents such as solasodine, caffeoylquinic acid derivatives, 5-caffeoyl, and 3-malonyl-5-caffeoyl-[4-(1-beta-[6-(5-caffeoyl) quinate] glucopyranosyl)] and quinic acid (Kausar and Singh, 2018). The fruits belonging to the Solanaceae family can accumulate high levels of alkaloids (Cipollini and Levey, 1997), which makes the study interesting since alkaloid compounds can be highly toxic to vertebrates (Braguini et al., 2018). However, reports about techniques, extraction yield, and chemical characterization of fruits and roots of *S. viarum* have not been found in the scientific literature up to date, which makes this study extremely important.

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The production of secondary metabolites by plants for defense is quite common in the plant kingdom, and may be present in agricultural crops and invasive weeds (Günthardt et al., 2018). Recently, there has been a significant increase in the use of active substances found in plant extracts due to their applications in the pharmaceutical, food and agricultural sectors (De Melo et al., 2014; Kulkami and Rathod, 2014; Zabot et al., 2016). However, some plant matrices represent potentially new applications whose knowledge, in most cases, has been empirically established or still lacks scientific coverage (De Melo et al., 2014). New techniques have been developed due to the increasing interest in bioactive compounds and in “green extraction” techniques, which overcome the problem of time and use of organic solvents (Easmin et al., 2014; Prakash Maran et al., 2017; Rouhani, 2019). Ultrasound-assisted extraction (UAE) (Wei et al., 2019) and supercritical fluid extraction (SFE) (Bubalo et al., 2015; Wei et al., 2019) are some examples of such technologies.

The type of extraction to be used plays an important role in bioactive compound extraction (Goltz et al., 2018), and UAE and SFE are being increasingly used (Kadam et al., 2013). SFE allows solvent-free extracts and, for this reason, it can be considered a green technology (Aladić et al., 2015; Moraes et al., 2015). It is also advantageous because the physicochemical properties can be adjusted by modifying the pressure and temperature conditions within the system, increasing the selectivity of extraction (Santos-Zea et al., 2019). The most commonly used solvent for extractions of bioactive compounds from natural matrices is carbon dioxide (CO\(_2\)) (Soares et al., 2018) which has advantages such as the use of mild critical temperature and pressure (31°C and 74 bar), and is non-toxic, non-flammable and non-polluting solvent (Gadkari et al., 2018).

UAE is also considered one of the main technologies to reach the target of sustainable “green” chemistry (Chemat et al., 2017). It has the advantage of decreasing extraction time and the amount of solvents (Chemat et al., 2017; Vinatoru et al., 2017; Kiss et al., 2018), as well as increasing the recovery of bioactive compounds from plants (Corbin et al., 2015). Based on this context, the objective of this work was to evaluate the UAE and SFE-CO\(_2\) technologies on obtaining extracts from fruits and roots of \(S.\) \textit{viarum}. The extract yield and chemical composition were determined.

**MATERIALS AND METHODS**

**Plant material collection and preparation**

Roots and fruits of \(S.\) \textit{viarum} Dunal were collected from a pasture area with compacted soil located at Getúlio Vargas, Rio Grande do Sul, Brazil (S: 27° 55’ 39.43/ W: 52° 7’ 37.14). The samples were dried at 40°C until reaching a moisture content of approximately 10%. Thereafter, the samples were milled (Marconi, SP, Brazil) and the particles were classified by the Mean Sauter Diameter using the Tyler series. The sizes ranging from 8 mesh to 48 mesh (0.3-2 mm) were used for subsequent steps. The samples were maintained at −4°C until the extractions.

**Soxhlet extraction**

Soxhlet extraction was performed using 1 g of vegetable matrix and 150 mL of n-hexane for 150 min in a Soxhlet apparatus (Marconi, Model MA491/6, Brazil). The extracted mass was quantified by the gravimetric method. The assays were performed in triplicate and the responses were expressed as a mean ± standard deviation. The Soxhlet extractions were used as a reference for comparing the yields and compositions obtained by SFE-CO\(_2\).

**Supercritical CO\(_2\) extraction (SFE-CO\(_2\))**

The experimental assays of SFE-CO\(_2\) were performed on a laboratory scale equipment, using carbon dioxide (CO\(_2\)) as the solvent. Details of the apparatus and procedure have been described by Confortin et al. (2019). For the extractions, approximately 10 g of samples (dried and milled) were used for each matrix and the CO\(_2\) flow rate was 4 g/min. During the kinetic extraction curves, the extracts were collected at equal intervals of 15 min. The curves were constructed for all matrices under different experimental conditions in order to determine the extraction yields as a function of time using Equation (1).

\[
\text{Yield (wt.%) = } \frac{\text{Mass of extract (g)}}{\text{Initial dry mass of the matrix (g)}} \times 100 \quad (1)
\]

The extractions were performed at temperatures of 40, 50 and 60°C and at pressures of 150, 200 and 250 bar. The fluid density (\(\rho\)) was obtained from the Chemistry WebBook - National Institute of Standards and Technology (NIST). The Tukey test was applied to determine the significant differences among the yields at the 5% uncertainty level using STATISTICA 8.0® (Statsoft Inc., USA).

**Ultrasound-assisted extraction**

Data about experimental apparatus have been described by Sallet et al. (2019). A hydroalcoholic solution (60 mL of ethanol HPLC and 40 mL of distilled water) was used for the extractions based on previous work (Dal Prá et al., 2015). The ultrasonic probe was placed at the center of the bioreactor containing 2.5 g of matrix and 100 mL of hydroalcoholic solution. Subsequently, the temperature was adjusted to 40°C ± 2°C by circulating water through a jacket. After the
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Comparison of extraction yields obtained

Yields of extracts from SFE-CO₂

The extraction yields obtained by Soxhlet and SFE-CO₂ from fruits and roots of Solanum viarum are shown in Table 1. The highest yields were obtained using Soxhlet extraction for the fruits (1.87 wt.%) and roots (0.52 wt.%). Both matrices showed similar behavior in relation to yields by SFE-CO₂, where the highest yields (0.69±0.001 wt.% fruits and 0.37±0.001 wt.% roots) were obtained in the lowest temperature and the highest pressure (40°C/250 bar) assay, while the lowest yields (0.40±0.002 wt.% fruits and 0.12±0.001 wt.% roots) were obtained in the assay of higher temperature and lower pressure (60°C/150 bar).

The increase of pressure from 150 to 250 bar resulted in a higher yield of extract. Otherwise, the increase in temperature from 40 to 60°C caused a lower yield. This effect is due to solvent density because the increase of pressure increases the density of the supercritical fluid. Consequently, the solvating power of the solvent is increased. An increase in temperature causes a density decrease and, consequently, a decrease of solubility of extracts in CO₂ (Soares et al., 2016; Goyeneche et al., 2018), Goyeneche et al. (2018) reported the same behavior of this study when studying radish leaves. The increase of pressure favored the yield while the increase of temperature hampered the yield. This effect was also identified in the works of Alvarez et al (2019) and Elgndi et al. (2017).

An important aspect to be considered when studying extraction processes are the general extraction curves (Dal Prá et al., 2016). The extraction curves are useful for improving the processes and calculating the manufacturing costs (Martínez-Correa et al., 2017). One of the difficulties of using supercritical technology is the high initial cost of investment (Goyeneche et al., 2018), which could be overcome by optimizing process parameters (Zabot et al., 2018). However, despite the high initial cost, some studies have demonstrated the economic feasibility of SFE-CO₂ (Farias-Campanoanes et al., 2013, Prado et al., 2012, Zabot et al., 2015).

The extraction curves obtained in this work (Figure 1) are divided into three stages. The first is the constant

| Table 1. Comparison of extraction yields obtained from Solanum viarum using SFE-CO₂ and Soxhlet. |
|-----------------------------------------------|
| Assay | T (°C) | P (bar) | P (kg/m³) | Yield (wt.%) |
| | | | | Fruits Roots |
| SFE-CO₂ | | | | |
| 1 | (-1) 40 (+1) 150 | 750.501 | 0.49 ± 0.002<sup>D</sup> | 0.17 ± 0.001<sup>D</sup> |
| 2 | (-1) 40 (+1) 250 | 887.334 | 0.69 ± 0.001<sup>B</sup> | 0.37 ± 0.001<sup>B</sup> |
| 3 | (+1) 60 (-1) 150 | 565.509 | 0.40 ± 0.002<sup>AE</sup> | 0.12 ± 0.001<sup>AE</sup> |
| 4 | (+1) 60 (-1) 250 | 776.890 | 0.63 ± 0.002<sup>CE</sup> | 0.24 ± 0.001<sup>CE</sup> |
| 5 | (0) 50 (0) 200 | 765.942 | 0.44 ± 0.002<sup>BE</sup> | 0.14 ± 0.003<sup>BE</sup> |
| 6 | (0) 50 (0) 200 | 765.942 | 0.43 ± 0.002<sup>AE</sup> | 0.13 ± 0.003<sup>AE</sup> |
| 7 | (0) 50 (0) 200 | 765.942 | 0.44 ± 0.002<sup>E</sup> | 0.14 ± 0.003<sup>E</sup> |

| Soxhlet | 8 | Nd | Nd | 1.87 ± 0.224<sup>A</sup> | 0.52 ± 0.118<sup>A</sup> |

(1): inferior coded level; (+1): superior coded level.

-<sup>a</sup>-<sup>e</sup> Different letters in the same column represent a significant difference at 95% (p < 0.05 - Tukey test) among the assays for each solvent.

-<sup>A</sup>-<sup>F</sup> Different letters in the same column represent a significant difference at 95% (p < 0.05 - Tukey test) among the assays between both solvents.

<sup>T</sup>: temperature; <sup>P</sup>: pressure; <sup>ρ</sup>: density; <sup>Nd</sup>: not determined.

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rate period controlled by the convective mass transfer mechanism. Posteriorly, the other period is known as diffusion and convection, where extraction occurs. The third period, where the extraction rate is low, is mainly controlled by diffusion (Alvarez et al., 2019). As can be seen in Figure 1, there was a high extraction rate at the beginning of the process and there was a rapid reduction in the extraction rate in the subsequent minutes. According to Martinez-Correa et al. (2017), this behavior indicates that the process is controlled by diffusion.

The constant extraction rate was verified for fruits in the first 60 min, while for the roots it was in the first 15 min. In these periods, most of the solutes are removed from this matrix, predominating the convective mass transfer (Valente et al., 2018). For fruits, fractions of 75.5%, 66.7%, 73.8%, 93.6% and 93.6% (mass basis) of the total mass of extract available in such matrix were recovered in assays 1-5, respectively. For roots, fractions of 66.7%, 91.5%, 84.6%, 86.9% and 80.0% (mass basis) of the total mass of extract available in such matrix were recovered in assays 1-5, respectively.

Table 2. Yields obtained from fruits and roots from *Solanum viarum* using UAE.

| Assay | Ultrasound intensity (W/cm²) | Pulse cycle (-) | Yield (wt.%) |
|-------|-------------------------------|-----------------|--------------|
|       |                               |                 | Fruits Roots |
| 1     | 26.89 (-1)                    | 0.57 (-1)       | 10.5 1.3     |
| 2     | 75.11 (1)                     | 0.57 (-1)       | 18.6 8.7     |
| 3     | 26.89 (-1)                    | 0.93 (1)        | 16.6 6.4     |
| 4     | 75.11 (1)                     | 0.93 (1)        | 20.8 11.7    |
| 5     | 17 (-1.41)                    | 0.75 (0)        | 12.0 2.9     |
| 6     | 85 (1.41)                     | 0.75 (0)        | 19.5 10.2    |
| 7     | 51 (0)                        | 0.50 (-1.41)    | 16.9 6.9     |
| 8     | 51 (0)                        | 1.0 (1.41)      | 18.2 8.2     |
| 9     | 51 (0)                        | 0.75 (0)        | 17.1 7.1     |
| 10    | 51 (0)                        | 0.75 (0)        | 17.9 7.2     |
| 11    | 51 (0)                        | 0.75 (0)        | 17.8 7.5     |
| 12a   | 0                             | 0               | 9.7 1.1      |

*a - Maceration - control (Extraction without ultrasound).*

to 20.8 wt.% for fruits and from 1.3 wt.% to 11.7% wt.% for roots. The two matrices demonstrated very similar behaviors, having the highest yields in assay 4 with high intensity (75.11 W/cm²) and a pulse cycle of 0.93. The lowest yield was obtained in assay 1 with the lowest intensity and pulse cycle.

Comparing the assays 4 (highest yield) and 12 (maceration control), the yields increased approximately 2 and 10 times for fruits and roots, respectively. These data are corroborated by Rouhani (2019), who obtained a higher yield (79%) in stevioside extraction from *Stevia rebaudiana* using UAE, when compared to the control (22%). According to Sallet et al. (2019), whose findings also corroborate the results of the current work, the main reason for this behavior is the increase of mass transfer in the system when ultrasound is employed. In the current study, the effect of cavitation and its subsequent effects allowed a breakdown of the cell walls of the plant matrix, improving the penetration of solvent and facilitating the release of extractable compounds, consequently increasing the yields, as reported elsewhere (Picó, 2013; Bernardo et al., 2018).

The data presented in Table 2 were used to calculate the linear, quadratic and interaction terms of the process variables in the response, which were expressed as a Pareto chart in Figure 2. The linear terms for the pulse cycle and ultrasound intensity were statistically significant (p < 0.05) for both matrices. The intensity of ultrasound showed a positive effect, whose increase may lead to higher yields for both matrices. When comparing assays 1-2 and 3-4, the highest yields were obtained at the highest ultrasound intensity (75.11 W/cm²), when the pulse cycle is maintained constant at levels -1 and +1, respectively. According to Chemat et al. (2017), the ultrasound intensity is a relevant parameter that strongly affects the efficiency of extraction because its increase causes a strong bubble collapse, breaking the plant structures.

**Figure 1.** Kinetic curves for extraction of bioactive compounds from *Solanum viarum* using SFE-CO₂ for (a) fruits and (b) roots matrices.

**Ultrasound-assisted extraction**

Table 2 shows the extraction yields obtained in the eleven assays of CCRD, ranging from 10.5 wt.% to 20.8 wt.% for fruits and from 1.3 wt.% to 11.7% wt.% for roots. The two matrices demonstrated very similar behaviors, having the highest yields in assay 4 with high intensity (75.11 W/cm²) and a pulse cycle of 0.93. The lowest yield was obtained in assay 1 with the lowest intensity and pulse cycle.

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The quadratic term for ultrasound intensity showed a negative effect on matrix yields. The negative signals of the quadratic term indicate the presence of a maximum point for the ultrasound intensity in the evaluated range, showing that it would be interesting to optimize the ultrasound intensity before extractions on a larger scale (Kulkarni and Rathod, 2014), since it can reduce the energy costs (Sallet et al., 2019).

In order to show the influence of ultrasound intensity and pulse cycle on the extraction yield, contour plots were generated (Figure 3). UAE of extracts from fruits of *Solanum viarum* yielded the maximum extraction yield for ultrasound intensity ranging from 55 W/cm² to 85 W/cm², and pulse cycle ranging from 0.5 to 1. For the roots, the maximum extraction yield was obtained for ultrasound intensity ranging from 60 W/cm² to 85 W/cm² and pulse cycle ranging from 0.75 to 1. The increase of intensity and pulse cycle favors the recovery of extracts from the matrices studied in this work.

The UAE technique also presented yields superior to SFE-CO₂ and Soxhlet techniques, and in a shorter time. The UAE increased the efficiency of canola oil extraction compared to the Soxhlet method (Jalili et al., 2018). Positive effects of UAE for obtaining euphol from *Euphorbia tirucalli* (Vuong et al., 2015) and *Moringa peregrina* oil (Mohammadpour et al., 2019) were also reported. According to Mohammadpour et al. (2019), the application of ultrasound has become a recent and promising method in oil extraction from plants.

To better understand the efficiency of the different techniques and the structural changes that occurred before and after extractions, the surface of the particles of *S. viarum* matrices was evaluated by SEM. For fruits, the seeds were analyzed separately. The surface of matrix tissues showed integrity before extraction, but was slightly damaged after extractions. With the application of all of the techniques, there were changes in the tissue surface (Figures 4 and 5).

SFE-CO₂ showed damage to cells in the form of cracking and scaling, much higher than when the Soxhlet was applied. In maceration, a crushing occurred, while cavitation breaks the entire surface of
leaves. The UAE process showed more visible changes such as openings in the cellular structures that could be correlated with explosive rupture. This could be evidence of the occurrence of cavitation phenomena, which facilitate the general extraction process (Patil and Akamanchi, 2017). The images revealed that more cracks and pores were created by ultrasound, favoring the extraction more rapidly.

The implosion of cavitation bubbles on the surface of matrices induces the erosion of released structures in the extraction medium. Some authors have reported the erosion of plant material when it was treated by ultrasound. For example, UAE of boldo leaf was studied by Petigny et al. (2013) using an ultrasound probe. Mohammadpour et al. (2019) also described structural changes in *Moringa peregrina* using different

Figure 4. SEM images of roots before and after extraction: (A) fresh (before extractions); (B) Soxhlet extraction; (C) SFE-CO$_2$; (D) maceration; and (E) UAE.

Figure 5. SEM images of fruits before and after extraction: (A) in nature; (B) Soxhlet extraction; (C) SFE-CO$_2$; (D) maceration; (E) UAE; (1) seeds and (2) husks.
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extraction techniques (UAE and Soxhlet). Zhou et al. (2018) also observed damage in mulberry leaves using the UAE technique, indicating this method as convenient and versatile for the effective extraction of bioactive phytochemical products. Zhong et al. (2018) suggest that ultrasound is the most effective way to destroy the cellular structure of seed samples and favor the release of the solvent extraction oil used.

**Chemical composition**

The extracts obtained from fruits and roots of *Solanum viarum* contain phytochemical constituents. Most of them are known to be biologically active compounds and are responsible for displaying various activities. Twenty compounds were identified and quantified in extracts from both matrices obtained under different conditions using SFE-CO$_2$ (Table 3).

Almost all the identified compounds were found in the two morphological parts studied. However, the percentage is different for some of them. The major compounds for SFE-CO$_2$ in both matrices were 1,2-benzenedicarboxylic acid, octadecanoic acid, hexadecanoic acid, and Vitamin E. These compounds were also identified and reported as to their bioactivity by various authors. For instance, Ali et al. (2017) describes 1,2-benzenedicarboxylic acid as an antifungal compound, responsible for growth control of *Fusarium oxysporum* and *Lycopersici sp.*. Verma et al. (2014) extracted 1,2-benzenedicarboxylic acid from *Aspergillus flavipes* and tested its antifungal efficacy against *Sclerotin sclerotiorum*. Govindappa et al. (2014) and Krishnan et al. (2014) reported antimicrobial, cytotoxicity, antioxidant, anti-inflammatory and antiviral activities for this referenced compound.

According to Linton et al. (2013), octadecanoic acid exhibits antiviral, antioxidant and antibacterial activities, and according to Selvamangai and Bhaskar (2012), it is responsible for hypcholesterolemic, antiarthritic and nematocide actions. Sanseera et al. (2013) studied the chemical composition and biological activities of leaf essential oil of *Cleidion javanicum*, which presented significant antimicrobial, antioxidant and anticancer activity, wherein its composition is hexadecanoic acid (26.77%). Selvamangai and Bhaskar (2012) and Agoramorothy et al. (2007) attribute anti-inflammatory, antioxidant and antibacterial activities to this compound.

Another aspect to be highlighted is the higher yields and the target compounds recovered in the processes. Different compounds were observed under conditions of temperature and pressure (Table 3). The condition of the highest yield was not the same as the one with the highest percentage of area in the major compounds. These findings indicate that SFE-CO$_2$ was more selective to extract the largest number of compounds in less quantity of extract. Interfering compounds or inhibitors can be extracted in larger quantities under better yield conditions, thus reducing the quality of extract (Confortin et al. 2017). When comparing the percent area of compounds, it is evident that supercritical CO$_2$ exceeded the conventional method of Soxhlet extraction. Although the Soxhlet technique can provide higher yields, the extract has

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**Table 3.** Chemical compounds obtained by SFE-CO$_2$ and Soxhlet for fruits and roots matrices of *Solanum viarum*.

| Assay | Relative area (%) | Fruits | Roots |
|-------|-------------------|--------|-------|
|       | Compounds | SFE-CO$_2$ | Soxhlet | SFE-CO$_2$ | Soxhlet |
| 2-Heptenal | 1.13 2.56 | 1.66 | 1.04 | 1.85 2.5 | 2.1 | 1.2 | 1.89 | 0.89 |
| 2,4-Decadienal | 3.58 1.66 | - | - | 2.0 | 1.86 2.86 | 2.56 | 2.10 | 1.52 |
| Hexadecanethioylecctosiolane | 1.05 1.01 | 0.89 2.61 | 2.52 1.68 | 1.52 1.58 | 1.89 1.92 | 1.83 2.0 |
| 2-Hexadecen-1-ol, 3,7,11,15-tetramethyl- | 1.38 1.14 | 0.98 2.53 | 1.04 2.0 | 0.89 0.65 | 0.96 0.88 | 1.0 0.70 |
| 3,7,11,15-Tetramethyl-2-hexadecen-1-ol | 0.92 1.12 | - | - | - | - | - | - |
| Benzoic acid | 1.22 1.20 | 1.31 1.27 | 1.23 2.49 | 1.69 1.63 | 2.89 3.89 | 4.72 1.0 |
| 9-Octadecenoic acid | 1.50 2.71 | 1.57 1.85 | 3.21 1.42 | 1.86 1.05 | 1.89 2.13 | 2.05 0.89 |
| Hexadecanoic acid | 11.15 8.67 | 9.22 8.45 | 8.08 4.37 | 8.56 4.52 | 5.56 3.58 | 5.58 2.05 |
| Nonadecane | 1.93 1.16 | 1.10 2.56 | 1.96 2.89 | 1.56 1.30 | 1.89 1.93 | 2.0 0.2 |
| 9,12-Octadecanoic acid | 12.55 10.0 | 15.10 18.11 | 19.10 3.41 | 10.52 11.65 | 13.56 10.58 | 9.85 5.58 |
| 1,2-Benzenedicarboxylic acid | 40.0 25.0 | 22.31 25.08 | 25.28 4.91 | 25.58 22.10 | 25.58 30.52 | 28.56 10.52 |
| Octadecanol | 2.30 3.26 | 1.26 | 4.10 | 2.86 2.1 | 2.0 2.23 | 2.32 1.56 |
| Neophytadiene | 1.09 1.19 | - | - | 2.0 | 1.5 1.6 | 0.76 0.86 | 0.86 |
| Linalool | 1.90 1.10 | 2.45 1.94 | 2.20 1.0 | 1.01 1.02 | 2.02 2.65 | 3.58 - |
| Eicosane | - 2.37 | 1.25 1.52 | 2.58 - | 1.58 1.96 | 1.86 2.0 | 2.01 1.2 |
| Vitamin E | 10.84 10.17 | 20.31 19.81 | 20.88 5.58 | 8.56 6.58 | 7.62 8.58 | 8.02 4.28 |
| Farnesol | 2.25 1.05 | 2.89 1.60 | 3.0 1.62 | 1.89 1.25 | 1.10 2.0 | 1.96 - |
| Octadecane | 2.52 2.85 | 3.96 3.69 | 3.85 1.02 | 1.50 1.83 | 2.76 3.06 | 3.89 0.42 |
| Cetylmonoil | 1.0 2.0 | 1.30 2.89 | 2.35 0.89 | 1.56 0.89 | 0.96 0.89 | 1.10 - |
| 2,6,10,14,18-Pentamethyl | 0.82 1.50 | 1.12 | 1.0 | 1.72 0.50 | 0.96 1.0 | - |
| Waxes | 1.96 18.38 | 13.05 2.17 | 1.0 | 60 | 22.05 33.03 | 20.90 19.54 | 16.68 65.95 |
higher amounts of waxes, influencing the identification of bioactive compounds.

The 46 compounds identified using UAE (Table 4) were higher than those obtained by SFE-CO\textsubscript{2} (non-polar solvent). The results found in this work can be explained by higher solubility of extracts in hydroalcoholic solution, as compared to CO\textsubscript{2}. As CO\textsubscript{2} is non-polar, the solubility of highly polar compounds is low (Cabaleero, 2018; Alvarez et al., 2019). There are some reports confirming that the ultrasound-assisted method results in increased extraction yield of various phytochemicals at reduced extraction time and lower solvent consumption (Jalili et al., 2018; Zhong et al., 2018).

The highest number of compounds was obtained at 85 W/cm\textsuperscript{2} of ultrasound intensity and 0.75 of pulse

### Table 4. Chemical compounds obtained by UAE for fruits and roots matrices of *Solanum viarum*.

| Assays                                      | 1   | 2   | 3   | 4   | 5   | 6   | 7   | 8   | 9/10/11 | 12  | US  | Control |
|---------------------------------------------|-----|-----|-----|-----|-----|-----|-----|-----|---------|-----|-----|---------|
| Carboxylic acid                            | 0.14| 0.05| 0.59| 0.56| 0.76| 0.36| 0.41| 0.87| 0.87/0.82| 0.74|     |         |
| Benzoic acid                                | 1.11| 1.29| 1.87| 1.87| 0.41| 0.66| 0.87| 0.87| 0.79/0.77| 0.63|     |         |
| Fumaric acid                                | 1.19| 1.87| 1.87| 1.87| 0.79| 0.66| 0.87| 0.87| 0.79/0.77| 0.63|     |         |
| Benzenedicarboxylic acid                    | 1.14| 1.13| 1.79| 3.11| 2.56| 0.49| 0.91| 0.91| 0.79/0.91| 0.82|     |         |
| 2-Methoxy-4-vinylphenol                     | 1.00| 1.00| 1.00| 0.49| 0.91| 1.12| 0.96| 0.96| 0.79/0.91| 0.82|     |         |
| Citrinol                                    | 0.74| 1.00| 0.87| 0.49| 0.91| 1.12| 0.96| 0.96| 0.79/0.91| 0.82|     |         |
| 9-octadecenoic acid                         | 4.00| 4.06| 3.24| 5.26| 4.25| 4.25| 4.28| 3.25| 3.96/0.40|     |     |         |
| Butane-1,2,4-triol, 3-benzyl ether           | -   | 0.92| 1.39| -   | 0.51| 0.94| 0.72|     | 0.89/0.72| 0.89|     |         |
| 2-Fumaric acid                              | 0.44| 0.44| 0.75| 1.16| 0.50| 1.93| 0.77| 0.64| 0.63/0.63|     |     |         |
| 1,2-Ethanedithiol, diaceteic acid           | 0.38| 0.38| 0.37| -   | 0.71| -   | 0.50|     | 0.15/0.12| 1.32|     |         |
| Cyclpentanone                               | 0.24| 0.24| 0.33| -   | 0.40| 0.40| 0.58| 0.69| 0.96/0.63|     |     |         |
| 1-Propyl-4-one, 3-hydroxy-2-methylphenol     | 3.25| 3.25| 0.98| 3.61| 3.12| 1.06| 1.85| 2.00| 2.97/1.98|     |     |         |
| Phenol, 2-methoxy                          | 3.65| 3.30| 2.90| 2.30| 3.64| 3.43| 3.81| 1.48| 2.58/1.21|     |     |         |
| 2-oxepanone                                 | 0.51| -   | 0.51| -   | 0.90| 0.33| 1.06| 0.32| 1.12/1.12|     |     |         |
| Propionoic                                  | 1.65| 0.98| 1.38| 3.23| -   | 0.51| 1.53|     | 1.12/1.03| 1.65|     |         |
| Ethanol, 1-(2,6,6-trimethyl-1-cyclohexen-1- y) | 0.72| -   | 0.72| -   | 1.45| 1.79| 1.16|     | 1.60/1.60|     |     |         |
| 2,7-dimethyl-4,4-octadiol                   | -   | 1.17| 0.62| 0.80| 0.84| 0.90| 0.50|     | 0.44/0.44|     |     |         |
| 4-Methyl-1,2,5-dimethoxybenzaldehyde        | -   | 2.31| -   | 1.79| -   | 2.89| -   | 0.46/0.29| 0.30/0.36| 1.02/1.40|     |         |
| 1-Hexanal-2-amine                           | -   | -   | 1.00| 1.00| 1.01| 1.00| 0.33|     | 0.35/0.28|     |     |         |
| Glutamic acid                               | 0.89| 0.84| 0.90| 1.00| 0.87| 1.12| 1.14| 1.13| 1.56/1.29|     |     |         |
| di-Proline                                  | -   | -   | -   | -   | -   | -   | -   | 0.15/0.27| 0.46/0.32| 0.81/0.31|     |         |
| 1,8-Diazabicyclo[5.4.0] undec-7-ene           | 2.56| 2.56| 2.07| 4.00| 3.56| 4.48| 3.56| 2.26| 2.89/2.45|     |     |         |
| Ethyl linoleate                             | 3.25| 4.48| 4.12| 5.53| 5.53| 5.70| 5.74| 5.84| 5.30/6.25|     |     |         |
| 2-Pyrrolidin-1-methyl                       | -   | -   | -   | -   | -   | -   | -   | 1.51/0.75| 0.46/0.32| 0.81/0.31|     |         |
| Decanoic acid                               | 3.56| 3.56| 3.56| 3.56| 3.56| 3.56| 3.56| 3.56| 2.45/2.45|     |     |         |
| N-Methyl-4-proline                          | -   | 1.00| 1.00| 1.00| 1.00| 1.00| 1.52| 2.00| 2.00/2.00|     |     |         |
| Milbemycin                                  | 1.12| 1.12| 1.25| 1.25| 1.25| 1.25| 1.25| 1.25| 1.25/1.25|     |     |         |
| Assays                                      | 1   | 2   | 3   | 4   | 5   | 6   | 7   | 8   | 9/10/11 | 12  | US  | Control |
| Compounds                                   | 0.63| 1.59| 1.89| 1.89| 0.96| 1.56| 0.44| 1.37| 1.18/2.0|     |     |         |
| Table 4. Chemical compounds obtained by UAE for fruits and roots matrices of *Solanum viarum*. |
cycle, showing that high ultrasound intensities lead to extraction of more quantity of bioactive compounds. As previously mentioned, the positive effect of increased power on yield can be explained by violent collapses of cavitation bubbles, causing further destruction of the cell walls of the raw material and, therefore, facilitating the access of solvent to the analytes (Goltz et al., 2018; Santos et al., 2019).

The major compounds were the same for both matrices: quinic acid, cytidine, 1,2-benzenedicarboxylic

Table 5. Activities reported in the literature of some compounds found in this work.

| Compound                        | Bioactivity                      | Reference               |
|---------------------------------|----------------------------------|-------------------------|
| Farnesol                        | Cytotoxic                        | Santos et al. (2019)    |
|                                 | Antifungal                       | Piochon et al. (2009)  |
| 2-heptenal                      | Aromatic compound                | Miranda et al. (2008)  |
|                                 |                                  | Chen et al. (2018)      |
| 2,4-decadienal                  | Aromatic compound                | Miranda et al. (2008)  |
|                                 |                                  | Chen et al. (2018)      |
| Phenol                          | Antimicrobial                    | Muthulakshmi et al. (2012) |
|                                 | Antioxidant                      | Barreto and Vootla (2018) |
| Celidoniol                      | Antimicrobial                    | Barreto and Vootla (2018) |
| Nonadecane                      | Antimicrobial                    | Barreto and Vootla (2018) |
| Eicosane                        | Cytotoxic                        | Hsouna et al. (2011)   |
| Octadecane                      | Antitumoral                      | Amanian and Brindha (2013) |
| Glucitol                        | Surfactants                      | Abu backer and Devi (2015) |
|                                 | Emulsifying and stabilizing effects | Marques et al. (2016) |
| Ethyl linoleolate               | Fragrance                        | Aminah et al. (2013)   |
| Linalool                        | Antimicrobial                    | Sanseera et al. (2013) |
|                                 | Antioxidant                      | Lichtfouse (2013)      |
| Propanetriol                    | Anti-inflammatory                | Casuga et al. (2016)   |
|                                 | Anticancer                       | Foo et al. (2015)      |
| 2,3-Dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one | Anti-inflammatory | Foo et al. (2015)      |
| Benzoic acid                    | Antioxidant                      | Foo et al. (2015)      |
| 2-Methoxy-4-vinylphenol         | Antioxidant                      | Foo et al. (2015)      |
| Benzofuran, 2,3-dihydro         | Anti-inflammatory                | Foo et al. (2015)      |
| Phenol, 2-methoxy               | Antimicrobial                    | Foo et al. (2015)      |
| Decanoic acid                   | Nematicidal                      | Selvamangai and Bhaskar (2012) |
|                                 | Pesticide                        |                         |
|                                 | High toxicity                    |                         |
|                                 | Insecticidal                     |                         |
| 2-Propanoic acid                | Herbicidal                       | Fraga et al. (2017)    |
| Piperazine                      | Nematicidal                      | Demuner et al. (2001)  |
| Mannofuranoside                 | Anti-bacterial                   | Hugar and Londonkar (2017) |
| Ascaridole                      | Antifungal                       | O’Neill et al. (2010)  |
|                                 | Hepatotoxic                      | Bossou et al. (2013)   |
| 1,8-Diazabicyclo[5,4,0]undec-7-ene | Herbicidal                 | De Castro et al. (2016) |
| Pyrethroid                      | Antimicrobial                    | Chattapadhyay and Dureja (2006) |
|                                 | Insecticidal                     | Viegas Júnior (2003)   |
acid, alpha mannofuranoside, octadecenoic acid and spirosol-5-en-3-ol (solasodine). These activities have been reported in the literature. Walters et al. (2004) reported that 9-octadecenoic acid or oleic acid is an antifungal agent against Crinipellis pernicosa and Pythium ultimum. Ali et al. (2017) corroborate this result, presenting the fungal activity for this compound. It is also described as an antimicrobial and nematicide by Chandrasekaran et al. (2008). The compound milbemycin is reported to be an antiparasitic (Hugar and Londonkar, 2017) and Martin et al. (2017) attributed antiviral action to cytidine, which is a pyrimidine core compound that plays a vital role in biological activities such as antifungal. The compound solasodine is a highly toxic glycoalkaloid commonly found in the Solanaceae family, described by some authors (Al et al., 2016; Kausar and Singh, 2018; Patel et al., 2013; Yuan et al., 2017). Zeng et al. (2009) reported quinic acid anti-inflammatory activity.

In addition, as can be seen in Tables 3 and 4, other bioactive compounds were identified in S. viarum extracts (Table 5). The findings of chemical constituents corroborate that described by Kausar and Singh (2018) when studying the leaves of S. viarum, such as solasodine and quinic acid. Braguini et al. (2018) reported toxicity of S. viarum fruits against Artemia salina. High percentages of polyphenols and tannins were described, concluding that the fruits are highly toxic and may be a risk to livestock because they are available in the pastures.

CONCLUSIONS

In conclusion, this study showed that higher temperature and higher pressure presented higher extraction yields for SFE-CO₂. However, this condition did not provide the highest percentage of the major compounds. When comparing this technique with Soxhlet, it showed higher yields. However, the superiority of SFE-CO₂ in the composition is visible. For UAE, yields and chemical composition were higher than SFE-CO₂, noting that higher values of intensity and pulse cycle favored the increase of yields. In general, it can be concluded that the UAE and SFE-CO₂ techniques are efficient for extraction from S. viarum and the extracts obtained are a potential source of bioactive compounds, which must be further studied regarding the potential activities.

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