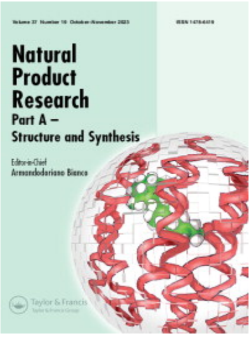

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Natural Product Research

Formerly Natural Product Letters

ISSN: (Print) (Online) Journal homepage: www.tandfonline.com/journals/gnpl20

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To cite this article: Oseias Souza da Silva Júnior, Celeste de Jesus Pereira Franco, Ângelo Antônio Barbosa de Moraes, Mayara Pastore, Marcia Moraes Cascaes, Lidiane Diniz do Nascimento, Mozaniel Santana de Oliveira & Eloisa Helena de Aguiar Andrade (2023) Chemical variability of volatile concentrate from two *Ipomoea* L. species within a seasonal gradient, Natural Product Research, 37:19, 3344-3351, DOI: 10.1080/14786419.2022.2070618

To link to this article: https://doi.org/10.1080/14786419.2022.2070618

NATURAL PRODUCT RESEARCH
2023, VOL. 37, NO. 19, 3344–3351
https://doi.org/10.1080/14786419.2022.2070618

SHORT COMMUNICATION

Chemical variability of volatile concentrate from two *Ipomoea* L. species within a seasonal gradient

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ABSTRACT

In the present study, are extracted volatile concentrate from *Ipomoea asarifolia* Poir. and *Ipomoea setifera* (Desr.) Roem. & Schult. in five-month seasonal gradient. The flowers were subjected to simultaneous distillation – extraction (SDE). The chemical composition of the volatile concentrate was determined by gas chromatography (CG/MS) and (CG-FID). Principal Component Analysis (PCA) and Hierarchical Clustering Analysis (HCA) were performed with the chemical constituents. It was observed that the chemical composition of *I. asarifolia* varied more with seasonality in relation to the species *I. setifera*. Furthermore, there is a possibility that germacrene D and α-copaene, the main components of the variation volatile of *I. asarifolia* and with higher concentrations in the rainy months, have ecological importance, attracting specific pollinators for the rainy season. This is the first study to report the chemical composition of the volatile compounds of *I. asarifolia* and *I. setifera* along a seasonal gradient.

ARTICLE HISTORY
Received 21 November 2021
Accepted 20 April 2022

KEYWORDS
Ipomoea asarifolia; *Ipomoea setifera*; melittophily; chemical ecology; seasonality; floral visitors; pollination

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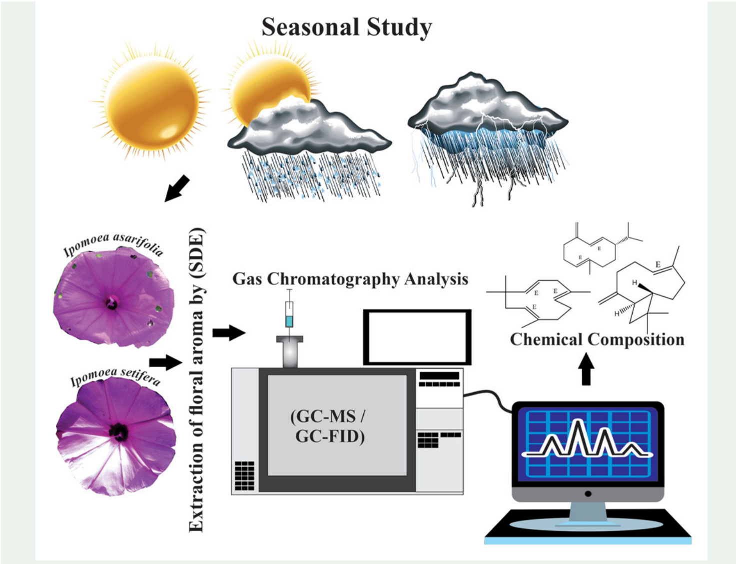
Supplemental data for this article can be accessed online at https://doi.org/10.1080/14786419.2022.2070618.



1. Introduction

Convolvulaceae comprises about 1880 species, 59 genus and 12 tribes (Stefanović et al. 2003; Staples et al. 2012), known primarily for *Ipomoea batatas* (L.) Lam., the 'batata doce' (Srisuwan et al. 2006), various ornamental plants, such as 'glórias da manhã' (Stefanović et al. 2002), and also for various weeds for agriculture (Kissmann and Groth 1992; Lorenzi 2014). This family mainly includes vines, herbaceous or woody, less often with prostrate, ascending or erect branches and rarely trees (Simão-Bianchini and Pirani 1997; Austin 2004; Delgado Júnior et al. 2014).

The *Ipomoea* L. genus is the most abundant and richest of the family (Austin and Bianchini 1998; Wilkin 1999) with pantropical distribution and about 800 species (Wood et al. 2020). *Ipomoea* flowers are often showy and colorful, but with little or no perceptible odor to the human sense of smell (Austin and Cavalcante 1982; Staples et al. 2012).

Some ruderal species of *Ipomoea* with a long flowering period are frequent in Northern Brazil: *Ipomoea asarifolia* (Desr.) Roem. & Schult. which blooms from March to December, and *Ipomoea setifera* Poir., which blooms all year round (Austin and Cavalcante 1982). Both species are possibly melitophilous, pollinated by bees (Schlising 1970; Kiill and Ranga 2003). *Ipomoea asarifolia* has been confirmed to occur in South and Central America, tropical Africa, East Asia and India (Khaled et al. 2017; Wiersema 2019). This species of *Ipomoea* causes several cases of poisoning in livestock, especially during the dry season (Austin and Bianchini 1998; Costa et al. 2011; Carvalho et al. 2014; Souza da Silva Júnior et al. 2021).

Ipomoea setifera is more frequent in tropical America, place of its probable origin, occurring in forest edges, roadsides, and also being considered a grass weed (Simão-Bianchini 2002; Hassler et al. 2021). The variation in the aromatic profile directly impacts the pollination dynamics and is linked to the physiological response of each species. Thus, the present study describes the influence of the transition from the dry to the rainy season on the aromatic profile of the flowers of ruderal of *Ipomoea* species, in an urban region within the Amazon Forest.

2. Results and discussion

More than 90% of the aromatic profiles of the samples of *I. asarifolia* and *I. setifera* were characterized, obtaining a total of 37 identified constituents (Table S3), with alkanes and sesquiterpenes being the most abundant constituents in the samples. (*E*)-caryophyllene (29.4 – 35.84%), *n*-pentadecane (35.13 – 64.15%) and β-elemene (2.54 – 5.62%) were the major constituents of the volatile concentrate of *I. setifera*. While *I. asarifolia* had the volatile concentrate characterized by high concentrations of *n*-pentadecane (16.54 – 47.79%) and sesquiterpenic hydrocarbons germacrene D (17.45 – 28.21%) and (*E*)-caryophyllene (16 – 31.4%).

The relative results of the Pearson correlation (Table S2) for the classes of compounds in relation to the climatological data indicate that the strongest linear correlations were between the volatiles of *I. asarifolia* versus the averages of precipitation and insolation.

The alkanes from *I. asarifolia* showed a directly proportional correlation with the variation in insolation ($r = 0.936$ and p-value of 0.019) and inversely proportional to the variation in precipitation ($r = -0.837$ and p-value of 0.077). The sesquiterpenes showed an inverse behavior: correlation inversely proportional to the AWI variation ($r = -0.849$ and p-value of 0.069) and directly proportional to the AWR variation ($r = 0.27$ and p-value of 0.023).

The main volatile among the alkanes is *n*-pentadecane, the majority compound in *I. asarifolia* samples from August to October. The concentration of *n*-pentadecane decreases along the gradient as mean insolation decreases and precipitation increases. This contradicts studies carried out with other species, which indicate an increase in the production of this constituent in rainier seasons (Assad et al. 1997; Syamsudin et al. 2019). On the other hand, *I. setifera* showed low correlations in all analyzes performed, with the highest correlation index between its sesquiterpenes and temperature variation ($r = 0.313$ and p-value of 0.608). These results may indicate that the aromatic profiles of the two species of *Ipomoea* have different responses to the change in precipitation and insolation of the seasonal gradient.

In this study, temperature and humidity parameters remained constant during the seasonal gradient, but considerable variation was observed in precipitation and insolation values, key factors to delineate the transition from summer to winter in the Amazon Region (Harris et al. 2004; Souza et al. 2009). Previous studies have shown the influence of rainfall variation on volatile production and composition (Gaspar et al. 2021), including in the volatile floral production (Vallat et al. 2005; Fernandes et al. 2017; Syamsudin et al. 2019). However, no studies were found reporting the influence of seasonality on the chemical composition of the volatile compounds of Convolvulaceae species.

The HCA and PCA multivariate analysis were applied to the chemical compounds identified in the volatile concentrate of *I. setifera* and *I. asarifolia*, collected during the seasonal gradient from August to December 2019 (Figures S1 and S2). The HCA

(Figure S1) indicated the formation of four distinct groups: group I formed by samples ISAUG, ISSEP, ISDEC and ISOCT (52.12%); group II formed only by the ISNOV sample (similarity of 26.41%); group III formed by the IAAUG sample (25.21% similarity); and the fourth and last group, formed by the samples IASEP, IANOV, IADEC and IAOCT (similarity between them of 51.68%).

A difference was observed between the volatile concentrate of the two analyzed species and, possibly, a seasonal variation in the aromatic profile of *I. asarifolia*, with the sample collected in August different from the others. The PCA axis 1 eigenvalue (PC1) explained 47.2% of the data variation, while axis 2 eigenvalue (PC2) explained 23.3% of the variations. The two axes together explain 70.5% of the variation (Figure S2). Chemometric analyzes such as this can be used in work on natural products, especially with essential oils and volatile concentrates, to look for possible similarities between the chemical profile of samples of the same species or species of the same genus (Fattahi et al. 2016; Buriani et al. 2017; S.G. Silva et al. 2018; Ayub et al. 2021; Franco et al. 2021).

Group I correspond to all samples of *I. setifera*, except ISNOV. The compounds with the highest weights in the formation of this group were the oxygenated monoterpene linalol, the alkane *n*-pentadecane and the sesquiterpenes (*E*)-caryophyllene, spathulenol, (*E,E*)-α-farnecene, β-elemene and pogostol. The *n*-pentadecane was the major constituent in all samples of *I. setifera*, being one of the main attractive components of orchids pollinated by males of Hymenoptera (Singer 2002; Bohman et al. 2020). The compounds (*E*)-caryophyllene and β-elemene were described as attractive volatiles for bees, especially when the two compounds are combined (Zhang et al. 2016; Zhang 2018). The presence of these constituents in the volatile concentrate of *I. setifera* tends to be consistent with the melitophyte pollination syndrome of the species (C.E. Silva et al. 2018).

Group II corresponds to the ISNOV sample and was characterized by the high presence of the α-humulene sesquiterpene hydrocarbon. Seasonal variations can affect the production of this volatile (Facanali et al. 2020), however, no studies were found relating α-humulene variations with precipitation variation. Furthermore, group II is formed only by the November sample of *I. setifera* (ISNOV), isolated from the December and October samples. This implies that the volatile concentrate composition of the species probably did not vary with the seasonal gradient. Humidity and temperature data did not vary along the gradient, while precipitation and insolation data do not justify the formation of this group. Thus, there is the possibility that another environmental factor could be related to the divergence in the composition of the volatile concentrate of the November sample of *I. setifera* in relation to the other months.

Group III corresponded to the IAAGO sample and was characterized by the presence of *n*-octadecanol. This sample formed a group distant from the samples of the following months, mainly the samples from the months of November and December. According to INMET data, August was the least rainy month of the seasonal gradient (4.93 mm) and November (10.57 mm) and December (11.13 mm) were the wettest. This result indicates that, possibly, there was a variation in the volatile compounds of *I. asarifolia* according to the seasonal gradient. However, there are no studies indicating the relationship between seasonality and the concentration of *n*-octadecanol in plant volatiles. However, there is a record of seasonal variation of this compound in the odor produced by certain birds (Soini et al. 2007).

Group IV corresponded to all samples of *I. asarifolia*, except IAAGO. The compounds with the highest weights for the formation of this group were the sesquiterpenes bicyclogermacrene, δ-elemene, δ-cadinene, ß-copaene, α-copaene, germacrene D and the alkanes *n*-heneicosan, *n*-tricosane and *n*-tetracosane.

There are no studies indicating a direct relationship between the production of these components with variations in precipitation or insolation, key factors in the studied seasonal gradient. However, several of these volatiles are related to the attraction of pollinators, which in turn are influenced by changes in rainfall (Lawson and Rands 2019; Mukherjee et al. 2019).

Based on these results, it was observed that *I. setifera* and *I. asarifolia* have different chemical profiles. The composition of the volatile concentrate of *I. asarifolia* may have been influenced by the seasonal gradient, while *I. setifera* did not show a clear relationship between the chemical composition of its volatile compounds and the variation in rainfall.

The variation in the volatile concentrate of *I. asarifolia*, more specifically the increase in the concentration of germacrene D, bicyclogermacrene, δ-elemene and α-copaene in the rainiest months, may be related to a strategy to attract a new pollinator guild that is more present in this period, as rainy season bees and flies.

3. Conclusions

In this study, variations in the composition of the volatile concentrate of both species of *Ipomoea* were observed. However, no relationship between the variation in the aromatic profile of *I. setifera* and the seasonal gradient was found. However, it was observed that the volatile concentrate of *I. asarifolia* possibly varied according to the seasonal gradient, with a greater divergence between the sample from the driest month (August) and the samples from the wettest months (November and December). Previous studies have shown that some chemical components identified in the floral aromas of *I. setifera* and *I. asarifolia* are related to pollination in other species. There is a possibility that germacrene D, bicyclogermacrene, δ-elemene and α-copaene have an ecological role, attracting a specific pollinator guild typical of the rainy season, with these constituents having higher concentrations in the volatile concentrate of *I. asarifolia* in the rainy months. Further studies are needed to confirm the dynamics of the relationship between the volatile concentrate of these species, the seasonal gradient and how these factors affect the plant-pollinator relationship.

Acknowledgements

The author Dr Mozaniel Santana de Oliveira, thanks PCI-MCTIC/MPEG, as well as CNPq for the scholarship process number: 300983/2022-0.

Disclosure statement

No potential conflict of interest was reported by the author.

Funding

The author(s) reported there is no funding associated with the work featured in this article.

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