CSIRO PUBLISHING

Australian Journal of Botany, 2018, **66**, 601–608
https://doi.org/10.1071/BT18138

Chemical constituents and allelopathic activity of the essential oil from leaves of *Eremanthus erythropappus*

Ana Paula Resende Pinto[A], *Janaína Brandão Seibert*[B], *Orlando David Henrique dos Santos*[C], *Sidney Augusto Vieira Filho*[C] *and Andréa Mendes do Nascimento*[D,E]

[A]Postgraduate Program in Chemistry, Federal University of Ouro Preto (UFOP), 35400-000 Ouro Preto, Brazil.
[B]Postgraduate Program in Biotechnology, Federal University of Ouro Preto (UFOP), 35400-000 Ouro Preto, Brazil.
[C]Department of Pharmacy, Federal University of Ouro Preto (UFOP), 35400-000 Ouro Preto, Brazil.
[D]Department of Chemistry, Federal University of Ouro Preto (UFOP), 35400-000 Ouro Preto, Brazil.
[E]Corresponding author. Email: andnascimen@yahoo.com.br

Abstract. The essential oil from leaves of *Eremanthus erythropappus* (DC.) MacLeish (Asteraceae) collected in South-eastern Brazil was extracted using a combination of water and steam distillation and investigated by chromatography/mass spectrometry (GC-MS). The sesquiterpene hydrocarbons were predominant (46.6%), followed by oxygenated sesquiterpenes (29.3%) and monoterpene hydrocarbons (18.8%). The major compounds were (β)-caryophyllene (29.3%), caryophyllene oxide (22.1%) and β-pinene (12.8%). The allelopathic activity of *E. erythropappus* essential oil on the seed germination and radicle length of seedlings was evaluated in lettuce (*Lactuca sativa* L.), tomato (*Lycopersicum esculentum* Mill.) and in two field weeds (field mustard – *Brassica rapa* L. and hairy beggarticks – *Bidens pilosa* L.). Among the weed species tested, the most significant inhibition of seed germination was observed in field mustard, as measured by the half–minimal inhibitory concentration, IC_{50} (IC_{50} = 26.5 μL mL^{-1}), and the most significant inhibitory effect on radicle length of seedlings was observed in seedlings of hairy beggarticks (IC_{50} = 16.3 μL mL^{-1}). In contrast, the lowest allelopathic effects of *E. erythropappus* essential oil on seed germination and radicle length of seedlings were observed in tomato (IC_{50} = >200 μL mL^{-1} and 130.1 μL mL^{-1} respectively) and lettuce (IC_{50} = 97.1 μL mL^{-1} and 35.1 μL mL^{-1} respectively). These results revealed significant allelopathic potential of *E. erythropappus* essential oil against weeds, but minimal effect on lettuce and tomato germination, and thus suggests it is feasible to use *E. erythropappus* sustainability as an eco-friendly bioherbicide in cropping settings.

Additional keywords: Asteraceae, chemical response, tropical weed ecology.

Received 6 July 2018, accepted 20 November 2018, published online 18 December 2018

Introduction

Plant performance is determined by several interacting factors such as plant genotype and environmental challenges, including general biotic and abiotic stress, herbivory and competition (Cahill and Lamb 2007). Plants compete for light, water and nutrients, revealing a continuous competition between the species living in a community; such competitive ability contributes to their survival in the ecosystem (Marco *et al.* 2012). Some plants develop defence mechanisms, producing specific secondary metabolites that, when released into the environment, interfere negatively on some stage(s) of the lifecycle of other plant species, such as seed germination and seedling growth (Alves *et al.* 2004). Allelopathy is the term used to designate the process in which plants suppress neighbours by exuding phytotoxins into their proximate environment (Worthington and Reberg-Horton 2013). The discovery of allelopathy as a viable component of crop–weed interference prompted the idea of exploiting this phenomenon to control weeds (Belz 2007). Substances belonging to different categories of secondary metabolites, also known as natural products, mediate allelopathic effects (Alves *et al.* 2002). Natural products released from plants may help by reducing the use of synthetic herbicides in weed control, causing less pollution and yielding safer agricultural products, as well as decreasing human health concerns (Khan *et al.* 2008).

Essential oils are complex mixtures of volatile secondary metabolites that may contain over 300 compounds and can be isolated from different parts of plants (Vagionas *et al.* 2007; Dhifi *et al.* 2016). Essential oils have numerous therapeutic properties and many of them are used in perfumery, as well as flavourings in the food and beverage industry (Vagionas *et al.* 2007). According to some authors, there are almost 3000 plants with potential to produce essential oils, 300 of which are commercially exploited (Lima *et al.* 2013). The main constituents of essential oils are terpenoids and phenylpropanoids (Astani *et al*. 2010). Terpenoids are distinguished by having biological activities, such as allelopathy, that are crucial for the survival and adaptation of a plant to the environment (Silva *et al*. 2009).

Asteraceae is the largest angiosperm family, with ~24000 species distributed among 1600 to 1700 genera (Funk *et al*. 2009). *Eremanthus erythropappus* (DC.) MacLeish, a tree native to Brazil popularly known as Candeia, is one of the plants belonging to this family and is used as fence posts due to its natural high durability (Scolforo *et al*. 2016). This tree has drawn the attention of the essential oil industry because its stem produces essential oil rich in α-bisabolol (>50%), a nontoxic sesquiterpene alcohol that exhibits anti-inflammatory properties used in cosmetic and pharmaceutical products (Lima *et al*. 2013; Silvério *et al*. 2013). According to Sousa *et al*. (2008), the main compounds found in the essential oil from leaves of *E. erythropappus* were β-caryophyllene, β-pinene, β-myrcene and germacrene-D, but no α-bisabolol, the major constituent in the stem. Another study on chemical composition of essential oil from leaves of *E. erythropappus* found β-caryophyllene, germacrene D, α-copaene, β-pinene and δ-cadinene as major compounds; however, this essential oil also contained α-bisabolol 2.1% (Silvério *et al*. 2013). *E. erythropappus* has been used in folk medicine as an anti-inflammatory and antimicrobial agent (Souza *et al*. 2003).

Studies have documented the allelopathic effect of essential oils from several medicinal plants on seed germination and seedling growth in weed species, as demonstrated in *Rosmarinus officinalis* L. (rosemary), *Cuminum cyminum* L. (cumin) and *Eucalyptus globulus* Labill. (eucalyptus); and the inhibitory effect on seed germination and root growth of seedlings in the weed species *Silybum marianum* (L.) Gaertn. (milk thistle), *Cynodon dactylon* L. (Bermuda grass) and *Amaranthus blitoides* S. Watson (mat amaranth) (Saharkhiz *et al*. 2009; Rassaeifar *et al*. 2013; Saad and Abdelgaleil 2014).

Bidens pilosa L., is a weed species commonly distributed throughout the world, whose control is difficult because it presents a short annual cycle and many generations during the year (Vidal and Merotto 2001). Resistance to herbicides has become an important issue in crop production worldwide over the last two decades. In Brazil, only 36 specific cases have been reported so far in weed species, displaying resistance to at least one herbicide's mechanism of action, with *B. pilosa* being the most widespread among them (Takano *et al*. 2016). Also, the *Brassica* genus has approximately one hundred species, many of which are important weeds in many growing regions (Kahrizi 2014). Wild populations of *Brassica rapa* L. generated concern mainly in agricultural fields because their combined relative abundance has increased over the past few decades (Gulden *et al*. 2008). *B. rapa* has been reported as a serious weed capable of causing large reductions in yields in the rapeseed culture due to competition for light, nutrients and water, and also regarded as a major problem in other cereals and vegetables (Kahrizi 2014). Therefore, allelopathy represents an alternative tool that can be used to assist in the conventional methods for the control of *B. pilosa* and *B. rapa*.

Thus, in this study we aimed to analyse the chemical composition of *E. erythropappus* essential oil and to investigate its allelopathic activity on seed germination and radicle length of seedlings in lettuce (*Lactuca sativa*), tomato (*Lycopersicum esculentum*), field mustard weed (*Brassica rapa*) and hairy beggarticks (*Bidens pilosa*).

Materials and methods

Plant material and essential oil extraction

Fresh *Eremanthus erythropappus* leaves were collected in the municipality of Ouro Preto, State of Minas Gerais (March 2016). A voucher specimen was deposited in the Herbarium José Badini, Federal University of Ouro Preto-UFOP, under voucher OUPR 28379.

The *E. erythropappus* essential oil was extracted from fresh leaves (1.31 kg) by a combination of water and steam distillation using a pilot distiller (Linax, model D2, SP, Votuporanga, Brazil) for 4 h. The extracted essential oil was separated from the hydrolyte by liquid-liquid partitioning, transferred to amber glass vials using a Pasteur pipette, and stored at 4°C. Oil yield was calculated (w/w) on the basis of the total amount of fresh plant material. The extracted essential oil was used in the analysis of its composition by chromatography and in the investigation of its allelopathic activity.

Essential oils analysis

Analysis was carried out on a Shimadzu model QP-2010 gas chromatograph coupled to a mass spectrometer (GC-MS). The following conditions were used: ZB-5MS column Phenomenex Zebron (30 m × 0.25 mm × 0.25 μm); helium (99.9%) as carrier gas at a constant flow (1.1 mL min^{-1}); 1μL injection volume; split mode ratio of 1:40; injector temperature 240°C; electron ionisation mode at 70eV; ion-source temperature 280°C. The oven temperature was set from 100°C (isothermal for 5 min) with an increase of 10°C min^{-1} to 250°C (isothermal for 5 min), and 10°C min^{-1} to 280°C (isothermal for 15 min).

Individual identification of the constituents was accomplished by comparison between their GC retention index, determined with a homologous series of normal C_9–C_{25} alkanes as reference; and by comparison of the fragmentation patterns obtained in the mass spectra with those from the software database (Wiley 7 lib and Nist 08 lib). The linear retention index (LRI) was calculated for each constituent, according to the following equation $LRI = 100n + 100 \times (t_x - t_n)/(t_{n+1} - t_n)$; where t_x is the retention time of the constituent x; t_n is the retention time of the *n*-alkane eluate before of the constituent x; t_{n+1} is the retention time of the *n*-alkane eluded after of the constituent x and n is the number of carbon atoms in the *n*-alkane eluate before of the constituent x, previously described (van Den Dool and Kratz 1963; Viegas and Bassoli 2007), and the data were compared with those from pertinent literature (Adams 2009).

Allelopathic activity

Allelopathy tests were conducted at the Laboratory of Pharmacognosy, School of Pharmacy, Federal University of Ouro Preto, Ouro Preto-MG, using seeds of lettuce (*L. sativa*) and tomato (*L. esculentum*), both acquired in a local market (both seeds under the brand ISLA Sementes, 99.7% purity). In addition, seeds of the following weeds were used: field mustard (*B. rapa*), collected in the botanical garden 'Jorge Luiz da Silva', UFOP, voucher OUPR 29075, and hairy beggarticks (*B. pilosa*), collected at the campus of UFOP, voucher OUPR 29076. All seeds were exposed to *E. erythropappus* essential oil.

The essential oil was dissolved in diethyl ether, in 1 : 2 (v/v) ratio, to make a concentrated stock solution (200 μL mL^{-1}). Solutions with lower concentrations (100–1.56 μL mL^{-1}) were prepared by diluting a more concentrated solution in diethyl ether. Three replicates of each concentration were prepared. Subsequently, these samples (1 mL) were placed into 6.0 cm sterile plastic Petri dishes and lined with filter paper. After solvent evaporation, the final volume was replaced by distilled water. The pure solvent used in the dilutions was also tested. The testing solutions were added only in the beginning of the bioassays, after which only distilled water was added when necessary. The positive control was an aqueous solution of $ZnCl_2$ 5% (used to inhibit seed germination and radicle length of seedlings) and the negative control was distilled water.

Seeds were sterilised with sodium hypochlorite (20% v/v) for 10 min, dried and placed into Petri dishes. Seeds were selected based on their integrity and the damaged ones were discarded. Twenty seeds of lettuce and 10 seeds of tomato, field mustard and hairy beggarticks were placed on filter paper into Petri dishes. The quantity of seeds added in Petri dishes was determined in accordance to their size. The seed germination trials were maintained at room temperature (20–25°C) during a period according to the germination time of each species: 5 days for lettuce (Seibert 2015), 7 days for field mustard (Angeletti and Fonseca 1989), 8 days for hairy beggarticks (Adegas *et al*. 2003) and 9 days for tomato (Seibert 2015). After this period, the average number of germinated seeds and their radicle length were evaluated. The percentage of variation in germination (VG) was calculated using the following equation: $VG = ((NS - NC)/NC) \times 100$; where NS is the number of seeds germinated in each sample and NC is the number of seeds germinated in the negative control, with distilled water only. The percentage of variation of radicle length of seedlings (VRL) was calculated using the following equation: $VRL = ((LRS - LRC)/LRC) \times 100$; where LRS is the average length of the radicle (cm) in each sample and LRC is the average length of the radicle (cm) in the negative control. No germination occurred in the positive control.

The results were plotted in graphs with the values of the variables ± s.d., using the GraphPad Prism software, ver. 5.0. The half-minimal inhibitory concentration (IC_{50}) in inhibiting seed germination and radicle length of seedlings in each species was determined according to Dutka (1989), Greene *et al*. (1988) and Wang (1987).

Results

Chemical composition of the essential oil

The yield of the essential oil extracted from leaves of *E. erythropappus* was 0.12% (w/w). Forty-seven compounds were identified in the essential oil, representing 96.0% of its chemical composition (Table 1). This oil was characterised by a high amount of sesquiterpene hydrocarbons (46.6%), followed by oxygenated sesquiterpenes (29.3%), monoterpene hydrocarbons (18.8%), oxygenated monoterpenes (1.0%) and other compounds (0.3%). The major components were β-caryophyllene, caryophyllene oxide, and β-pinene, which

Table 1. Chemical composition of the essential oil from fresh leaves of *Eremanthus erythropappus*

Bold text indicates the most representative components (>5%)

LRI[A]	LRI (lit.)[B]	Compound	Yield %
926	930	α-Thujene	0.02
934	939	α-Pinene	2.16
974	975	Sabinene	0.58
978	979	β-Pinene	**12.83**
990	990	Myrcene	2.82
1006	1005	3*Z*-Hexenyl acetate	0.10
1023	1024	*p*-Cymene	0.15
1027	1029	Limonene	0.38
1096	1096	Linalool	0.07
1099	1100	*n*-Nonanal	0.03
1139	1139	*trans*-Pinocarveol	0.24
1164	1164	Pinocarvone	0.19
1176	1177	Terpinen-4-ol	0.09
1194	1195	Myrtenal	0.29
1325	1327	*p*-Mentha-1,4-dien-7-ol	0.07
1351	1351	α-Cubebene	0.10
1376	1376	α-Copaene	3.69
1386	1388	β-Bourbonene	0.56
1390	1390	β-Elemene	1.97
1408	1409	α-Gurjunene	0.19
1420	1419	β-Caryophyllene	**29.30**
1431	1432	β-Copaene	0.28
1437	1434	α-*trans*-Bergamotene	0.11
1441	1439	α-Guaiene	0.06
1454	1454	α-Humulene	2.62
1466	1466	*cis*-Muurola-4(14),5-diene	0.10
1479	1479	γ-Muurolene	0.60
1480	1481	Germacrene D	1.65
1488	1489	*cis*-Eudesma-6,11-diene	0.76
1497	1496	Valencene	0.47
1499	1500	Bicyclogermacrene	0.24
1499	1500	α-Muurolene	0.52
1508	1509	α-Bulnesene	0.39
1512	1513	γ-Cadinene	1.69
1526	1523	δ-Cadinene	1.13
1540	1538	α-Cadinene	0.18
1576	1578	Spathulenol	2.94
1581	1583	Caryophyllene oxide	**22.13**
1595	1592	Viridiflorol	0.47
1603	1602	Ledol	0.47
1606	1608	Humulene epoxide II	0.91
1632	1628	1-epi-Cubenol	0.19
1640	1640	Caryophylla-4(12),8(13)-dien-5α-ol	0.18
1644	1646	α-Muurolol	1.28
1654	1648	Agarospirol	0.19
1658	1654	α-Cadinol	0.52
2499	2500	*n*-Pentacosane	0.06
		Monoterpenes	
		Hydrocarbons	18.79
		Oxygenated	0.95
		Monoterpenes total	**19.74**
		Sesquiterpenes	
		Hydrocarbons	46.61
		Oxygenated	29.28
		Sesquiterpenes total	**75.89**
		Others	0.34
		Total identified	**95.97**

[A]Linear retention indices on ZB-5MS column (relative to *n*-alkanes).
[B]Linear retention indices. See Adams (2009).

represented 29.3, 22.1 and 12.8% of the essential oil composition respectively.

Allelopathic activity

The allelopathic activity of *E. erythropappus* essential oil on seeds development in lettuce, tomato, field mustard and hairy beggarticks is shown in Figs 1–4 and Table 2.

The obtained results of *E. erythropappus*' essential oil effect on the germination and seedling establishment of lettuce, tomato and two weeds (field mustard, *B. rapa* and hairy beggarticks, *B. pilosa*) indicated that the germination and growth of these four plants decreased significantly under the effect of essential oil.

The essential oil IC_{50} values that inhibited seed germination in lettuce, tomato, field mustard and hairy beggarticks

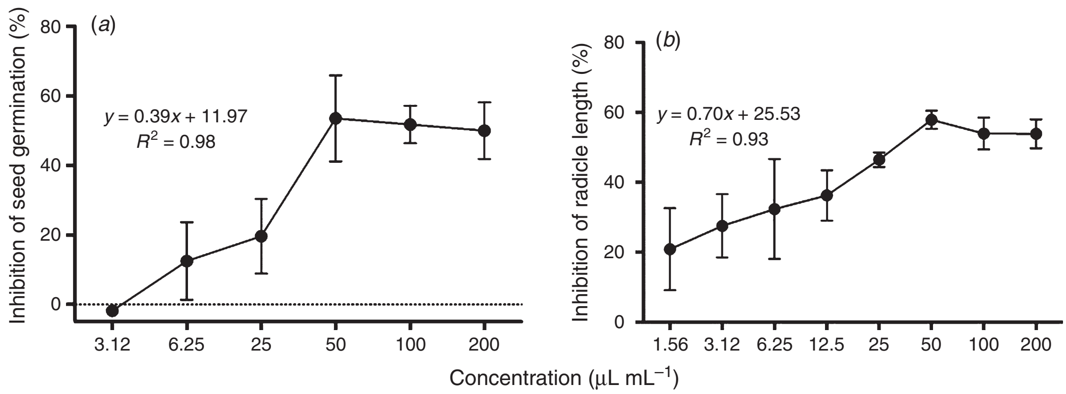

Fig. 1. Percentage inhibition of seed germination (*a*) and radicle length of seedlings (*b*) in lettuce – *Lactuca sativa* using different concentrations of *Eremanthus erythropappus* essential oil. Values are mean ± l.s.d.

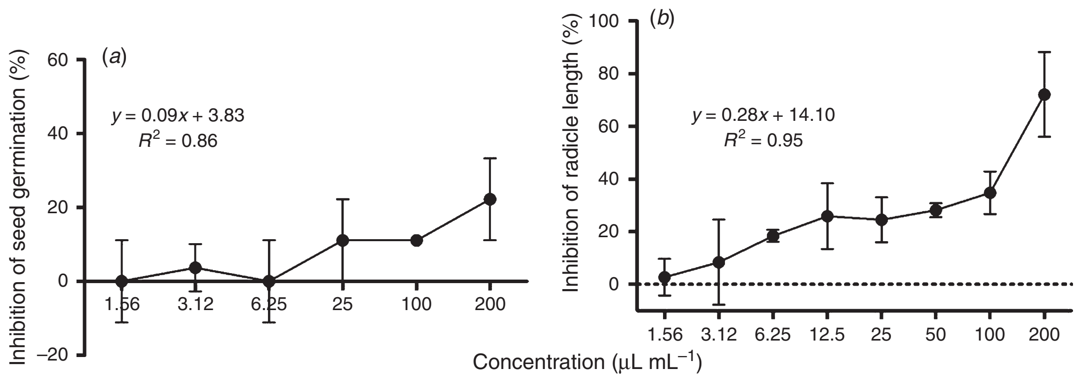

Fig. 2. Percentage inhibition of seed germination (*a*) and radicle length of seedlings (*b*) in tomato – *Lycoperiscum esculentum* using different concentrations of *Eremanthus erythropappus* essential oil. Values are mean ± l.s.d.

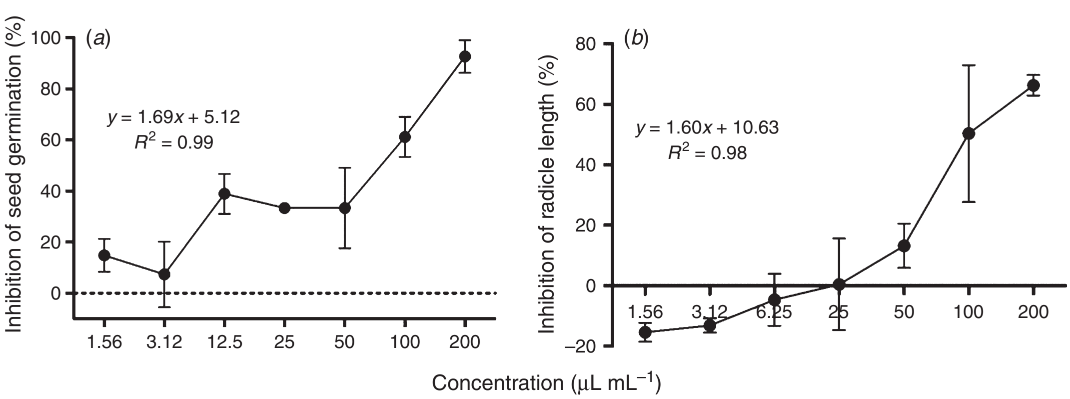

Fig. 3. Percentage inhibition of seed germination (*a*) and radicle length of seedlings (*b*) in field mustard – *Brassica rapa* using different concentrations of *Eremanthus erythropappus* essential oil. Values are mean ± l.s.d.

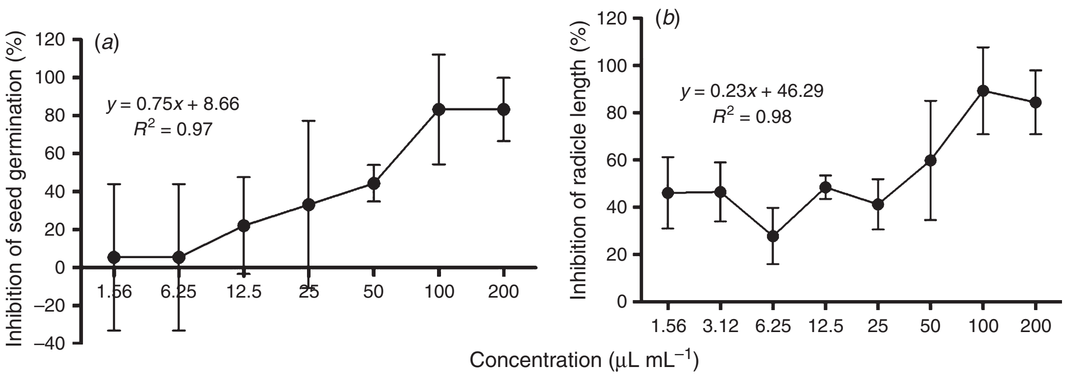

Fig. 4. Percentage inhibition of seed germination (*a*) and radicle length of seedlings (*b*) in hairy beggartick – *Bidens pilosa* using different concentrations of *Eremanthus erythropappus* essential oil. Values are mean ± l.s.d.

Table 2. Effect of *Eremanthus erythropappus* essential oil on seed germination and radicle length of seedlings in lettuce, tomato, filed mustard and hairy beggarticks, as measured by the half-minimal inhibitory concentration (IC_{50})

Seeds	IC_{50} (µL mL^{-1})[A]	
	Seed germination	Radicle length of seedlings
Lettuce-*Lactuca sativa*	97.1	35.1
Tomato-*Lycopersicum esculentum*	>200	130.1
Field mustard-*Brassica rapa*	26.5	24.5
Hairy beggarticks-*Bidens pilosa*	54.9	16.3

[A]IC_{50} values were obtained from Figs 1–4.

(Figs 1*a*, 4*a*) were 97.1, >200, 26.5 and 54.9 µL mL^{-1}, respectively (Table 2). The IC_{50} values for inhibition of radicle length of seedlings of lettuce, tomato, field mustard and hairy beggarticks (Figs 1*b*, 4*b*) were 35.1, 130.1, 24.5 and 16.3 µL mL^{-1} respectively (Table 2).

Discussion

The amount of essential oil obtained was smaller than that described in the literature for the same plant species, from fresh leaves and using a Clevenger apparatus for 2 h, i.e. 0.25% (Sousa *et al.* 2008) and 0.20% (Silvério *et al.* 2013). In the present study was used a pilot distiller for 4 h.

Essential oil was practically constituted by sesquiterpenes (75.9%), and among these, β-caryophyllene was the major compound. Silvério *et al.* (2013) conducted studies on the characterisation of the essential oil from fresh leaves of *E. erythropappus* and reported 25 components, 68% of which were equivalent to those described in the present study. Sesquiterpene hydrocarbons and oxygenated sesquiterpenes were also abundant components of the *E. erythropappus* essential oil. Sousa *et al.* (2008) identified 30 compounds for the essential oil extracted from mature leaves of *E. erythropappus*, whose main components were β-caryophyllene, β-pinene, β-myrcene and germacrene D, showing 70% similarity with the chemical composition of the essential oil analysed in this study.

The difference found in the composition of these essential oils has probably occurred as a result of several factors, such as genetic factors, growing location, regional climate and time of day at which the plant material was collected (Burt 2004).

In general, increasing the essential oil concentration significantly increased the percentage inhibition of seed germination in all species (Figs 1*a*, 4*a*). However, the percentage inhibition of seed germination was lower in lettuce and tomato when compared with field mustard and hairy beggarticks (Table 2), indicating that lettuce and tomato were less sensitive to the effects of *E. erythropappus* essential oil. As weeds were more sensitive to it, its allelopathic effects can be extremely beneficial for economic reasons, since they can ensure a longer-term survival of plants of agricultural interest against weedy plants. The variation in the germination behaviour is probably related to the physiological processes of the target species, which respond differently to allelochemicals (Rizzi *et al.* 2016). In the seed germination trials, the lettuce seeds were more affected by the phytotoxic action of this essential oil than tomato seeds. It was not possible to determine the exact IC_{50} value for tomato seed germination through the data from the Table 2 and Fig. 2*a*, because this value exceeded the highest concentration tested (200 µL mL^{-1}). As regards the weeds tested in this study, field mustard showed the lowest IC_{50} value for seed germination (26.5 µL mL^{-1}). Hillen *et al.* (2012) reported that *E. erythropappus* essential oil also caused inhibition of seed germination in maize, soybean and bean; however, unlike the present study, the essential oil was extracted from stem and not from the leaves.

Increasing the essential oil concentration also increased the inhibitory effect on radicle growth, except in field mustard, which exhibited stimulatory effects at low concentrations, i.e. concentrations below 25 µL mL^{-1} (Fig. 3*b*). Bioassays for allelopathic activity often show increased radicle elongation in newly germinated seeds when allelochemicals are present at low concentrations (Gliessman 2014). Inhibition of radicle growth was lower in lettuce and tomato than in the two weeds tested. Accordingly, the highest inhibition of radicle growth was observed in seedlings of the weed hairy beggarticks, with IC_{50} value equal to 16.3 µL mL^{-1} (Table 2). In general, the inhibitory effect of *E. erythropappus* essential oil was stronger on radicle growth than on seed germination. Root necrosis is a common effect associated with allelopathy (Carvalho *et al.* 2015). In an ecological context, the inhibition of seedling growth is a more efficient mechanism than the prevention of seed germination (Jacobi and Ferreira 1991).

The results of several studies have shown a correlation between the chemical composition of essential oils and their effects on seed germination and seedling growth. In general, a potent phytotoxic was associated with a higher amount of oxygenated monoterpenes (Dhifi *et al*. 2016). The properties of monoterpenes in affecting germination and growth of plants can be explained by the ability of these compounds to cause morphological and physiological changes, such as inhibition of the respiratory chain in isolated mitochondria and mitosis, alteration of integrity of cell membrane, deterioration of cuticular waxes, increased perspiration, and lipid peroxidative damage to microtubes (Yoshimura *et al*. 2011). Batish *et al*. (2004) mentioned that the decrease in respiratory activities caused by monoterpenes reduces photosynthesis, disturbing both growth and germination.

In the present study *E. erythropappus* essential oil did not present a high percentage of oxygenated monoterpenes (1.0%) and the allelopathic activity of this essential oil on seed germination and seedling growth could not be assigned only to this class of compounds, as described in the literature (Vokou *et al*. 2003; De Almeida *et al*. 2010; Dhifi *et al*. 2016).

In contrast, the results on the inhibition of seed germination have been assigned to several monoterpenes, oxygenated and hydrocarbons (e.g. linalool, camphor, pinenes, cineoles, carvacrol and thymol) in their pure forms or mixtures (Saharkhiz *et al*. 2009). The essential oil extracted and analysed in this work had a high percentage of pinene (α-pinene 2.2% and β-pinene 12.8%). El Ayeb-Zakhama *et al*. (2016) demonstrated, in turn, that the oil extracted from the root of *Tipuana tipu*, rich in sesquiterpene hydrocarbons, mainly represented by β-caryophyllene (24.1%), germacrene D (20.0%) and α-humulene (9.1%), showed a very potent inhibitory effect on the germination of lettuce seeds at a dose of 1 mg mL^{-1}, compared with the oil extracted from other parts of this plant. Furthermore, β-caryophyllene has been reported to inhibit the development of seedlings in various plant species. This is consistent with the findings of the present study, since β-caryophyllene was the major constituent (29.3%) among the chemical compounds found here. Mabrouk *et al*. (2013) reported that the essential oil isolated from aerial parts of *Conyza sumatrensis* significantly delayed seed germination and seedlings growth in radish, with these effects being attributed to the main compounds caryophyllene oxide, spathulenol and limonene. Here, a significant amount of caryophyllene oxide (22.1%) was detected in the *E. erythropappus* essential oil. Thus, pinenes, β-caryophyllene and caryophyllene oxide are likely responsible for at least a considerable part of *E. erythropappus* oil phytotoxicity. Furthermore, allelopathic inhibition typically results from the joint action of a group of allelochemicals, and the toxicity effects may be enhanced due to a synergistic interaction, rather than being caused by a particular compound (Saharkhiz *et al*. 2010).

Conclusions

In total, 47 compounds were identified in the essential oil extracted from leaves of *E. erythropappus*, with sesquiterpenoids as main constituents (75.9%). This essential oil showed allelopathic inhibitory potential on seed germination and radicle length of seedlings in two weed species, field mustard and hairy beggarticks, without considerably affecting lettuce and tomato. The most marked effect on inhibition of seed germination was observed in field mustard, with an IC_{50} value equal to 26.5 µL mL^{-1}, whereas the most marked effect on inhibition of radicle length of seedlings was observed in hairy beggarticks, with an IC_{50} value equal to 16.3. Comparing tomato and lettuce, tomato was the less affected by effect of the essential oil on seed germination and radicle length of seedlings. Hence, the essential oil extracted from leaves of *E. erythropappus* may represent a good biodegradable and a green herbicide alternative, although this hypothesis needs further studies.

Conflicts of interest

The authors declare no conflicts of interest.

Acknowledgements

Authors are grateful to FAPEMIG (Grant number: CEX-APQ-00537-11) and Federal University of Ouro Preto-UFOP for their financial support.

References

Adams RP (2009) ‘Identification of essential oil components by gas chromatography/mass spectrometry.’ (Allured Books: Carol Stream, IL, USA)

Adegas FS, Voll E, Prete CEC (2003) Imbibition and germination of hairy beggartick seeds (*Bidens pilosa*). *Planta Daninha* **21**, 21–25. doi:10.1590/S0100-83582003000100003

Alves SM, Arruda MSP, Souza Filho APS (2002) Biossíntese e distribuição de substâncias alelopáticas. In ‘Alelopatia: princípios básicos e aspectos gerais’. (Eds AP Souza Filho, SM Alves) pp. 70–102. (Embrapa Amazônia Oriental: Belém, Brazil)

Alves MCS, Medeiros Filho S, Innecco R, Torres SB (2004) Allelopathy of plant volatile extracts on seed germination and radicle length of lettuce. *Pesquisa Agropecuária Brasileira* **39**, 1083–1086. doi:10.1590/S0100-204X2004001100005

Angeletti MP, Fonseca AFA (1989) Instruções técnicas para o cultivo comercial de hortaliças em Rondônia. In ‘Circular Técnica 11’. p. 7. (Embrapa-UEPAE: Porto Velho, Brazil)

Astani A, Reichling J, Schnitzler P (2010) Comparative study on the antiviral activity of selected monoterpenes derived from essential oils. *Phytotherapy Research* **24**, 673–679.

Batish DR, Setia N, Singh HP, Kohli RK (2004) Phytotoxicity of lemon-scented eucalypt oil and its potential use as a bioherbicide. *Crop Protection* **23**, 1209–1214. doi:10.1016/j.cropro.2004.05.009

Belz RG (2007) Allelopathy in crop–weed interactions-an update. *Pest Management Science* **63**, 308–326. doi:10.1002/ps.1320

Burt S (2004) Essential oils: their antibacterial properties and potential applications in foods-a review. *International Journal of Food Microbiology* **94**, 223–253. doi:10.1016/j.ijfoodmicro.2004.03.022

Cahill JF, Lamb EG (2007) Interactions between root and shoot competition and plant traits. *HortScience* **42**, 1110–1112.

Carvalho FP, Melo CAD, Machado MS, Dias DCFS, Alvarenga EM (2015) The allelopathic effect of *Eucalyptus* leaf extract on grass forage seed. *Planta Daninha* **33**, 193–201. doi:10.1590/0100-83582015000200004

De Almeida LF, Frei F, Mancini E, De Martino L, De Feo V (2010) Phytotoxic activities of Mediterranean essential oils. *Molecules* **15**, 4309–4323. doi:10.3390/molecules15064309

Dhifi W, Bellili S, Jazi S, Bahloul N, Mnif W (2016) Essential oil chemical characterization and investigation of some biological activities: a critical review. *Medicines* **3**, 25. doi:10.3390/medicines3040025

Dutka B (1989) 'Short-term root elongation toxicity bioassay. Methods for toxicological analysis of waters, wastewaters and sediments.' (National Water Research Institute, Environment Canada: Toronto, Canada)

El Ayeb-Zakhama AE, Sakka-Rouis L, Bergaoui A, Flamini G, Jannet HB, Harzallah-Skhiri F (2016) Chemical composition and allelopathic potential of essential oils from *Tipuana tipu* (Benth.) Kuntze cultivated in Tunisia. *Chemistry & Biodiversity* **13**, 309–318. doi:10.1002/cbdv.201500083

Funk VA, Susanna A, Stuessy TF, Bayer RJ (2009) 'Systematics, evolution, and biogeography of the Compositae.' (Eds VA Funk, A Susanna, TF Stuessy, RJ Bayer) p. 171. (International Association for Plant Taxonomy: Vienna)

Gliessman SR (2014) 'Agroecology: the ecology of sustainable food systems.' (CRC Press, Taylor & Francis: New York)

Greene JC, Bartels CL, Warren-Hicks WJ, Parkhurst BR, Linder GL, Peterson SA, Miller WE (1988) 'Protocols for short term toxicity screening of hazardous waste sites.' EPA/600/3-88/029. (Environmental Research Laboratory: Corvallis, OR, USA)

Gulden RH, Warwick SI, Thomas AG (2008) The biology of Canadian weeds. 137. *Brassica napus* L. and *B. rapa. Canadian Journal of Plant Science* **88**, 951–996. doi:10.4141/CJPS07203

Hillen T, Schwan-Estrada KRF, Mesquini RM, Cruz MES, Stangarlin JR, Nozaki M (2012) Antimicrobial activity of essential oils on the *in vitro* control of some fungal phytopathogens and on seed treatment. *Revista Brasileira de Plantas Medicinais* **14**, 439–445. doi:10.1590/S1516-05722012000300003

Jacobi US, Ferreira AG (1991) Efeitos alelopáticos de *Mimosa bimucronata* (DC) sobre espécies cultivadas. *Pesquisa Agropecuária Brasileira* **26**, 935–943.

Kahrizi D (2014) Reduction of EPSP synthase in transgenic wild turnip (*Brassica rapa*) weed via suppression of *aro*A. *Molecular Biology Reports* **41**, 8177–8184. doi:10.1007/s11033-014-3718-0

Khan ZR, Amudavi DM, Midega CAO, Wanyama JM, Pickett JA (2008) Farmers' perceptions of a 'push-pull' technology for control of cereal stemborers and *Striga* weed in western Kenya. *Crop Protection* **27**, 976–987. doi:10.1016/j.cropro.2007.12.001

Lima FWJ, Dantas-Barros AM, Soares BM, Santos DA, Resende MA, Carvalho MAR, Siqueira EP, Nelson DL (2013) The composition and anti-microbial activity of the essential oil from *Eremanthus erythropappus* (DC) Macleish (Candeia). *International Journal of Medicinal and Aromatic Plants* **3**, 1–10.

Mabrouk S, Salah KBH, Elaissi A, Jlaiel L, Jannet HB, Aouni M, Harzallah-Skhiri F (2013) Chemical composition and antimicrobial and allelopathic activity of Tunisian *Conyza sumatrensis* (Retz.) E.Walker essential oils. *Chemistry & Biodiversity* **10**, 209–223. doi:10.1002/cbdv.201200117

Marco CA, Teixeira E, Simplício A, Oliveira C, Costa J, Feitosa J (2012) Chemical composition and allelopathyc activity of essential oil of *Lippia sidoides* Cham. *Chilean Journal of Agricultural Research* **72**, 157–160. doi:10.4067/S0718-58392012000100025

Rassaeifar M, Hosseini N, Asl NHH, Zandi P, Aghdam AM (2013) Allelopathic effect of *Eucalyptus globulus*' essential oil on seed germination and seedling establishment of *Amaranthus blitoides* and *Cyndon dactylon. Trakia Journal of Sciences* **11**, 73–81.

Rizzi ES, Pereira KCL, Abreu CAA, Lima Silva BCF, Fernandes RM, Oliveira AKM, Matias R (2016) Allelopathic potential and phytochemistry of cambarazinho (*Vochysia haenkeana* (Spreng.) Mart.) leaves in the germination and development of lettuce and tomato. *Bioscience Journal* **32**, 98–107. doi:10.14393/BJ-v32n1a2016-29614

Saad MMG, Abdelgaleil SAM (2014) Allelopathic potential of essential oils isolated from aromatic plants on *Silybum marianum* L. *Global Advanced Research Journal of Agricultural Science* **3**, 289–297.

Saharkhiz MJ, Ashiri F, Salehi MR, Ghaemghami J, Mohammadi S (2009) Allelopathic potential of essential oils from *Carum copticum* L., *Cuminum cyminum* L., *Rosmarinus officinalis* L. and *Zataria multiflora* Boiss. *Medicinal and Aromatic Plant Science and Biotechnology* **31**, 32–35.

Saharkhiz MJ, Smaeili S, Merikhi M (2010) Essential oil analysis and phytotoxic activity of two ecotypes of *Zataria multiflora* Boiss. growing in Iran. *Natural Product Research* **24**, 1598–1609. doi:10.1080/14786411003754280

Scolforo JRS, Altoe TF, Scolforo HF, De Mello JM, Castro e Silva CP, Ferraz Filho AC (2016) Management strategies of *Eremanthus erythropappus* (DC.) MacLeish under different initial spacing. *Ciência e Agrotecnologia* **40**, 298–304. doi:10.1590/1413-70542016403042715

Seibert JB (2015) Desenvolvimento de nanoemulsão a partir do óleo essencial de folhas de *Cymbopogon densiflorus* (Steud.) Stapf.: avaliação da sazonalidade e atividades biológicas. MSc thesis, Graduate program in biotechnology, Federal University of Ouro Preto. Available at: www.repositorio.ufop.br/handle/123456789/6502 (accessed 23 November 2018).

Silva CB, Simionatto E, Hess SC, Peres MTLP, Simionatto EL, Junior AW, Poppi NR, Faccenda O, Cândido ACS, Scalon SPQ (2009) Chemical composition and allelopathic activity of essential oil from *Hydrocotyle bonariensis* Lam (Araliaceae). *Quimica Nova* **32**, 2373–2376.

Silvério MS, Del-Vechio-Vieira G, Pinto MAO, Alves MS, Sousa OV (2013) Chemical composition and biological activities of essential oils of *Eremanthus erythropappus* (DC) McLeisch (Asteraceae). *Molecules* **18**, 9785–9796. doi:10.3390/molecules18089785

Sousa OV, Dutra RC, Yamamoto CH, Pimenta DS (2008) Chemical composition and biological activity of the essential oils' comparative study from *Eremanthus erythropappus* (DC) McLeisch leaves. *Brazilian Journal of Pharmacy* **89**, 113–116.

Souza OV, Oliveira MS, Rabello SV, Cunha RO, Costa BLS, Leite MN (2003) Estudo farmacognóstico de galhos de *Vanillosmopsis erythropappa* Schult. Bip. – Asteraceae. *Revista Brasileira de Farmacognosia* **13**, 50–53. doi:10.1590/S0102-695X2003000300019

Takano HK, Oliveira RS Jr, Constantin J, Braz GBP, Franchini LHM, Burgos NR (2016) Multiple resistance to atrazine and imazethapyr in hairy beggarticks (*Bidens pilosa*). *Ciência e Agrotecnologia* **40**, 547–554. doi:10.1590/1413-70542016405022316

Vagionas K, Ngassapa O, Runyoro D, Graikou K, Gortzi O, Chinou I (2007) Chemical analysis of edible aromatic plants growing in Tanzania. *Food Chemistry* **105**, 1711–1717. doi:10.1016/j.foodchem.2007.05.029

van Den Dool H, Kratz PD (1963) A generalization of the retention index system including linear temperature programmed gas-liquid partition chromatography. *Journal of Chromatography. A* **11**, 463–471. doi:10.1016/S0021-9673(01)80947-X

Vidal RA, Merotto A, Jr (2001) Herbicidas inibidores da PROTOX. In 'Herbicidologia'. (Eds RA Vidal, A Merotto Jr) p. 152. (Evangraf: Porto Alegre, Brazil)

Viegas MC, Bassoli DG (2007) Linear retention index for characterization of volatile compounds in soluble coffee using GC-MS and HP-Innowax column. *Quimica Nova* **30**, 2031–2034. doi:10.1590/S0100-40422007000800040

Vokou D, Douvil P, Blionis GJ, Halley JM (2003) Effects of monoterpenoids, acting alone or in pairs, on seed germination and subsequent seedling growth. *Journal of Chemical Ecology* **29**, 2281–2301. doi:10.1023/A:1026274430898

Wang W (1987) Root elongation method for toxicity testing of organic and inorganic pollutants. *Environmental Toxicology and Chemistry* **6**, 409–414. doi:10.1002/etc.5620060509

Worthington M, Reberg-Horton C (2013) Breeding cereal crops for enhanced weed suppression: optimizing allelopathy and competitive ability. *Journal of Chemical Ecology* **39**, 213–231. doi:10.1007/s10886-013-0247-6

Yoshimura H, Sawai Y, Tamotsu S, Sakai A (2011) 1,8-Cineole inhibits both proliferation and elongation of BY-2 cultured tobacco cells. *Journal of Chemical Ecology* **37**, 320–328. doi:10.1007/s10886-011-9919-2

Handling Editor: Olusegun Osunkoya