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Full Length Article

Comparative GC-MS Profile for Bioactive Compounds of Foliar Surface Extracts of *Senecio biafrae* and *Crassocephalum crepidioides*

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Abstract

This study was carried out to assess the GC-MS profiles of foliar surface extracts of *Senecio biafra*e and *Crassocephalum crepidioides* to provide information on the bioactive compounds present to establish these vegetables as a potential source of plant-based drugs. Dewaxed leaf surface extracts of the two vegetables were obtained by soaking the fresh leaves in chloroform for a period of 3 days. The compounds in the foliar surface extracts of the two vegetables were analyzed, identified and characterized using GC-MS. The GC-MS analysis showed that *C. crepidioides* contained eighteen compounds with ficusin being the most abundant with a peak area of 13.14%. Other abundant compounds included oleic acid (9.78%) and Z, Z-3,13-octadecadien-1-ol (7.34%). *S. biafrae* extract contained fourteen compounds with 3-buten 2-one, 4-(2,6,6-trimethyl-1-cyclohexen-1-yl) being the most abundant one with a peak area of 16.14%. Other abundant compounds included oleic acid and ficusin with a peak area of 14.26 and 11.24%, respectively. The findings in this research further established the beneficial and therapeutic use of these vegetables; also, the information provided could be exploited for future drug development.

Keywords: Bioactive compounds; *Crassocephalum crepidioides*; Foliar surface; GC-MS; *Senecio biafra*e

Introduction

Industrialization and urbanization have impacted public health significantly. There is an increase in food, air, and water pollution. Carbon emissions, electromagnetic waves, and exposure to radioactive compounds have also increased due to industrialization (Pachoulis *et al.* 2022). These have increased human susceptibility to diseases such as cancers, diabetes, immune dysfunction and other chronic diseases (Liu 2010). New diseases such as COVID-19 are also being discovered. Other phenomena such as natural drug resistance which could be as a result of genetic mutation or acquisition of resistance by one species from another are some of the factors necessitating the need for the development of more effective new drugs without adverse effects. Hence, there is a need for the identification of more plant-based natural products today as a result of increased drug resistance, viral mutation, and more complex medical conditions (Khan and Javaid 2021; Javaid *et al.* 2022). Recent studies have shown that plants are a rich source of bioactive compounds (Naqvi *et al.* 2020). The foliar surface is a very important part of every plant. It is the first stage of defence for plants against external attack. Bioactive compounds on the foliar surface act as defence mechanisms against insects, pathogens, and other herbivores. These compounds are referred to as allelochemicals (Hickman *et al.* 2021). For example, isopentylamine is a defensive compound recently identified by Aboshi *et al.* (2021) as a deterrent to insect herbivores in rice.

C. crepidioides and *S. biafrae* belong to the Asteraceae family, which is one of the largest plant families with about 24,000 species (Michel *et al.* 2020). It has been found to have a lot of medicinal species with therapeutic properties such as antimicrobial, anti-inflammatory, antioxidant, and antihypertensive properties (Ajani *et al.* 2022). *S. biafrae* also known as Bologi is a climbing herb that grows in secondary rainforest and is spread across the land via roadsides and waste areas in tropical Africa (Olasupo *et al.* 2017). In Nigeria, it is consumed as a vegetable and as a veritable component of soups and sauces. *C. crepidioides* is known in southwestern Nigeria locally as "Ebolo" is a popular food supplement in Nigeria. It is also known as fireweed

(Omotayo *et al*. 2015). The plant grows in large numbers undercover in tree crop plantations and is mostly underutilized. It is also largely consumed as soup in southwestern Nigeria. The study of the foliar surface extract of these vegetables has not been well-established, hence the scarcity of information on their suitability as nutraceuticals and pharmaceuticals. Therefore, this study is aimed at providing information on the comparative advantages of the foliar surfaces of these vegetables in addition to identifying the bioactive compounds present. The identified compounds could be of significant nutraceutical properties. This study, therefore, was aimed at profiling the foliar surface extracts of two indigenous vegetables *S. biafrae* and *C. crepidioides* using gas chromatography-mass spectrometry (GC-MS).

Materials and Methods

Plant material

Fresh leaves of *S. biafrae* and *C. crepidioides* were sourced from a farm in Ogbese, Ondo State, Nigeria, around March 2021. Ogbese lies between longitudes 5.3704° East of the Greenwich Meridian, and latitudes 7.2579° North of the Equator. The vegetables were authenticated by the Department of Plant Science and Biotechnology, Achievers University, Owo, Ondo State, Nigeria.

Plant preparation and extraction

Fresh leaves from plants that had not yet flowered were immersed in chloroform (5 mL/g) for three minutes. The chloroform ($CHCl_3$) was decanted, evaporated and the residual material was gathered in a little amount of acetone and allowed to cool overnight. The extracted plant waxes were filtered out of the acetone solution and evaporated, the crude extract of both vegetables 4.00 g and 6.07 g of *Crassocephalum crepidioides* and *Senecio biafrae* respectively were obtained and subjected to GC-MS analysis.

GC-MS analyses

The samples were subjected to chromatographic analysis using a Varian 3800/4000 gas chromatograph-mass spectrometer equipped with an Agilent equipped with a BP5 (30 m × 0.25 mm × 0.25 μm) capillary column. The carrier gas used was nitrogen with rate flow of 1 mL min^{-1}. The injection volume employed was 2 μL with split ratio of 10:1 at a temperature of 250°C. An electron ionization device with an ionizing energy of 70 eV was employed for GC-MS detection. The temperature of the oven was set at 110°C for 2 min, which gradually increased at the rate of 10°C min^{-1} to 200°C followed by 5°C min^{-1} to 280°C and 9 min iso thermal at 280°C. The mass spectra were read at 70 ev, 0.5 s scan interval, 30–800 Da fragments, and a total running time of 43 min.

Results

Bioactive compounds in *S. biafrae* extract

GC-MS spectrometric and chromatographic analyses revealed the presence of 14 compounds in the foliar surface extracts of *S. biafrae* and the six abundant compounds found in the foliar extract are 3-Buten-2-one, 4-(2,6,6-trimethyl-1-cyclohexen-1-yl) also known as *β*-ionone; oleic acid; ficusin; *n*-hexadecanoic acid; 9-octadecenoic acid (Z)-, methyl ester and 1-hydro, 4-nitro-pyrazole (Fig. 1 and Table 1).

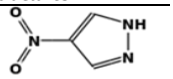
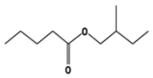
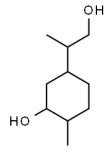
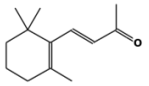
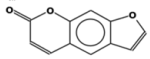
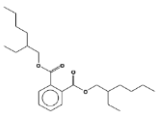
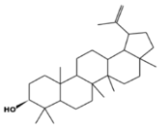
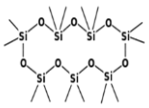
Bioactive compounds in *C. crepidioides* extract

The most abundant constituents of the foliar surface extracts of *C. crepidioides* are ficusin; oleic acid; Z, Z-3,13-octadecadien-1-ol; 9-octadecenoic acid (Z)-methyl ester; 2,7-anhydro-l-galacto-heptulofuranose; lupeol; 1,5-naphthalenediol; 2-dodecanol; 5-(1-hydroxy-2-propanyl)-2-methylcyclohexanol; 3-pyridinecarboxaldehyde O-acetyloxime (E)- and phytol (Fig. 2; Table 2).

Discussion

Fourteen compounds were present in the foliar surface extracts of *S. biafrae*, the six abundant compounds found in the foliar extract are 3-buten-2-one, 4-(2,6,6-trimethyl-1-cyclohexen-1-yl) also known as *β*-Ionone has a retention time of 18.49 and a peak area of 16.14%. It has been established in literature to possess anticancer, anti-inflammatory, antifungal and antibacterial properties (Dong *et al*. 2019). Oleic acid identified has 23.23 as the retention time and a peak area of 14.26%, when compared to the GC-MS result of *C. crepidioides*, oleic acid eluted faster in the foliar extract of *S. biafrae* and had larger peak area. Ficusin was also present in the foliar extract of *C. crepidioides* had a larger peak area when compared to the GC-MS profile result of the foliar extract of *S. biafrae*. The fourth most abundant compound is *n*-hexadecanoic acid popularly known as palmitic acid is an antioxidant, hypocholesterolemic (Abubakar and Majinda 2016) anti-inflammatory, nematicide, pesticide, anti-androgenic flavor, haemolytic, 5-alpha reductase inhibitor, potent mosquitoes larvicide, anticancer and antimicrobial (Hameed *et al*. 2015). The fifth most abundant compound is 9-octadecenoic acid (Z)-, methyl ester also known as Oleic acid methyl ester. It was also present in the foliar extract of *C. crepidioides*. However, it has a larger peak area of 9.03% in comparison to its peak area of 7.34% in *C. crepidioides*. The sixth most abundant compound is 1H-pyrazole, 4-nitro with a peak area of 8.29% and retention time of 9.76. The most abundant constituent of the foliar surface extracts of *C. crepidioides* is ficusin. Ficusin with a peak area of 13.14% and retention time of 32.50, and also 5-(1-hydroxy-2-propanyl)-2-methylcyclohexanol is found to

Table 1: Gas Chromatography-Mass Spectrometric analysis of *Senecio biafrae*

Peak #	RT	Compound Detected	Mol. Formula	MW	Peak Area %	Comp %wt.	m/z	Structures
1	9.76	1H-Pyrazole, 4-nitro-	$C_3H_3N_3O$	97	8.29	4.12	39, 82, 97	
2	10.50	1,5-Naphthalenediol	$C_{10}H_8O_2$	160	4.50	3.70	51, 131, 160	
3	12.00	Pentanoic acid, 2-methyl butyl ester	$C_{10}H_{20}O_2$	172	5.95	6.90	57, 85, 172	
4	14.51	5-(1-Hydroxy-2-propanyl)-2-methyl cyclohexanol	$C_{10}H_{20}$	172	6.38	8.31	43, 90, 172	
5	18.49	3-Buten-2-one, 4-(2,6,6-trimethyl-1-cyclohexen-1-yl)-	$C_{13}H_{20}O$	192	16.14	5.75	43, 57, 254	
6	20.00	n-Hexadecanoic acid	$C_{16}H_{32}O_2$	256	9.76	3.87	43, 73, 256	
7	21.00	Ficusin	$C_{11}H_6O_3$	186	11.24	15.83	51, 158, 186	
8	23.23	Oleic Acid	$C_{18}H_{34}O_2$	282	14.26	7.54	41, 55, 282	
9	23.56	9-Octadecenoic acid (Z)-, methyl ester	$C_{19}H_{36}O_2$	296	9.03	11.85	41, 55, 296	
10	26.47	Phytol	$C_{20}H_{40}O$	296	6.01	7.31	43, 71, 296	
11	31.61	11,14,17-Eicosatrienoic acid, methyl ester	$C_{21}H_{36}O_2$	320	3.75	4.07	67, 79, 320	
12	42.98	Bis(2-ethylhexyl) phthalate	$C_{24}H_{38}O_4$	390	4.69	4.62	57, 149, 390	
13	46.78	Lupeol	$C_{30}H_{50}O$	426	6.01	4.40	43, 95, 426	
14	48.80	Cycloheptasiloxane, tetradecamethyl-	$C_{14}H_{42}O_7Si_7$	519	8.04	3.69	73, 281, 519	

be known as psoralen is an antioxidant and is very effective in the prevention and reduction of free radicals (Javaid *et al*. 2021). It has been found to inhibit the growth of bone cancer; osteosarcoma (Li and Tu 2022). Oleic Acid with a peak area of 9.78% was eluted at 29.01RT. It has been found to have hepatoprotective properties (Hameed *et al*. 2015), antibacterial (Abubakar and Majinda 2016) and apoptotic activities (Fontana *et al*. 2013).

The third most abundant is Z, Z-3,13-Octadecadien-1-ol has a peak area of 7.34% and was eluted at 34.50RT, this has shown antibacterial and antifungal properties (Channabasava *et al*. 2014). Also, 9-octadecenoic acid (Z)-, methyl ester has been used as an alpha-glucosidase inhibitor (Channabasava *et al*. 2014). According to this study by Alkooranee *et al*. (2020), it has antimicrobial properties against *M. smegmatis*, *S. aureus* and *E. coli*. Furthermore, 2,7-anhydro-l-galacto-heptulofuranose has a peak area of 7.33% though no biological activity was recorded. Lupeol

Table 2: Gas Chromatography-Mass Spectrometric analysis of *Crassocephalum crepidioides*

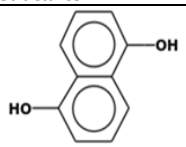
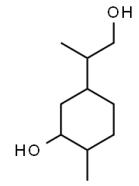
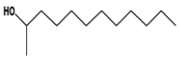
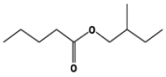
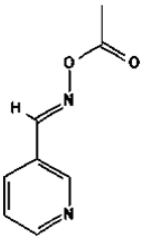
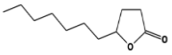
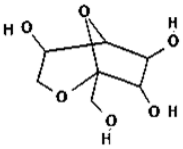
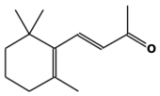
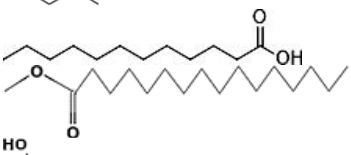
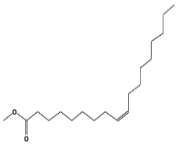
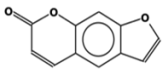
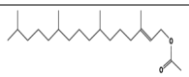
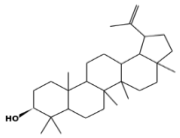
Peak #	RT	Compound Detected	Mol. Formula	MW	Peak Area %	Comp. (%wt.)	m/z	Structures
1	4.30	1,5-Naphthalenediol	$C_{10}H_8O_2$	160	1.83	2.84	51, 131, 160	
2	9.02	5-(1-Hydroxy-2-propanyl)-2-methyl cyclohexanol	$C_{10}H_{20}$	172	4.60	8.21	43, 90, 172	
3	11.10	2-Dodecanol	$C_{12}H_{26}O$	186	1.82	1.09	41, 45, 186	
4	15.98	Pentanoic acid, 2-methyl butyl ester	$C_{10}H_{20}O_2$	172	4.86	9.32	57, 85, 172	
5	19.50	3-Pyridinecarboxaldehyde, O-acetyloxime, (E)-	$C_8H_8N_2O_2$	164	4.31	7.52	44, 104, 164	
6	20.00	2(3H)-Furanone, 5-heptyldihydro-	$C_{12}H_{24}O_2$	184	4.32	7.54	85, 128, 184	
7	25.00	2,7-Anhydro-l-galacto-heptulofuranose	$C_7H_{12}O_6$	192	7.33	5.31	69, 73, 192	
8	25.61	3-Buten-2-one, 4-(2,6,6-trimethyl-1-cyclohexen-1-yl)-	$C_{13}H_{20}O$	192	4.28	6.86	43, 57, 254	
9	26.63	n-Hexadecanoic acid	$C_{16}H_{32}O_2$	256	5.53	7.91	43, 73, 256	
10	28.00	Hexadecanoic acid, methyl ester	$C_{17}H_{34}O_2$	270	5.19	3.31	43, 74, 270	
11	29.01	Oleic Acid	$C_{18}H_{34}O_2$	282	9.78	7.31	41, 55, 282	
12	31.00	9-Octadecenoic acid (Z)-, methyl ester	$C_{19}H_{36}O_2$	296	7.34	9.20	41, 55, 296	
13	32.50	Ficusin	$C_{11}H_6O_3$	186	13.14	16.95`	51, 158, 186	
14	34.50	Z, Z-3,13-Octadecadien-1-ol	$C_{18}H_{34}O$	266	7.97	4.41	43, 102, 266	
15	37.05	Phytol	$C_{20}H_{40}O$	296	5.56	3.48	43, 71, 296	

Table 2: Continued

Table 2: Continued

16	38.50	Phytol, acetate	$C_{22}H_{42}O_2$	338	2.75	2.74	57, 95, 338	
17	39.76	Lupeol	$C_{30}H_{50}O$	426	6.72	4.95	43, 95, 426	
18	41.50	Hexanedioic acid, dioctyl ester	$C_{22}H_{42}O_4$	370	3.85	5.01	57, 129. 370	

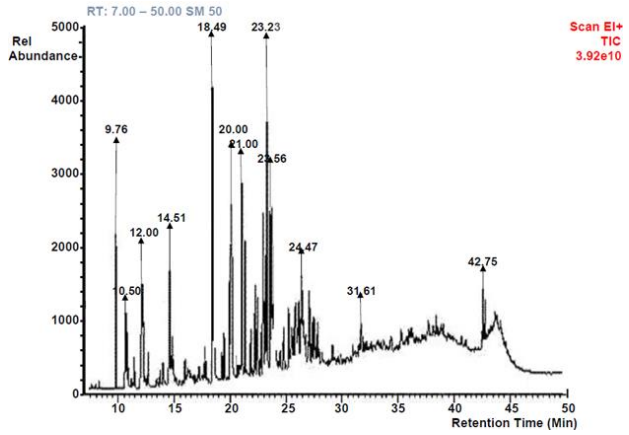


Fig. 1: GC-MS chromatograms of *Senecio biafrae*

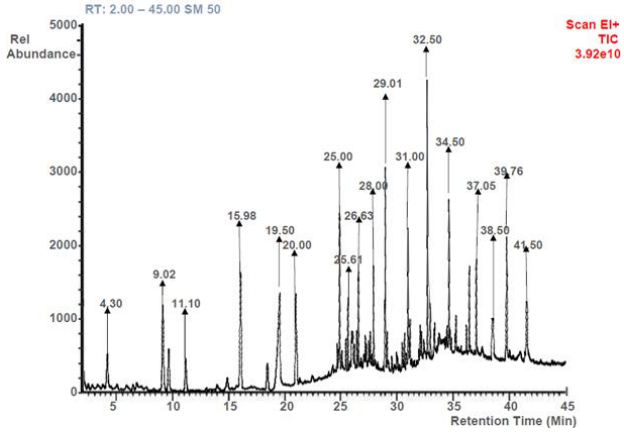


Fig. 2: GC-MS chromatograms of *Crassocephalum crepidioides*

was eluted at 39.76RT and is proven to have anti-inflammatory and anticancer properties (Somwong and Chuchote 2021). Lupeol has been reported to block tumorigenesis by inhibiting growth pathways involved in cell proliferation and death (Bociort *et al*. 2021). 1,5-Naphthalenediol, which is an organic compound used in the cosmetic industries mainly as dyes is also one of the compounds detected in the foliar surface of *C. crepidioides*. It is used as an oxidative and non-oxidative hair dye formulation (Nakagawa *et al*. 2021). Also, 2-dodecanol contains antibacterial and antifungal properties and is used in soap and cosmetic productions (Chanprapai *et al*. 2018). be effective against anticancer, antimicrobial, anti-inflammatory and sometimes used as flavour enhancement in food (Kamatou *et al*. 2013). 3-pyridinecarboxaldehyde, O-acetyloxime, (E)- possesses anti-bacterial property against *E. coli* and *S. albus*, a gram-negative bacterium (Gibson *et al*. 1998). *Staphylococcus aureus* was found to be susceptible to phytol's antibacterial strain activities. The phytol work done by damaging the cell membrane and causing leakage of potassium ions out of the bacteria (Saha and Bandyopadhyay 2020). It also gives effective results in the treatment of arthritis (Carvalho *et al*. 2020). Phytol is also a precursor for vitamins K and E and is proven to be potent against cancer cells in breast (de Alencar *et al*. 2019).

Bis(2-ethylhexyl) phthalate is used as a pesticide, solvent for ink and plasticizer for resins. In addition, a recent study showed that children are susceptible to the effects of bis(2-ethylhexyl) phthalate if they are highly exposed to it. According to (Karthik *et al*. 2020), Cycloheptasiloxane tetradeca-methyl is a bioactive compound and can also be found in Seaweed Liquid Fertilizer (SLF) prepared from the combination of two marine algae namely *Turbinaria ornata* and *Ulva reticulate*. It is a very important compound for plant growth and has antifungal inhibition properties.

Conclusion

The foliar surface of both vegetables supports the traditional usage of these vegetables and affirms that regular consumption of these vegetables is beneficial. The bioactive compounds present in the foliar surface of both vegetables have significant nutraceutical properties. Hence, the two vegetables are a potential source of plant-based drugs.

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Author Contributions

Folashade Habibat Omotehinwa: Performed the experiments, analyzed the data and wrote the manuscript; Olutosin Samuel Ilesanmi: Analysis of data, contributed reagents and revised the manuscript; David Adeniran Oyegoke: Interpretation of data, and proof-read the manuscript; Victory Ayo Olagunju: Contributed reagents and proof-read the mansucrip; Labunmi Lajide: Conceived the research, analysis and interpretation of the data and revised the manuscript.

Conflicts of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

The data will be made available on request

Ethics Approvals

The resaerch was carried out in accordance to the research ethics of the University.

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