Ukrainian Journal of Ecology

Ukrainian Journal of Ecology, 2022, 12(3), 57-65, doi: 10.15421/2022_354

ORIGINAL ARTICLE

Extraction and identification of some metabolites from three Algerian Sahara medicinal plants

I. Radjai*, G. Chakali

Department of Agricultural and Forestry Zoology, Plant Protection Laboratory in Agricultural and Natural Environments Against Crop Pests in the Regions of Algiers and Blida, National School of Agronomics, El Harrach, 16200, Algiers, Algeria

**Corresponding author E-mail: imerad50@gmail.com*

Received: *19-Feb-2022****, Manuscript No.*** *UJE-22-55007****; Accepted:*** *14-Mar-2022****, Pre QC No.*** *P-55007****; Editor assigned:*** *23-Feb-2022****, Pre QC No.*** *P-55007****; Reviewed:*** *01-Mar-2022****, QC No.*** *Q-55007****; Revised:*** *07-Mar-2022****, Manuscript No.*** *R-55007****; Published:*** *21-Mar-2022****.***

Metabolites are widely distributed in plant kingdom and have an ample range of biological properties. Several investigations have reported the medical and insecticidal activties.Thus, the objective of this study is to identify and qualify metabolites of three spontaneous plants:*Caroxylon imbricatum*, *Tetraena alba* and *Cotula cinerea* collected from two ecotypes and analyzed by two known conventional methods:Gas Chromatography-Mass Spectrometry GC QTOF(quadrupole time of flight) MS and Liquid Chromatography-Mass spectrometry LCQTOF(quadrupole time of flight) MS. The chemical study revealed the main metabolites which have biological activities as a part of an alternative to synthetic insecticides, including the presence of N-Butylbenzensulfonamide and Sulfoxycaprylicacid in the three plants. Both N-Carboxy-methionineresidue, butanoicacid and valine were found in *Cotula cinerea* and *Caroxylon imbricatum* (Forssk.). While, Artomunoxanthentrione, Glycoaldehyde, Indoline, Benzensulfonamide and Oxoproline were detected in extracts of *Caroxylon imbricatum* (Forssk.) and *Tetraena alba* (L.f.). Furthermore, Pyrroline is the only compound common in *Cotula cinerea* and *Tetraena alba* (L.f.). A fundamental and applied study on the active compounds of the analyzed plants leads to chemical entities that enter the strategy of a biological control.

Keywords: Spontaneous plants, Metabolites, Insecticidal, Identification, Quantification.

Introduction

The research on the role of plant secondary metabolites as alternative defense to pests has developed considerably.Plant metabolites role against pests has developed considerably in the research for an alternative defence.The biological activity of various secondary metabolites, natural products, have prompted various researchers to take an interest in their medical and insecticidal applications. A variety of metabolites have a defensive role for the plants.

Currently, plant metabolomics are progressively applicable in agrochemistry and especially in biocontrol and development of alternative methods for crop protection (Schrader et al., 2010).

Metabolomics are as well a tool for apprehension plant-microorganism interactions and for the identification of bioactive compounds elaborate in those interactions that could induce inventive biocontrol products (Cedric et al., 2020). In this context, some plant metabolites have been evaluated against some aphids,such as pea aphid (*Acyrthosiphon pisum*). When tested in to evaluate their feeding deterrence and mortality effect on pea aphids by dual choice bioassays, 1-hexadecanol, gliotoxin, cyclopaldic acid and seiridin produced a high feeding deterrence. While aphid mortality was significant for 1-heptadecanol, cytochalasin A, 1-nonadecanol and gliotoxin. Moreover, the phytotoxicity estimation revealed low or non-perceptible plant damaged for cytochalasin A, seiridin and 1-nonadecanol. These results showed seiridin could be used as an alternative to synthetic insecticides. Nevertheless, more studies are required in spite of evaluate its realistic application (Fernández et al., 2018).

Many researchers have reported on the efficacy of plant extracts against insects, mainly thoseinfesting stored products. For example, *Mallotus repandus* leaves and stems extracts have insecticidal activity against *Culex quinquefasciatus* and *Sitophilus Oryzae* (Akhtar et al., 2013, Benzi et al., 2009, Djouahri et al., 2014, Hasan et al., 2015).

Pesticides produced from plants are generally pest-specific and are relatively harmless to organisms. They are also biodegradable and do not pose a risk for the environment. Natural products generally inhibit the development and behaviour of insects (Cedric et al., 2020, Righi et al., 2017). Additionally, they cause early moulting and alter regulatory hormones, consequently insect malformations, sterility, or death (Bhatt et al., 2014).

Some authors (Benzi et al., 2009, Dane et al., 2015) considered that the methanolic extracts of *Artemisia absinthium*, *Juniperus phoenicea,* and *Tetraclinis articulate* have favourable insecticidal and antifungal activities. The test conducted by this investigation showedthat the most toxic extract was that of *A. absinthium*, followed by that of *J. phoenicea*, and *T. articulate*. All three extracts reduced the growth of *Fusarium culmorum* and *F. graminearum*. *T. articulate* was the most effective against the fungi. Results revealed the potential of plant methanolic extracts to control pests common to stored grains due to the presence of several phenolic

acids, flavonoid glycosides, and flavonoid aglycones in plant extracts analyzed by UPLC/PDA/MS(Ultra-performance liquid. chromatographic photodiode array detector mass spectrometry).

Inuloxins A, B and C and α-costic acid, extracted from aerial parts of *Dittrichia viscosa* were evaluated against the cowpea seed beetle *Callosobruchus maculatus.* The results released that oviposition, adult emergence and sex ratio diversified according to the sex of the treated reproducing partner what indicate that compounds tested can have a property of male (or indirect female) chemosterilants bringing on low fecundity of untreated females that intercoursed with treated males (Gueribis et al., 2019).

(Rotundo et al., 2019) reported that *Dittichia viscose* compounds, in particular α-and γ-isomers of costic acid have a bio efficacy towards granary weevil *Sitophilus granarius*.

The results of *Schinus molle, Mirabilis rotundifolia* and *Satureja calamintha* essential oilseffect against *Ryzopertha dominica,* which is the insect most frequently causing serious damages to stored products,showed that the essential oil of *M. rotundifoli*a has a significant insecticidal effect, comparably the essential oil of *S. Calamintha* and *S. molle.* The repulsive effect results of the oils tested showed that the three essential oils have a remarkable repelling effect (Righi et al., 2017).

Studies have been performed in various regions of North Africa on the importance and activity of these three Saharan plants metabolites which evolve on saline soils with difficult conditions, characterized by very pronounced drought; whose precipitations does not exceed 20 mm/year. According to inspections conducted in their environment, these spontaneous plants are used in traditional medicine (Mnafgui et al., 2015). Moreover, (Belguidoum et al., 2015, Belmimoun et al., 2016, Fathy et al., 2017, Kchaou et al., 2016, Khallouki et al., 2015, Lakhdari et al., 2015, Lakhdari et al., 2016) are interested in some biological activities among these insecticidal, acaricidal, antibacterial and antioxidant.

About classification and nomenclature*, Cotula cinerea* (Syn. *Brocchia cinerea* (Del) Vis), belonging to the family "Asteraceae", *Tetraena alba* (L.f.) Beier and Thulin (formerly *Zygophyllum album*) *Caroxylon imbricatum* (Forssk.) Akhani and Roalson (previously *Salsola foetida*) which had changed family from Salsolaceae to Amaranthaceae (Dobignard et al., 2011).

The present study was proposed to provide a qualitative and quantitative analysis of metabolites present in the three plants:*Caroxylon imbricatum* (Forssk.), *Tetraena alba* (L.f.) and *Cotula cinerea* using Gas Chromatography-Mass Spectrometry QTOF and Liquid Chromatography-Mass Spectrometry QTOF. Here, the investigation focused on the chemical analysis of extracts in order to discover new bioactive molecules and the development of effective biopesticides. The purpose which is to assured protection plants against many pests and protect consumer health from insecticide residues.

Materials and Methods

Plant material

The choice of the plants to be tested was done according to their medicinal importance. Aerial parts of *Tetraena alba* and *Cotula cinerea* were collected in winter 2019 from the Oued Souf area located in southern Estand represented by dry weather. The third plant, *Caroxylon imbricatum* was assembled in august from Adrar area positioned in southern west of Algeria with hyper-dry weather. The Table 1 shows the characteristics study sites. After confirmation of species identified, aerial parts of each plant were air-dried and then powdered by using electric grinder then stored away from humidity in bags of conservation.

Table 1. Characterisation of sampling sites.

Loction	Geographical coordinates	Mean annual temperature	Mean annual precipitation	Area (Km^2)	Altitude a (m)
Oued Souf	33°21′21″ N 6°51′47″ E	21	77	35706	80
Adrar	27°52′27″ N0°17′37″ O	24	12	427300	276

Experimental procedures

The extraction was perfomed in the laboratory of Genetics, Breeding and Biochemistry Group of Brassicas, Misión Biológica de Galicia (MBG-CSIC)(Pontevedra, Spain). For each extraction method we considered 3 samples of 100 mg per plant.

Gas chromatography-mass spectrometry GC QTOF(quadrupole time of flight)_MS

Samples were prepared by adding 1 mL of ethylacetate, placing them in Vortex during 15 seconds and putting them in the sonicate during 15 minutes. The material thus prepared is centrifuged for 10 minutes to separate the liquid from the solid. Samples were filtered using a 0.2 µm WHATMAN filter. A dilution of 10 µL of filtered sample and 990 µL of ethylacetate was prepared.

Metabolites were analysed using a Gas Chromatograph coupled to a Quadrupole Time of Flight Mass Spectrometer (GC/QTOF/MS) consisting of a Gas Chromatograph (7890B Agilent Technologies), QTOF Mass Spectrometer (7200 Agilent Technologies) and an autosampler (GC Sampler 120 Agilent Technologies). The column was an HP5-MS (30 m × 0.25 mm inner diameter, 0.25 µm film thickness) from Agilent Technologies. The GC/MS/QTOF is used to separate volatile and semi volatile compounds. The separation of the mixture depends on the length and temperature of the column (T=290°C).

Liquid chromatography-mass spectrometry LC/QTOF(quadrupole time of flight)_MS

The extraction method was modofied from Plant Metabolomics by Hardy and Hall, chapter 8,Ric de Vos, for Brassicaceae from Plant Metabolomics.. Chapter by Ilana Rogachev and Asaph Aharani.

Sample extraction solution

Three products are utilised for this procedure:Methanol (MeOH) HPLC grade, Acetate Buffer:2.3 mL acetic acid and 3.41 g ammonium acetate in 1 L Milipore, Water the Buffer should have a pH of 4.8, if not adjust with 0.1 Ml or 0.1 M NaOH (Reagent grade or ACS grade) and Extraction Buffer:mix acetate buffer (25%) with Methanol (75%), e.g., mix 250 mL with acetate buffer with 750 ml methanol.

Extraction process

The solutions were prepared by dissolving 100 mg of biological material into 1 mL extraction solution, shacked in the vortex for 10 seconds and in the sonicate for 5 minutes at 30 Hz. Tubes were centrifuged for 10 minutes, solutions were Transferred into a new labelled Eppendorf tube. 1 mL extraction solution was added to the pellet of the 1st extraction, shacked for 10 seconds and in the sonicate during 5 minutes at 30 Hz, centrifuge for 10 minutes at 20.000 g. Then, the supernatant was combined with the one from the first extraction and centrifuged for 5 minutes. Afterwards 200 µL of solution was transferred in a HPLC vial with 800 µL of the extraction solution to obtain a dilution of 1/5. Finally, the vials were transferred to the LC/MS for analysis.

The chromatographic apparatus composed of an LC Agilent Infinity System is equipped with an Infinity 1260 gradient pump, a 1260 HiPals automatic injector, a columnthermostat 1290, a photodiode array detector Infinity 1290, Accurate-Mass Quadrupole Time of Flight 6520 (QTOF/MS) Mass Spectrometer equipped with an electrospray ionization source, and a computer with Mass Hunter software for data acquisition andprocessing (Agilent Technologies). The LC/MS is used to separate soluble and untargeted compounds. It also use solvents H_2O and Acetonitril for create a gradient of polarity.(95% water and 5% acetonitril). The measuring parameters of LC/MS QTOF are:a flow of 0.4 mL/min and a pressure of 250 bar.

Metabolites identification

Metabolites were analysed by GC and LC with QTOF/MS detection. The parameters:retention time, molecular formula, m/z experimental and calculated, m/z of the principal fragments and error and Score were allowed the identification of these compounds. The data bases were considered for identification are Metaboscape from Bruker, Sirius, Metlinscripps and Knapsack metabolomics. Specific software for HPLC is Chromeleon Chromatography Data System.

Results

Identification of compounds by GC/MS/QTOF

The results of the GC/MS analysis showed the presence of a substantial diversity of metabolites. Some compounds for each plant were retained for identification(the most abundant chemical components were chosen). The constituents identified are shown in the Tables 2-4.

Table 2. Chemical composition of *Cotula cinerea* analysed by GC/MS/QTOF.

RT	m/z meas	Formula	MsMs	Compound
2.08	141.00	$C_3H_8O_2S_2$	Yes	Ethyl methanethiosulfonate
2.67	90.06	$C_9H_{25}N_5O_2S$	No	Unkonown
2.76	103.07	$C_5H_{10}O_2$	No	Valericacid
7.39	371.10	$C_{16}H_{22}N_2O_4S_2$	Yes	Unkonown
9.3	429.09	$C_{26}H_{20}O_7$	No	Unkonown
13.92	371.10	$C_{27}H_{14}O_2$	No	Unkonown
2.17	158.03	$C_6H_9NO_3S$	Yes	N-Carboxy-methionineresidue
2.17	214.09	$C_{10}H_{15}NO_2S$	Yes	N-Butylbenzenesulfonamide
2.6	89.06	$C_4H_8O_2$	No	Butanoicacid
2.64	61.03	$C_2H_4O_2$	No	Glycoaldehyde
3.38	214.09	$C_{10}H_{15}NO_2S$	Yes	N-Butylbenzenesulfonamide
5.62	223.06	$C_8H_{16}O_6S$	Yes	Sulfoxycaprylicacid
7.16	115.07	$C_6H_{12}O_3$	No	Hydroxyhexanoicacid
7.74	153.12	$C_{10}H_{16}O$	No	Perilylalcohol
11.65	519.14	$C_{18}H_{48}OS_8$		Unkonown

RT: retention time, m/z meas: mass divided by charge number, Ms Ms: tandem mass spectrometry.

Table 3. Chemical composition of *Caroxylon imbricatum* analysed by GC/MS/QTOF.

RT	m/z meas	Formula	MsMs	Compound
2.76	103.07	$C_5H_{10}O_2$	No	Valeric acid
7.39	355.07	$C_{26}H_{10}O_2$	No	Unkonown
7.39	371.1	$C_{27}H_{14}O_2$	Yes	Unkonown
9.3	445.12	$C_{26}H_{20}O_7$	No	Artomunoxanthentrione
9.3	429.09	$C_{14}H_{36}S_7$	No	Unkonown
13.92	371.1	$C_{27}H_{14}O_2$	No	Unkonown
2.17	158.03	$C_6H_9NO_3S$	Yes	N-Carboxy-methionine residue
2.6	89.06	$C_4H_8O_2$	No	Butanoic acid
2.64	61.03	$C_2H_4O_2$	No	Glycoaldehyde
5.57	225.05	$C_{14}H_8O_3$	No	Hydroxyanthraquinone
5.62	223.06	$C_8H_{16}O_6S$	Yes	Sulfoxycaprylicacid
7	299.06	$C_{20}H_{10}O_3$	No	Unknown
11.65	519.14	$C_{18}H_{48}OS_8$		Unknown
11.65	503.11	$C_{26}H_{30}O_2S_4$	Yes	3-[[4-[(2-hydroxy-3phenylsulfanylpropyl)sulfanylmethyl] phenyl]methylsulfanyl]-2-phenylsulfanylpropanol
13.36	593.16	$C_{18}H_{50}N_4O_2S_8$	No	Unkonown
16.03	371.1	$C_{27}H_{16}O_3$	Yes	Unkonown

RT: retention time, m/z meas: mass divided by charge number, Ms Ms: tandem mass spectrometry.

Table 4. Chemical composition of *Tetrana alba* analysed by GC/MS/QTOF.

RT	m/z meas.	Formula	MsMs	Compound
2.67	90.06	$C_9H_{25}N_5O_2S$	No	Unkonown
2.76	103.07	$C_5H_{10}O_2$	No	Valericacid Acides gras alphatiques
6.7	223.06	$C_{10}H_{10}N_2O_4$	No	6,7-Dimethoxyquinazoline-2,4(1H,3H)-dione
7.39	355.07	$C_{26}H_{10}O_2$	No	Unkonown
7.39	371.10	$C_{27}H_{14}O_2$	Yes	Unkonown
8.15	223.06	$C_6H_{14}N_4S_2$	No	Unkonown
9.3	445.12	$C_{26}H_{20}O_7$	No	Artomunoxanthentrione
9.3	429.09	$C_{26}H_{20}O_7$	No	Unkonown
2.17	214.09	$C_{10}H_{15}NO_2S$	Yes	N-Butylbenzenesulfonamide
2.64	61.03	$C_2H_4O_2$	No	Glycoaldehyde
2.76	61.03	$C_2H_4O_2$	No	Glycoaldehyde
3.38	214.09	$C_{10}H_{15}NO_2S$	Yes	N-Butylbenzenesulfonamide
4.87	149.04	$C_6H_6N_4O_2$	No	Methylxanthine
5.62	223.06	$C_8H_{16}O_6S$	Yes	Sulfoxycaprylicacid
7	299.06	$C_{20}H_{10}O_3$	No	Unkonown
8.49	355.07	$C_{26}H_{12}O_3$	No	Unkonown
13.36	593.16	$C_{18}H_{50}N_4O_2S_8$	No	Unkonown

RT: retention time, m/z meas: mass divided by charge number, Ms Ms: tandem mass spectrometry.

Identification of compounds by LC/MS/QTOF

The results of LC/MS analysis are representing in the Tables 5-7.

The biological material tested revealed a diversity of plant-specific metabolites. Common compounds were identified in the plants tested. It should be noted that for the same retention time, the analyzed metabolites have different spectrometric masses and remain dependent on their composition. The compounds identified for Cotula cinerea are:Ethylmethanethiosulfonate, N Butylbenzenesulfonamide, Glycoaldehyde, Hydroxyhexanoic acid, Perilyl alcohol, Proline, Pipecolic acid, 7-Hydroxy-2-methoxyflavone, Pechueloic acid, N,N-Dimethyldodecylamine N-oxide, Dodecalactone and Octene.

In Caroxylon imbricatum (Forssk.) the following compounds identified are:Hydroxyanthraquinone, Sulfoxycaprylic acid and 3-[[4-[(2-hydroxy-3-phenylsulfanylpropyl) sulfanylmethyl] phenyl] methylsulfanyl]-2-phenylsulfanylpropanol. Valericacid, Methylxanthine and Methylisoxazole are identified from *Tetraena alba*.

Those results reveal the presence of N-Butylbenzensulfonamide and Sulfoxy-caprylicacid in the three plants. N-Carboxy-methionine residue, Butanoic acid and Valine were found in those of *Cotula cinerea* and. *Caroxylon imbricatum* (Forssk.) Artomunoxanthentrione, Glycoaldehyde, Indoline, Benzensulfonamide and Oxoproline were detected in extracts of *Caroxylon imbricatum* (Forssk.) and *Tetraena alba*. Pyrroline is the only compound common in *Cotula cinerea* and *Tetraena alba*.

Table 5. Chemical composition of *Cotula cinerea* analysed by LC/MS/QTOF.

RT	m/z meas.	Formula	Compound
0.53	23.60	$C_{10}H_{15}NO_2S$	N-Butylbenzensulfonamide
0.95	11.80	$C_5H_{11}NO_2$	Valine
0.99	11.60	$C_5H_9NO_2$	Proline
0.99	7.00	C_4H_7N	Pyrroline
1.07	13.00	$C_6H_{11}NO_2$	Pipecolicacid
17.98	36.42	$C_{21}H_{25}N_5O$	Unkonown
19.96	28.70	$C_{15}H_{10}O_6$	7-Hydroxy-2-methoxyflavone
20.1	23.11	$C_{15}H_{20}O_3$	Pechueloic acid
21.74	23.11	$C_{15}H_{20}O_3$	Same. Could be an isomer or other compound
22.68	23.02	$C_{14}H_{31}NO$	N,N-DimethyldodecylamineN-oxide
27.2	30.92	$C_{17}H_{26}N_4$	Unkonown
27.21	11.11	C_8H_{14}	Unkonown
27.21	19.91	$C_{12}H_{22}O_2$	Dodecalactone
27.34	29.72	$C_{16}H_{34}O_3$	Unkonown
29.59	59.32	$C_{34}H_{36}N_6O_4$	Unknown
29.71	23.60	$C_8H_9N_7S$	Unkonown
30.01	47.73	$C_{26}H_{54}N_4S$	Unkonown
31.16	14.90	$C_8H_6O_4$	Unkonown
31.16	11.31	C_8H_{16}	Octene

RT: retention time, m/z meas: mass divided by charge number.

Table 6. Chemical composition of *Caroxylon imbricatum* (Forssk.) analysed by LC/MS/QTOF.

RT	m/z meas.	Formula	Compound
3.67	12.00	C_8H_9N	Indoline
0.53	23.60	$C_{10}H_{15}NO_2S$	N-Butylbenzensulfonamide
0.67	14.10	$C_6H_4O_2S$	Unkonown
0.65	15.80	$C_6H_7NO_2S$	Benzensulfonamide
0.77	23.90	$C_5H_{11}N_4O_3PS$	Unkonown
0.83	23.60	$C_{10}H_{15}NO_2S$	N-Butylbenzensulfonamide
0.93	21.70	$C_5H_{10}N_6O_5$	Unkonown
0.95	11.80	$C_5H_{11}NO_2$	Valine
1.54	13.00	$C_5H_7NO_3$	Oxoproline
19.27	46.93	$C_{31}H_{40}N_4$	Unkonown
27.2	30.92	$C_{17}H_{26}N_4$	Unkonown
27.21	11.11	C_8H_{14}	Unkonown
27.34	29.72	$C_{16}H_{34}O_3$	Unkonown
29.71	23.60	$C_8H_9N_7S$	Unkonown
30.01	47.73	$C_{26}H_{54}N_4S$	Unkonown
31.16	14.90	$C_8H_6O_4$	Unkonown
31.16	11.31	C_8H_{16}	Unkonown

RT: retention time, m/z meas: mass divided by charge number.

Table 7. Chemical composition of *Tetrana alba* analysed by LC/MS/QTOF.

RT	m/z meas.	Formula	Compound

3.67	12.00	C_8H_9N	Indoline
0.53	23.60	$C_{10}H_{15}NO_2S$	N-Butylbenzensulfonamide
0.66	21.40	$C_{10}H_{15}NO_2S$	N-Butylbenzensulfonamide
0.67	14.10	$C_6H_4O_2S$	Unkonown
0.65	15.80	$C_6H_7NO_2S$	Benzensulfonamide
0.83	23.60	$C_{10}H_{15}NO_2S$	Unkonown
0.99	7.00	C_4H_7N	Pyrroline
1.54	13.00	$C_5H_7NO_3$	Oxoproline
1.54	8.40	C_4H_5NO	Methylisoxazole
27.2	30.92	$C_{17}H_{26}N_4$	Unkonown
27.21	11.11	C_8H_{14}	Unkonown
27.34	29.72	$C_{16}H_{34}O_3$	Unkonown
29.71	23.60	$C_8H_9N_7S$	Unkonown
30.01	47.73	$C_{26}H_{54}N_4S$	Unkonown
31.16	14.90	$C_8H_6O_4$	Unkonown
31.16	11.31	C_8H_{16}	Unkonown

RT: retention time, m/z meas: mass divided by charge number.

Discussion

Many investigations have reported on identification of medicinal plants main constituents because of their interesting biological properties. Essential oil extracted from aerial parts of *Cotula cinerea* analyzed by GC/MS (Gas Chromatography-Mass Spectrometry) showed that (E)-citral, limonene epoxide cis-, thymol methyl ether, carvacrol, trans-carveol, carvone and trans-piperitol were the considerable compounds (Djellouli et al., 2015). Likewise, (Chouikh et al., 2015) noted the presence of 3-carène, thujone, santolinatriene and camphor from the oil obtained from a specimen collected in Oued Souf area. Phytochemical charactezisation of *Cotula cinerea* by GC/MS (Gas Chromatography-Mass Spectrometry) showed that the oil contains trans thujone, santalina triéne, α-pinéne, sabinene, cineole, δ-terpinene, camphor, β-terpineol and terpin-4-ol as the major constituents (BenAmor *et al.*,2019). The chiral flavanones analysis isolated from methanol extract of *cotula cinerae* aerial parts by TLC methods showed the presence of three compounds:hesperidin, hesperitin and eriodoctyol (Belboukhari et al., 2012).

Khan et al., (2003) reported the identification of three metabolites from whole plant of *Caroxylon imbricatum*: N-[2-(3,4-dihydroxyphenyl)-2-hydroxyethyl]-3-(4-methoxyphenyl) prop-2-enamide, N-[2-(3,4dihydroxyphenyl)-2-hydroxyethyl]-3-(3,4 dimethoxyphenyl) prop-2-enamide and N-[2-(3-hydroxy-4-methoxyphenyl)-2-hydroxy ethyl] 3-(4-methoxy phenyl)-prop-2-enamide. Those new phenolic compounds have an appreciable antioxidant activity and tyrosinase inhibition. These results are in accordance with many other studies (Asif et al., 2016, Lee et al., 2012, Ghorab et al., 2017, Zhao et al., 2020, Tundis et al., 2007). The primary and secondary metabolites of *salsola collina* were investigated using ultra high performance liquid chromatography-electrospray ionization-tandem mass spectrometry (UPLC/ESI/MS/MS). The main compounds were flavonoids, phenolic acids, lipids, aminos acids and derivatives. The flavonoids were the major putative antioxidant components (Shipeng et al., 2021). Phytochemical analysis of *Salsola imbricata* showed the presence of anthraquinones, reducing sugar, tannins, saponins, flavonoids, alkaloids and cardiac glycosides (Ajaib et al., 2019). In addition, two new triterpenes have been isolated from *Salsola imbricata* growing in Egypt. Their structures have been established as 3-O-b-D-xylopyranosyl-(1-2)-O-b-D-glucuronopyranosyl-akebonic acid 28-O-b-D-glucopyranoside and 3-O-b-D-xylopyranosyl-(1-2)-O-b-D-glucuronopyranosyl-29-hydroxyoleanolic acid 28-O-b-D-glucopyranoside (Arafa et al., 2011). HPLC(high performance liquid chromatography) and LC/MS(liquid chromatography) analysis was applied to recognize the main components in the ethyl acetate extract D-galacturonic acid, orsellic acid, protocatechuic acid, caffeic acid, salicylic acid,vanillic acid, syringic acid, 4-hydroxycinnamic acid, ferulic acid, 4-hydroxybenzoic acid (Xin et al., 2020). Furthermore, *Salsola imbricata* phytochemical screening has been reported and showed the presence of triterpene (Hamed et al., 2011, Uzma et al., 2014).

Also, the evaluation of antioxidant activity of *Tetraena alba* (L.f.) extracts showed that acid ascorbic have a considerable antioxidant effect (Benslama et al., 2016). Two phenolic compounds; Gallic acid and ascorbic acid were extracted from *Tetraena alba* (L.f.) floral honey, had a significatively antioxidant activity (Mesbahi et al., 2019). Phytochemical investigation of the aerial parts of *Zygophyllum coccineum* L. led to the isolation of 3-O-[β-D-(2-O-sulphonyl)-quinovopyranosyl] quinovic acid, 3-O-[β-D-glucopyranosyl] quinovic acid,-28-O-β-D-glucopyranosyl ester, 3-O-[β-D-quinovopyranosyl] quinovic acid-28-O-β-D-glucopyranosyl ester, 3-O-[α-L-arabinopyranosyl-(1→2)-β-D-quinovopyranosyl]quinovicacid, 3-O-[α-L-arabinopyranosyl-(1→2)-β-D-quinovopyranosyl]quinovicacid-28-O-β-D-glucopyranosyl ester, 3-O-[β-D-(2-O-sulphonyl)quinovopyranosyl]quinovicacid-28-O-β-D-glucopyranosylester, and 3-O-[β-D-(2-O-sulphonyl) glucopyranosyl] quinovicacid, isorhamnetin-3-O-rutinoside, and β-sitosterolglucoside (Amin et al., 2011, Smati et al., 2007). Similar observations have been made for (Ayad et al., 2008): purification and separation conducted by different chromatographic methods induced identification of two compounds, β-sitosterol isolated for the first time in the Zygophyllaceae and 3-*O*-rhamnoglucosyl isorhamnétine as a new compound of *Zygophyllum* genus.

Phytochemical content of *Cotula cinerea* analyzed to find principal components which may combine its use as a medicinal plant in the southeast of Morocco. The high echinoids and flavonoids quantity (Khallouki et al., 2015). Likewise,the chemical composition was dominated by the presence of oxygenated monoterpenes followed by monoterpene hydrocarbons, oxygenated sesquiterpenes and sesquiterpene hydrocarbons (Djellouli et al., 2015, Fathy et al., 2017, Abdenbi et al., 2014). In addition, (Chouikh et al., 2015) indicated that *cotula cinerea* essential oil analyzed by GC/MS, constituted of 22 chemical compounds dominated by: 3-Carène, Thujone, Santolinatriene and Camphor during the flowering stage. While 21 chemical compounds were obtained during the fruiting period with the dominance: Thujone, 3-Carène, Eucalyptol, Santolinatriene and Camphor. *Cotula cinerea* essential oil and hexane extract have been used in Moroccan traditional medicine as a source of potent anticancer products, due to their various bioactive compounds (Guaouguaou et al., 2018).

Conclusion

The three spontaneous plants collected from desert are rich of metabolites which ones could be applied as a potential source to control pests of insects. Also, it appears that these compounds are cure for some disease and possesses several biological activities such as antifungal, antibacterial, antioxidant. Further investigation on the isolation and identification of biocompounds in the plan may lead to chemical entities with the potential for biocontrol strategy use.

Acknowledgment

The study was funded by the Ministry of High Education and Scientific Research, Ecole Nationale Supérieure Agronomique, Phd Program, Algeria. We would like to thank everyone who have provided a lot of help during the progress of this experiment. We thank Pablo Velasco (MBG-CSIC) for providing doctoral research collaboration.

References

Abdenbi, A., Abdelwahed, D., Bouaaza, M., Touati, B. (2014). Screening phytochimique et activite antibacterienne de l'huile essentielle de Cotula Cinerea (Gartoufa) dans la région de Bechar. International Journal of Research in Engineering and Technology, 2:49-54.

Ajaib, M., Farooq, S., Khan, K.M., Perveen, S., Shah, S. (2019). Phytochemical analysis and anthelmintic activity of Salsola imbricata. Journal of the Chemical Society of Pakistan, 41:198-202.

Akhtar, M., Arshad, M., Raza, A.B.M., Chaudhary, M.I., Iram, N., Akhtar, N., Mahmood, T. (2013). Repellent effects of certain plant extracts against rice weevil Sitophilus oryzae L (Coleoptera:Curculionidae). International Journal of Agricultural and Applied Sciences, 5:69-73.

Amin, E., El-Hawary, S.S., Fathy, M.M., Mohammed, R., Ali, Z., Tabanca, N.J. (2011). Zygophylloside S, a new triterpenoid saponin from the aerial parts of *Zygophyllum coccineum* L. Planta Medica, 77:488-491.

Arafa, I.H., Milena, M., Mohamed, G.S., Usama, A.M., Moatz, M.T., Angela, P., Sonia, P. (2011). Triterpene saponins from Salsola imbricata. Phytochemistry Letters, 4:353-356.

Asif, R.M., Younas, M., Schlecht, E. (2016). *In vitro* efficacy of selected medicinal plants from Cholistan desert, Pakistan, against gastrointestinal helminths of sheep and goats. Journal of Agriculture and Rural Development in the Tropics and Subtropics, 117:211-224.

Ayad, R. (2008). Recherche et détermination structurale des métabolites secondaires de l'espèce: Zygophyllum cornutum (Zygophyllaceae). Mémoire de Magister, Université de Constantine, p:124.

Belboukhari, N., Cheriti, A., Bouanini, M. (2012). Chiral separation of flavanones from Cotula cinerea by TLC methods. Phytochem and Biosub Journal, 6:2170-1768.

Belguidoum, M., Dendougui, H., Kendour, Z. (2015). *In vitro* antioxidant properties and phenolic contents of *Zygophyllum album* L. from Algeria. Journal of Chemical and Pharmaceutical Research, 7:510-514.

Belmimoun, A., Meddah, B., Meddah, A.T., Sonnet, P. (2016). Antibacterial and antioxidant activities of the essential oils and phenolics extracts of Myrtus communis and Zygophylum album from Algeria. Journal of Fundamental and Applied Sciences, 8:510-552.

BenAmor, M.L., Benchikha, N., Eman, R., Neghmouche, N.S. (2019). Phytochemical characterization, *in vitro* cytotoxic and antibacterial activity of Cotula cinerea (Delile) vis essentail oil. Journal of Natural Remedies.

Benslama, A., Harrar, A. (2016). Free radicals scavenging activity and reducing power of two Algerian sahara medicinal plants extracts. International Joural of Herbal Medicine, 4:158-161.

Benzi, V., Stefanazzi, N., Ferrero, A.A. (2009). Biological activity of essential oils from leaves and fruits of pepper tree (Schinus molle L.). Chilean Journal of Agricultural Research, 69:154-159.

Bhatt, P., Thodsare, N., Srivastava, R.P. (2014). Toxicity of some bioactive medicinal plant extracts to Asian army worm, Spodoptera litura. Journal of Applied and Natural Sciences, 6:139-143.

Cedric, B., Azucena, G.C., Claire, P.C. (2020). Plant metabolomics to the benefit of crop protection and growth stimulation. Advances in Botanical Research.

Chouikh, A., Mayache, B., Maazi, M.C., Hadef, Y., Chefrour, A. (2015). Chemical composition and antimicrobial activity of essential oils in Xerophytic plant Cotula cinerea Del (Asteraceae) during two stages of development:flowering and fruiting ARTIC. Journal of Applied Pharmaceutical Science, 5:029-034.

Dane, Y., Mouhouche, F., Canela, G.R., Delpino, R.A. (2015). Phytochemical analysis of methanolic extracts of *Artemisia absinthium* L. 1753 (Asteraceae), *Juniperus phoenicea* L., and Tertraclinis articulata Mast, (Cupressaceae) 1892 and evaluation of their biological activity for stored grain protection. Arabian Journal for Science and Engineering.

Djellouli, M., Benmehdi, H., Mammeri, S., Moussaoui, A., Ziane, L., Hamidi, N. (2015). Chemical constituents in the essential oil of the endemic plant Cotula cinerea (Del.) from the southwest of Algeria. Asian Pacific Journal of Tropical Biomedicine, 5:870-873.

Djouahri, A., Saka, B., Boudarene, L., Benseradj, F., Aberrane, S., Aitmoussa, S., Chelghoum, C., Lamari, L., Sabaou, N., Baaliouamer, A. (2014). *In vitro* synergistic/antagonistic antibacterial and anti-inflammatory effect of various extracts/essential oil from cones of Tetraclinis articulata (Vahl) masters with antibiotic and anti-inflammatory agents. Industrial Crops and Products, 56:60-66.

Dobignard, A., Chatelain, C. (2011). Index synonymique et bibliographique de la flore d'Afrique du Nord. C.J.B.G. Genève:Editeur des conservatoire et Jardin botanique ville de Genève, 2:430.

Fathy, K.F., Dekinash, M.F., Beltagy, A.M., Moataz, B.E.M., Khattab, A.R. (2017). Chemical composition and biological activity of essential oil from Cotula cinerea (Del.) growing wildly in the middle east:a short review. International Journal of Pharmacognosy and Chinese Medicine.

Fernández, T.A., Cimmino, A., Masi, M., Rubiales, D., Evidente, A. (2018). Antifeedant activity of long-chain alcohols, and fungal and plant metabolites against pea aphid (Acyrthosiphon pisum) as potential biocontrol strategy. Natural Product Research, 33:2471-2479.

Ghorab, H., Khetta, F.A., Lehbili, M., Kabouche, A., Magid, A.A., Harakat, D., Voutquenne, N.L., Kabouche, Z. (2017). A new cardenolide and other compounds from *Salsola tetragona*. Natural Product Communications.

Guaouguaou, F.E., Ahl-Bebaha, M.A., Taghzouti, K., Bouyahya, A., Bakri, Y., Dakka, N., EsSafi, N. (2018). Cytotoxicological investigation of the essential oil and the extracts of Cotula cinerea and Salvia verbenaca from Morocco. Biomedical Research International.

Gueribis, F., Zermane, N., Khalfi, H.O., Siafa, A., Cimmino, A., Boari, A., Evidente, A. (2019). Bioefficacy of compounds from Dittrichia viscosa (Asteraceae) as protectant of chickpea seeds against the cowpea seed beetle Callosobruchus maculatus (Coleoptera:Chrysomelidae). Journal of Plant Diseases and Proectiont.

Hasan, M.R., Uddin, N., Hasan, M.M., Hossain, M.M., Kamal, M.M., Fatema, K., Mazumder, H.M.L.R., Bashar, K., Rana, M.S. (2015). Larvicidal and insecticidal effects of different extracts of mallotus repandus (willd.) Muellarg leaf and stem against Culex quinquefasciatus say (Diptera:Culicidae) and Sitophilus oryzae linn. (Coleoptera:Curculionidae). International Journal of Pharmacy, 5:127-133.

Kchaou, M., BenSalah, H., Mnafgui, K., Abdennabi, R., Gharsallah, N., Elfeki, A., Damak, M., Allouche, N. (2016). Chemical composition and biological activities of Zygophyllum album (L.) essential oil from Tunisia. Journal of Agricultural Science and Technology, 18:1499-1510.

Khallouki, F., Sellam, K., Koyun, R., Ricarte, I., Alem, C., Elrhaffari, L.,Owen, W.R. (2015). Phytoconstituents and *in vitro* evaluation of antioxidant capacities of Cotula cinerea (Morocco) methanol extracts. Records of Natural Products, 9:572-575.

Khan, K.M., Maharvi, G.M., Abbaskhan, A., Hayat, S., Khan, M.T.H., Makhmoor, T., Choudhary, M.I., Shaheen, F., Atta, U.R. (2003). Three tyrosinase inhibitors and antioxidant compounds from salsola foetida. Helvetica Chimica Acta, p:86.

Lakhdari, W., Dehliz, A., Acheuk, F., Soud, A., Hammi, H., Mlik, R., Doumandji, M.B. (2015). Acaricidal activity of aqueous extracts against the mite of date palm Oligonychus afrasiaticus Meg (Acari:Tetranychidae). Journal of Medicinal Plants Studies, 3:113-117.

Lakhdari, W., Dehliz, A., Acheuk, F., Mlik, R., Hammi, H., Maatallah, S., Doumandji, M.B. (2016). Biological control assay against date palm diseases by using an aqueous extract of Zygophyllum album in the southeast of Algeria. International Journal of Herbal Medicine, 4:31-37.

Lee, H.J., Pan, C.H., Kim, E.S., Kim, C.Y. (2012). Online high performance liquid chromatography (HPLC)-ABTS+ based assay and HPLC-electrospray ionization mass spectrometry analysis of antioxidant phenolic compounds in Salsola komarovii. Journal of the Korean Society for Applied Biological Chemistry, 55:317-321.

Mesbahi, M.A., Ouahrani, M.R., Rebiai, A., Amara, D.G., Chouikh, A. (2019). Characterization of *Zygophyllum album* L. monofloral honey from el-oued, Algeria. Current Nutrition and Food Sciences, 15:476-483.

Mnafgui, K., Kchaou, M., BenSalah, H., Hajji, R., Khabbabi, G., Elfeki, A., Allouche, N., Gharsallah, N. (2015). Essential oil of *Zygophyllum album* inhibits key-digestive enzymes related to diabetes and hypertension and attenuates symptoms of diarrhea in alloxan-induced diabetic rats. Pharmaceutical Biology, pp:1-8.

Righi, K., Righi, F.A., Boubkeur, A., Boungab, K., Elouissi, A., Djendara, A.C. (2017). Toxicity and repellency of three algerian medicinal plans agianst pests of stored product:Ryzopertha dominica (Fabricius) (Coleoptera:Bostrichidae). Banat's Journal of Biotechnology.

Rotundo, G., Paventi, G., Barberio, A., DeCristofaro, A., Notardonato, I., Russo, M.V., Germinara, G.S. (2019). Biological activity of Dittrichia viscosa (L.) Greuter extracts against adult Sitophilus granarius (L.) (Coleoptera,Curculionidae) and identification of active compounds. Scientific Reports, 9:6429.

Schrader, K.K., Andolfi, A., Cantrell, C.L., Cimmino, A., Duke, S.O., Osbrink, W., Wedge, D.E., Evidente, A. (2010). A survey of phytotoxic microbial and plant metabolites as potential natural products for pest management. Chemistry and Biodiversity, 7:2261-2280.

Shipeng, L., Ye, C., Ying, D., Yinhui, Z., Di, Z., Liyan, Z., Huiyuan, Y. (2021). Widely targeted metabolomics analysis of different parts of salsola collina pall. Molecules, 26:1126.

Smati, D., Offer, A.C.M., Miyamoto, T., Hammiche, V., Dubois, M. (2007). Ursane-type triterpene saponins from Zygophyllum geslini. Helvetica Chimica Acta, 90:712-719.

Tundis, R., Loizzo, M.R., Statti, G.A., Menichini, F. (2007). Inhibitory effects on the digestive enzyme-amylase of three Salsola species (Chenopodiaceae) *in vitro*. Pharmazie, 62:473-475.

Uzma, M., Anjum, P., Syeda, Q. (2014). Comparative pharmacognostic evaluation of some species of the genera Suaeda and Salsola leaf (Chenopodiaceae). Pakistan Journal of Pharmaceutical Sciences, 27:1309-1315.

Xin, L.Z., Wen, Y., Zhao, Z.L., Hong, J., Yan, L.G. (2020). Salsola collina ethyl acetate extract alleviates diabetic gastroparesis possibly through oxidative stress inhibition. Paper presented at The 3rd International Conference on Agricultural and Food Science, IOP Conference Series:Earth and Environmental Science, 559:012021.

Zhao, X., Wang, H., Zhang, Z., Jin, H., Gong, Y. (2020). Effects of ethyl acetate extract of salsola collina on brain-gut peptides and interstitial cells of gastric Cajal in rats with diabetic gastroparesis. Iranian Journal of Basic Medical Sciences, 23:1218-1224.

Citation:

Radjai, I., Chakali, G. (2022). Extraction and identification of some metabolites from three Algerian Sahara medicinal plants. *Ukrainian Journal of Ecology.* 12:57-65.