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Chemotypes investigation of essential oils of "Guertoufa" herbs

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Abstract
The famous saharian herbs called "guertoufa", used in North African folk medicine, are related to many Asteraceae species belonging to the genus *Cotula* (*C. cinera*), *Chrysanthemum* (*C. macrocarpum, C. fuscatum, C. trifrucatum, C. derticolum*) and *Matricaria* (*M. pubescens*). Our investigation on chemotypes of essential oils of *Cotula, Chrysanthemum and Matricaria* species showed the main presence of santolinatriene (7.2%-13.0%) thujone (12.9%-41.4%) and camphor (5.5%-27.4%) in essential oils of *Cotula*. α-Pinene (0.2%-14.8%), *β*-pinene (0.1%-9.5%), limonene, (0.2%-21.8%), 1,8-cineole (4.9%-30.4%), camphor (0.9%-29.2%), *β*-farnesene (0.2%-25.0%) and germacrene D (0.2%-10.6%) were characterizing *Chrysanthemum* essential oils. Spathulenol (0.1%-19.4%), *α*-bisabolol oxide B (0.1%-7.0%), *α*-bisabolol (5.2%-56.9%) and *α*-bisabolol oxide A (10.2%-53.6%) were mainly found in essential oil of *Matricaria*.

Keywords: Asteraceae, chemotype, *Cotula, Chrysanthemum, Matricaria, Guertoufa*

1-Introduction
The Asteraceae is the largest family with more than 24 000 species belonging to over 1700 genera distributed around the globe except for Antarctica [1]. This family contains many major ornamental and medicinal plants [2]. The Anthemideae is one of the largest tribe of Astaeraceae with more than 1740 species, predominantly distributed in Eurasia, North and South Africa, with fewer species in North America and Australia [3]. This tribe contains several species known as popular medicinal plants, in particular the famous herbs called "guertoufa" in North African Sahara [4-7], related to many Asteraceae species belonging to the genus *Cotula* (*C. cinera*), *Chrysanthemum* (*C. fuscatum*, *C. deserticolum, C. macrocarpum*, "guertoufa bayda", *C. trifurcatum* "guertoufa safra") and *Matricaria* (*M. pubescens*). In continuation of our works on Asteraceae [8-21], we report here a bibliographic investigation on the chemotypes of essential oils of *Cotula, Chrysanthemum* and *Matricaria* species grown which are responsible for the therapeutic effects of the corresponding oils used in folk-medicine as anti-inflammatory, analgesic, antiseptic and for the treatment of stomachache [22]. Several biological properties have been reported, such as antibacterial [23] an analgesic [24; 25]. In addition, established chemotypes could be tested as green corrosion inhibitors.

2. Experimental
Plant material
The investigated plants for their essential oils compositions, belonging to the genus *Cotula, Chrysanthemum* and *Matricaria,* related to "guertoufa" have been collected from different localities, as presented in table 1.

Extraction
Fresh aerial parts of each plant in table 1 were submitted to hydrodistillation for 3hours using a Clevenger-type apparatus. The obtained essential oils were dried with anhydrous sodium sulfate and stored at 4°C, until analyzed.

Table1. Plant material data of studied *Cotula*, *Chrysanthemum* and *Matricaria* essential oils

Code	Species (aerial parts)	Locality	Ref.
Cot1	*Cotula corronnopifolia* L.	Monastir, Tunisia	[26]
Cot2	*Cotula anthemoides* L.	Tiguentourine, Ain Amenas, Algeria	[27]
Cot3	*Cotula cinerea* L.	Province Zagora, Morocco	[28]
Chry1	*Chrysanthemum macrocarpum*	Ghardaia, Algeria	[9]
Chry2	*Chrysanthemum yoshinaganthum*	Naka River, Tokushima, Japan	[29]
Chry3	*Chrysanthemum cuneifolium*	Naka River, Tokushima, Japan	[29]
Chry4	*Chrysanthemum indicum* L.	Zhejiang, China	[30]
Chry5	*Chrysanthemum viscidehirtum* Schott. Tell.	Rabat, Morocco	[31]
Chry6	*Chrysanthemum coronarium* L.	Zaghouan, Tunisia	[32]
Chry7	*Chrysanthemum indicum* L.	Hubei, China	[33]
Chry8	*Chrysanthemum coronarium* L.	Mursia, Spain	[34]
Chry9	*Chrysanthemum trifurcatum* L	Zeramdine, Tunisia	[35]
Chry10	*Chrysanthemum morifolium Ramat* L.	Tongxiang, China	[36]
Mat1	*Matricaria chamomilla* L.	Neyshabur, Khorasan-Razavi, Iran	[37]
Mat2	*Matricaria chamomilla* L.	Fars province, sub-central of Iran	[38]
Mat3	*Matricaria recutita* L.	Isfahan, Iran	[39]
Mat4	*Matricaria pubescens* (Desf.) Schultz	Ghardaia, Algeria	[10]
Mat5	*Matricaria chamomilla* L.	Golestan, Iran	[40]
Mat6	*Matricaria matricarioides*	Tallinn, Estonia	[41]

3. Results and Discussion

From Table 2, the essential oils of Cot2 (*Cotula anthemoides* from Tinguentourine, Ain Amenas, Algeria) and Cot3 (*Cotula cinera* from Zagora, Morocco) contain similar main components camphor (27.4%; 5.5%), santolinatriene (13.0%; 7.2%), and thujone (12.9%; 41.4%), respectively. Camphene (10.7%) and *α*-curcumene (5.3%) are mainly found in Cot2 essential oil while *cis*-verbenyl acetate (24.7%) is exclusive to Cot3. The composition of Cot1 (*Cotula corronnopifolia* from Monastir,Tunisia) is

Table 2 Major components (≥ 5%) of essential oils of *Cotula* (**Cot1-Cot3)**

Compounds	Percentage %		
	Cot1 [26]	**Cot2** [27]	**Cot3** [28]
Santolinatriene	-	13.0	7.2
Camphene	-	10.7	1.6
Thujone	-	12.9	41.4
Camphor	-	27.4	5.5
cis-Verbenyl acetate	-	-	24.7
α-Amorphene	5.2	-	-
Agarospirol	10.4	-	-
α- Curcumene	-	5.3	0.2
Ethyl octadecanoate	21.7	-	-
1-Eicosanol	17.1	-	-
Hexacosane	31.7	-	-
Heptacosane	28.4	-	-
Octacosane	5.4	-	-

quite different with the main presence of hydrocarbons and their functionalized derivatives. Through these data, it appears that thujone (41.4%) is a chemotype of the Moroccan Species *Cotula cinerea* L. essential oil which may be tested as a green corrosion inhibitor [42].

Table 3 Major components (≥ 5%) of essential oils of *Chrysanthemum* species **(Chry1-Chry10)**

Compounds*	**Percentage %**									
	Chry1 [9]	**Chry2** [29]	**Chry3** [29]	**Chry4** [30]	**Chry5** [31]	**Chry6** [32]	**Chry7** [33]	**Chry8** [34]	**Chry9** [35]	**Chry 10** [36]
α-Pinene	-	1.0	5.7	3.6	0.5	0.4	1.4	14.8	5.3	0.2
Camphene	-	tr	2.2	0.3	-	1.1	2.0	5.2	0.1	1.0
β-Pinene	-	0.1	0.3	-	0.6	0.7	1.0	9.5	8.8	-
Limonene	-	tr	-	-	21.8	0.3	-	0.7	20.9	0.2
1,8-Cineole	-	6.8	23.0	4.9	-	-	30,4	-	10.6	-
ϒ-terpinene	-	-	-		0.5	0.2	0.5	-	19.1	-
Cis-Verbenol	-	-	-	21.7	-	-	-	-	-	-
Camphor	-	tr	14.7	-	-	6.0	23.5	29.2	0.9	-
Borneol	-	-	2.5	0.7	-	-	8.3	-	0.6	0.3
trans-Chrysenthenyl acetate	-	-	-	-	-	12.8	Tr	-	-	-
cis-Chrysenthenyl acetate	-	-	-	-	-	21.8	-	Tr	-	-
Isobornyl acetate	-	-	-	0.6	-	2.8	10.9	0.9	0.3	-
Myrtenol	-	54.8	tr		-	-	-	-	0.5	-
β-Farnesene	-	1.7	2.6		25.0	8.9	0.5		0.2	-
Germacrene D	-	10.6	7.7	6.2	-	8.9	3.4	2.7	0.2	0.5
2-(2,4-hexadi ynylidiene)-1,6 -dioxaspiro [4,4]non-3-ene	-	-	-	21.4	-	-	-	-	-	-
α-Neoclovene	-	-	-	5.1	-	-	-	-	-	-
3,4-dihydro-2,2-dimethyl-2H-1-benzo pyrane	-	-	-	0.1	-	-	-	-	-	6.1
β-Humulene	-	tr	0.2	-	-	-	-	-	-	16.3
Ledene oxide	-	-	-	-	-	-	-	-	-	9.0
cis-α-Bisabolene epoxide	-	-	-	-	-	-	-	-	-	6.2
Spathulenol	12.5	-	-	-	1.3	0.48	tr	-	1.62	0.35
Caryophyllene oxide	6.5	1.5	2.5	1.25	2.4	-	1.3	-	-	-
T-Cadinol	19.6	-	-	-	0.8	-	-	-	0.86	-
α-Cadinol	5.6	-	-	-	1.5	-	0.71	-	1.39	-
T-Muurolol	-	-	6.8		1.4	-	-	-	0.2	-

From Table 3, the essential oils of Chry1 (*Chrysanthemum*. *macrocarpum* from Algeria), Chry10 (*C. morifolium* from China) and Chry4 (*C. indicum* from China), are quite different from the other reported species. Chry1 essential oil was characterized by T-cadinol (19.6%), spathulenol (12.5%),

caryophyllene oxide (6.5%) and α-Cadinol (5.6%), whereas Chry10 oil is mainly represented by 3,4-dihydro-2,2-dimethyl-2H-1-benzopyran (6.1%), β-humulene (16.3%), ledene oxide (9.0%) and cis-Z-α-Bisabolene epoxide (6.2%). However, 2-(2,4-hexadiynylidiene)-1,6-dioxaspiro[4,4]non-3-ene (21.4%), α-neoclovene

(5.1%) and *cis*-verbenol (21.7%) are found to be the major constituents of Chry4 essential oil. Myrtenol (54.8%) is the major component in the essential oil of Chry2 (*C. yoshinaganthum* from Japan), when T-Muurolol (6.8%) is mainly found in Chry3 oil. The essential oil of C. *viscidehirtum* from Morocco (Chry5) is mainly constituted by limonene (21.8%) and *β*-farnesene (25.0%), the first is a major component (20.9%) of the the essential oil of Chry9 (*C. trifucatum* from Tunisia) while the second was a major constituent (8.9%) of the essential oil of *C. coronarium* from Tunisia (Chry6). In addition, the latter is mainly characterized by *cis* and *trans*- chrysenthenyl acetate (21.8%, 12.8%) respectively, whereas Chry8 (C. *coronarium* from Spain) and Chry9 oils share main components, α-pinene (14.8%, 5.3%) and β-pinene (9.9%, 8.8%), respectively. ϒ-Terpinene (19.1%) is mainly detected only in Chry9 oil. Borneol (18.3%) and isobornyl acetate (10.9%) characterize particularly the essential oil of Chry7 (*C. indicum* L. from Hubei, China), also mainly represented by 1,8-cineole (30.4%) and camphor (23.5%). The two latters are together mainly found in Chry3 (23.0%, 14.7%), Chry4 (4.9%, 21.7%) and Chry7 (30.4%, 31.5%) essential oils, respectively. From this table, we conclude that there is not a chemomarker of chrysanthemum oils.

From table 4, the couple α-bisabolol oxide B/α-bisabolol oxide A (7.0%, 6.6%, 4.6%)/ (21.5%, 53.6%, 10.2%) seems to be a chemotype of *Matricaria* oils from Iran, Mat1 (*M. chamomilla* L. from Neyshabur, Khorasan-Razavi), Mat3 (*M. recutita* L. from Isfahan), and Mat5 (*M. chamomilla* L. from Golestan), respectively while α-Bisabolol (5.2%, 56.9%, 7.3%) was found to be mainly common to essential oils of Mat1, Mat2 (*Matricaria chamomilla* L. from Fars province, sub-central of Iran) and Mat5, respectively. In addition, the latter was also characterized by guaiazulene (10.6%), *α*-farnesene

Table 4 Major components (≥ 5%) of essential oils of *Matricaria* species **(Mat1-Mat5)**

Compounds*	**Percentage %**					
	Mat1 [37]	**Mat2** [38]	**Mat3** [39]	**Mat4** [10]	**Mat5** [40]	**Mat6** [41]
Myrcene	-	-	-	-	tr	50.6
cis-*β*-Farnesene	-	7.1	1.2	-	24.2	4.2
trans-*β*-Farnesene	5.2	-	1.2	-	-	-
α-Farnesene	-	-	-	-	8.7	0.1
Spathulenol	9.4	0.2	1,32	19.4	0.4	0.1
Geranyl isovalerate	-	-	-	8.2	-	8.3
α-Bisabolol oxide B	7.0	-	6.6	-	4.6	0.1
α-Bisabolone oxide A	10.0	2.2	-	-	0.2	0.3
α-Bisabolol	5.2	56.9	-	-	7.3	Tr
α-Bisabolol oxide A	21.5	-	53.6	-	10.2	-
Bisabolone oxide	-	-	29.9	-	-	-
α-Cadinol	-	-	-	12.9	-	-
Isochrysanthemic ethyl ester	-	-	-	26.5	-	-
trans-Farnesol	-	15.6	-	-	-	-
Guaiazulene	-	4.24	-	-	10.6	-
En-yne-dicycloether	-	-	-	-	-	26.9

(8.7%) and *cis*-*β*-farnesene (24.2%). However, *trans*-*β*-farnesene (5.2%) was mainly found in the essential oil of Mat1 whereas *trans*-farnesol (15.6%) was mainly found in the essential oil of Mat2.

In another hand, the compositions of essential oils of Mat4 (*M. pubescens* from Algerian Sahara) and Mat6 (*M. matricariodes* from Estonia), which share the major component geranyl isovalerate (8.2%, 8.3%, respectively), are different from others *Matricaria* essential oils. The first one is also characterized by the main presence of spathulenol (19.4%), mainly found in Mat1 oil (9.4%), α-cadinol (12.9%) and isochrysanthemic ethyl ester (26.5%) while myrcene (50.6%) and en-yne-dicycloether (26.9%) are manly exclusive to the essential oil of Mat6.

On the basis of this bibliographic investigation, among the species related to the famous saharian herbs called "Guertoufa", belonging to the genus *Cotula* (*C. anthemoides*, *C. cinera*), *Chrysanthemum* (*C. macrocarpum*, *C.*

fuscatum, *C. trifrucatum*, *C. derticolum*) and *Matricaria* (*M. pubescens*), used in Algerian Sahara in "Ramadhan" soup and as hot drinks flavours, only five essential oils, Cot2 (*C. anthemoides*), Cot3 (*C. cinera*), Chrys1 (*C. macrocarpum*), Chrys 9 (*C. trifrucatum)* and Mat4 (*M. pubescens*) have been reported. Remarkably, the essential of Chrys 9 "Guertoufa safra" did not share any major component with other "Guertoufa" herbs (tables 2-4). The first couple Cot2/Cot3 share santolinatriene (13.0%, 7.2%), thujone (12.9%, 41.4%) and camphor (27.4%, 5.5%) whereas α-cadinol (5.6%, 12.9%) is shared by the second couple Chrys1/Mat4. However, spathulenol (24.7%, 12.5%, 19.4%) is a chemotype of the triplet Cot3/Chrys1/Mat4 (Table 5).

Table 5 Shared major components in essential oils of "Guertoufa herbs"

Compound	**Cot2** *"Guertoufa"* [28]	**Cot3** *"Guertoufa"* [28]	**Chrys1** « Guertoufa bayda » [9]	**Mat4** « Guertoufa » [10]
Santolinatriene	13.0	7.2		
Thujone	12.9	41.4		
Camphor	27.4	5.5		
Spathulenol		24.7	12.5	19.4
α-Cadinol			5.6	12.9

Conclusion

Through our investigation of chemotypes of essential oils of *Cotula*, *Chrysanthemum* and *Matricaria*, it appears that santolinatriene (13.0%, 7.2%), thujone (12.9%, 41.4%) and camphor (27.4%, 5.5%) are mainly common to essential oils of *Cotula anthemoides* growing at Tinguentourine, Ain Amenas, Algeria (Cot2) and *Cotula cinera* from Zagora, Morocco (Cot3), respectively. No chemomarker has been detected in essential oils of *Chrysanthemum* or *Matricaria* but α-pinene (0.2%-14.8%), *β*-pinene (0.1%-9.5%), limonene, (0.2%-21.8%), 1,8-cineole (4.9%-30.4%), camphor (0.9%-29.2%), *β*-farnesene (0.2%-25.0%) and germacrene D (0.2%-10.6%), for the first genus, and spathulenol (0.1%-19.4%), *α*-bisabolol oxide B (0.1%-7.0%), *α*-bisabolol (5.2%-56.9%) and *α*-bisabolol oxide A (10.2%-53.6%), for the second, are found to be shared, at least, by two species of each genus. Regarding the essential oils compositions of "Guertoufa" herbs (Cot2, Cot3, Chrys1, Chrys9, Mat4), the couple Cot2/Cot3 share the major components santolinatriene, thujone and camphor whereas α-cadinol is mainly shared by the second couple Chrys1/Mat4. Spathulenol may be considered as a chemotype of the triplet Cot3/Chrys1/Mat4). The essential of Chrys 9 "Guertoufa safra", which is morphologically different (whole yellow), did not share any major component the four other essential oils. Through these results, we can not justify the same local name "Guertoufa" of these five plants, only on the basis of their essential oils compositions but we can speculate that this common name may be related to shared therapeutic effects due to other secondary metabolites.

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