



## **Influence of Solar Drying on the Phytochemical Screen of *Equisetopsida Asterales***

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### **Authors' contributions**

*This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.*

### **Article Information**

DOI: 10.9734/CJAST/2019/v34i330133

#### **Editor(s):**

(1) Dr. Santiago Silvestre, Associate Professor, Telecommunications Engineering, Universitat Politècnica de Catalunya, Spain.

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Complete Peer review History: <http://www.sdiarticle3.com/review-history/18571>

**Received 17 February 2015**

**Accepted 06 July 2015**

**Published 04 April 2019**

**Review Article**

### **ABSTRACT**

We are interested by studying the influence of two drying methods: in the shade and solar energy, on phytochemical composition. For this, we choose the rhizomes of the plant *Equisetopsida Asterales*, a species that can be found in the poor soil of Provence and Corsica. It prefers dry, sunny places in the Mediterranean. It is native to Southwest Asia, the East and spread in North Africa (Algeria, Morocco, Tunisia, Libya), Australia and the Americas, and Europe (Greece, Italy, France, Portugal, Spain). *Equisetopsida Asterales* rhizomes are used in Algeria as a traditional cream, which contributes to the disappearance of scars generated by burning. The antimicrobial properties of medicinal and aromatic plants have been known since antiquity. However, it was not until the early 20<sup>th</sup> century that scientists are beginning to show interest. It is known that the treatment of microbial infection is mainly based on the use of antibiotics, several work is devoted to the study of the antimicrobial power of essential oils.

After some experiences in this work, the important result was obtained, time of *Equisetopsida Asterales* by solar energy is better than shade; and yield of essential oil extracted is better from the rhizomes dried by solar energy.

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**Keywords:** Solar energy drying; shade drying; plant; antimicrobial activity; drying time; yield; essential oils.

## 1. INTRODUCTION

Many food and organic products are not always available during the seasons; several solutions are proposed to overcome this handicap greenhouse cultivation, preservation by freezing and drying. The use of the latter proves a simple, safe and adequate solution for a large number of products. Drying is still one of the main methods of preserving agricultural products [1]. It is a step in the processing of certain products. It is used both in rural areas, in the industrial world, but also in the pharmaceutical world through medicinal and aromatic plants [2,3] Designed for extraction of water from solid, semisolid or liquid by evaporation. This requires a heat source. Several techniques can be used to dry and preserve the harvest: The field drying in the sun, drying cabinets, etc. The drying process is based on the intervention of mass transfer (water) and also energy (heat) to evaporate the water present in the air [2], and the air charge the same amount of water. These exchanges take place because of differences in temperature and humidity around the product [4], given existence of different drying methods and the biological sensitivity of plants and their essential oils.

## 2. MATERIALS AND METHODS

### 2.1 Plant

*Equisetopsida Asterales*, a plant with large heads (3 cm wide by 3-4 long), lonely at the top of the stem and branches, globular and ovoid, much shorter than the achenes egret (approximately 2 times ) plant 20-60 cm, simple rod backward little or rowler. Leaves glabrous or pubescent, strongly veined, oval or lanceolate storyteller, lower stalked, toothed or lyres - pinnatifid, sessile above - amplexicaul or gear - thorny. Involucre's external bracts, hair- pectinate, unarmed, internal to Appendix fimbriated, blue petals. Wholesale achenes subglobose or obscurely tetragones, smooth and white (Quezel and Santa, 1963). (Fig. 1).

Harvesting of the plant was carried out in March and June of 2011, the harvest was made by dry and warm after sunrise to avoid wetting the plant. Plants wet with rain or dew require more drying time, deteriorate, moldy, ferment and lose, anyway, any therapeutic value [5,6]. During sampling of the samples, the healthiest feet, free from any evidence of insects and molluscs are

retained, the diseased parts, faded and damaged are removed [7]. *Equisetopsida Asterales* samples were harvested Baghlia is located 50 km from the Boumerdes (Fig. 2). Longitude: 3° 51 '25" Latitude: 36° 49' Altitude: 36 meters.

### 2.2 Drying

Apart from plants that use fresh, it is necessary to dry those that wish to keep. In our study, two different drying methods are followed: the first is drying in the shade in the open air and away from the light, and the second is the solar drying using a solar dryer indirect type.

### 2.3 Preparation of Plant Material

The rhizomes are washed, scrubbed, peeled and cut into thin slices before being dried [8,7]. (Fig. 3). Material is transported as quickly as possible places for drying away from light and dust and avoid wilting, fermentation and loss of active ingredients caused by heat.

#### 2.3.1 Shade drying

The shade drying is performed at the Research Laboratory of Food Technology, Faculty of Engineering Sciences (ISP), Boumerdes. The raw material is spread thinly on screen (80 × 50 cm) (Fig. 4), made in the carpenter, and vented to the air and away from light and heat. The slats are turned frequently to ensure even drying. This step was conducted over 13 days at a temperature between 18-24°C. During this period, we followed the change in water loss from rhizomes.

#### 2.3.2 Solar drying

The measurements are carried out at the site of the development unit of solar equipment (UDES) located in Bou-Ismaïl-Tipaza, Algeria: Latitude 36° 39 ', Longitude 2° 40', altitude 10 meters.

They concern two days from March 23-24, which are characterized by a clear and sunny weather. By using an indirect solar dryer type (Fig. 5).

The solar radiation is collected by absorbent surfaces (sensors) that heat a heat transfer fluid (air) carried in an enclosed space (drying chamber) in contact with the product to dry [9]. After preparation of the plant material comes the drying operation is, first, to spread on the trays of the dryer (Fig. 6).

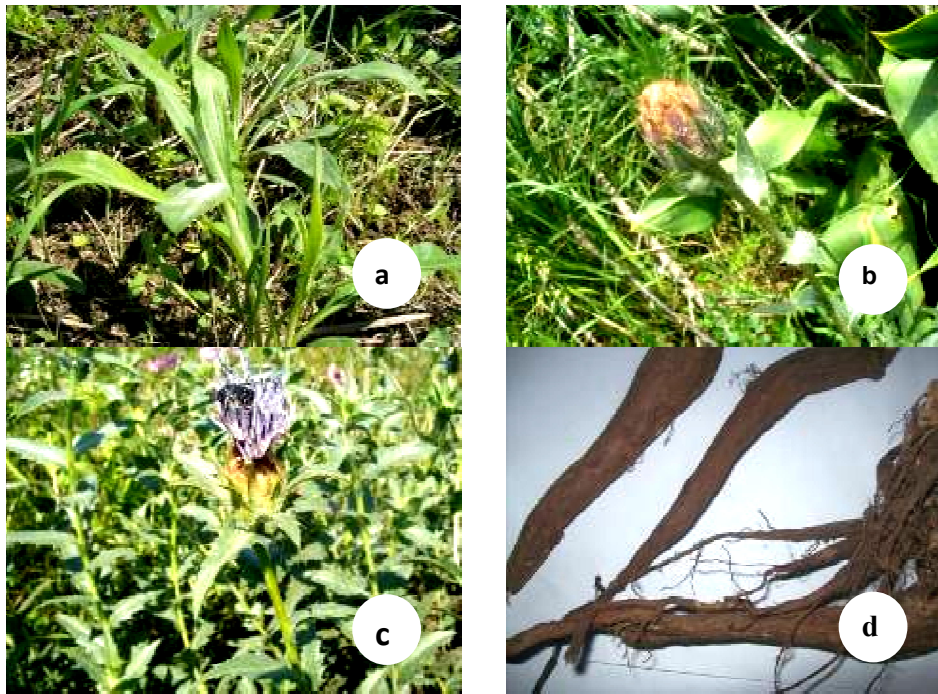


Fig. 1. The plant *Equisetopsida Asterales* pushing Baghlia  
a-leafy stem; b-flower not blown; c-blossoming flower; d-rhizomes with roots



Fig. 2. Administrative Map of Boumerdes amount Station harvest (DPAT, Boumerdes)

The velocity of the air entering the dryer is measured by an anemometer, remained substantially constant throughout the experiment. Its value varies between 1.2 and 2 m / s. The average temperature and radiation are respectively:  $T_m = 45^\circ\text{C}$  and  $HS_T = 653 \text{ W/m}^2$ .



Fig. 3. Preparation of rhizomes for drying



Fig. 4. Drying rhizomes shadow on screen



Fig. 5. Experimental setup: Indirect solar dryer



Fig. 6. Drying chamber rhizomes with three screens

### 2.3.3 Drying kinetics

To determine the loss curves relative humidity and drying rate of rhizomes *Equisetopsida Asterales* in the shade and in the indirect solar dryer, weighing are performed throughout the drying process: every 24 hours for rhizomes dried in the shade, and every half hour for those dried by solar energy, to stabilize the mass (end of the drying operation). The initial moisture content is derived at the end of the drying operation, by the difference between initial and final weight of the rhizome. It is understood that the dried rhizomes keep after drying, some evaluated by baking at  $105^\circ\text{C}$  for 12 hours humidity.

The relative humidity of the product  $W$  is given by the following relation [10].

$$W = \frac{\text{water mass content}}{\text{product mass}}$$

Drying speed  $V$ , derived from moisture loss, is given by the following relationship:

$$V = \frac{\Delta W}{\Delta t}$$

### 2.3.4 Grinding and conservation

After cutting and drying the rhizomes, they are then crushed in a clean mortar with finely ground using an electric shredder.

The powders obtained (Fig. 7) are kept away from light and moisture in sterile glass vials sealed.

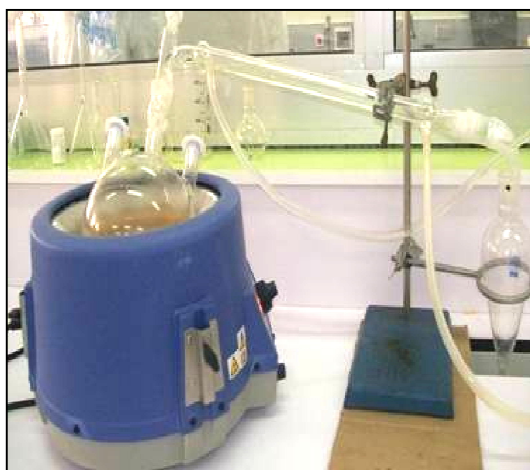


**Fig. 7. Powder of dried rhizome**  
Left: In the shade, Right: Solar energy

### 2.3.5 Extraction of natural substance *Equisetopsida Asterales*

#### 2.3.5.1 Extraction of essential oil

Extraction of essential oils from dried in the shade (SD) and solar energy (SE) rhizome is made by steam distillation with five tests per sample (Fig. 8).



**Fig. 8. Installation of the steam distillation**

#### 2.3.5.2 Calculation of performance

Essential oil content, expressed as g per 100 g of the distillate of dry matter, is given by the following relationship: [11].

$$R_{EO} = M_{EO} / M_D \cdot 100$$

$R_{EO}$ : essential oil yield (%).

$M_{EO}$ : Mass of collected oil (g).

$M_D$ : mass of dry material used (g).

#### 2.3.5.3 Extraction of the polar fraction in reflux condenser (lyophilized)

A 5 g of powdered rhizome (SD and SE) placed in an evaporation flask were added 50 ml of distilled water, the mixture is brought to boiling under cooling with magnetic stirring for half an hour (Fig. 9). After cooling, the mixture is filtered and the filtrate was lyophilized, dry extract is called freeze-dried [12].

### 2.4 Calculation of Performance

The yield is the ratio between the mass of the dry residue obtained after lyophilization, and the mass of the plant material used, expressed by the following relationship:

$$R_{Ly}\% = M_{RS} \cdot 100 / M_{VS}$$

$R_{Ly}$ : Yield lyophilisate (%).

$M_{RS}$ : the mass of the dry residue obtained after lyophilization.

$M_{VS}$ : the mass of dry plant.

### 2.5 Chromatographic Analysis of the Essential Oil of *Equisetopsida Asterales*

#### 2.5.1 TLC

Thin layer chromatography is a rapid physico-chemical control method which the absorbent or stationary phase consists of a thin, uniform plate of silica in our case. The eluent or mobile phase of migrates to the surface of the plate by capillary action. It is an analytical method which, at each stage of separation can: Follow the efficiency of extraction with different solvents; Make the best choice of solvent elution columns Check the purity of the isolated products [13].

In our work the observation spots is done under UV at 365 nm and the revelation is made with a solution of  $KMnO_4$ . The frontal reports ( $R_f$ ) for each essential oil is calculated as follows:

$$R_f = \frac{\text{Distance traveled by the substance}}{\text{Distance traveled by the solvent}}$$

### 2.5.2 Gas Chromatography coupled with Mass Spectroscopy (GC-MS)

The chemical composition of essential oils is determined by the technique of gas chromatography coupled with mass spectrometry (GC / MS).

### 2.6 Evaluation of Biological Activities of *Equisetopsida Asterales*

One of the most widely used to assess the antimicrobial activity of various extracts of medicinal plants is the technical diffusion method agar quoted [14]. The principle of diffusion method on agar medium it is to measure the in vitro susceptibility of a germ vis-à-vis a given substance, which must be

stable and retains its activity during the test [15].

The strains used in this work are widely encountered in various pathologies in humans. Microbial strains tested, their sources and references are shown in the Table 1.

To evaluate the antimicrobial activity of lyophilisate obtained from the rhizomes of *Equisetopsida Asterales* dried in the shade or solar energy, we followed the method of agar diffusion. This method is performed by depositing disc filter paper soaked lyophilized test on an agar medium previously poured into Petri dishes and inoculated with a microorganism. Evaluation of antimicrobial activity of a lyophilisate made by measuring the inhibition zone's diameter, it result a clear halo around the absorbent disk, after incubation.



Fig. 9. Extraction device by refrigerant under reflux

Table 1. Microbial strains tested

Microorganismes	Gram	Origin	Reference	
<b>Bactéries</b>	<i>Escherichia coli</i>	-	I.P.A	ATCC 25922
	<i>Staphylococcus aureus</i>	+	I.P.A	ATCC 25923
	<i>Pseudomonas aeruginosa</i>	-	I.P.A	ATCC 27893
	<i>Klebseilla ornithinolytica</i>	-	Hôpital de Tizi-uzou	Isolé à partir d'un patient.
	<i>Klebseilla pneumoniae</i>	-	I.P.A	N 828
	<i>Streptococcus faecalis</i>	+	I.P.A	ATCC 29212
<b>Champignons</b>	<i>Saccharomyces cereviceae</i>	/	IPA	ATCC 549
	<i>Fusarium sp</i>	/	E.N.S.A	Isolé à partir du sol

### 3. RESULTS AND DISCUSSION

#### 3.1 Drying

Experimental temperatures are indirect solar dryer by an automatic data recorder, are shown in Fig.10 (Solar collector) and Fig. 11 (drying chamber).

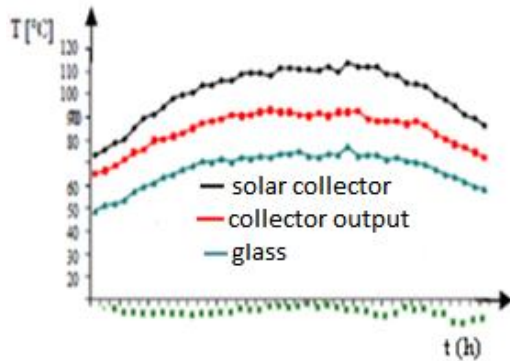


Fig. 10. Temperature in solar collector

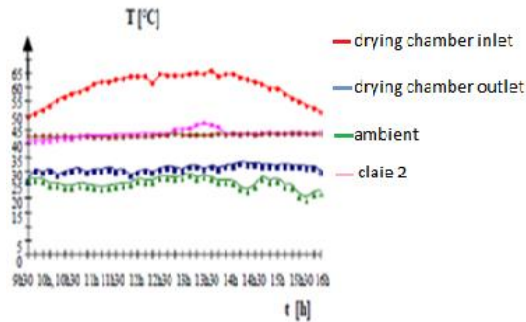


Fig. 11. Temperature in drying chamber

In terms of absorbing the surrounding temperature reaches values of 110°C. While in

the drying chamber, the temperature at the screens is almost constant and its value is equal to 45°C.

#### 3.1.1 Drying kinetic

##### 3.1.1.1 Humidity

The moisture content of fresh rhizomes of *Equisetopsida Asterales* rate (SD) is 65.32%, and 67.08% for rhizomes (SE) (Fig. 12). Both values are similar and comparable to that of fresh ginger (80%) [16], those of verbena (67%) and laurel (64%) [10].

##### 3.1.1.2 Characteristic drying curves

The loss curves relative humidity and drying rate of rhizomes *Equisetopsida Asterales* dried in the shade and solar energy are presented below (Fig. 13 and Fig.14).

Different drying curves obtained show the decreasing speed of the water content and drying rate according time. There are two stages of drying:

- The first drying step has a steep slope which indicates that the water loss is rapid *Equisetopsida Asterales* rhizomes, the free water which is more than half the total amount of water contained in the rhizomes is removed during this phase.
- The second phase of drying has a smaller slope indicates a slow water loss in this phase out the water the product is increasingly linked, and more difficult to evaporate. The evaporation of the water requires a lot of energy and time which is slowing the drying rate during this phase.

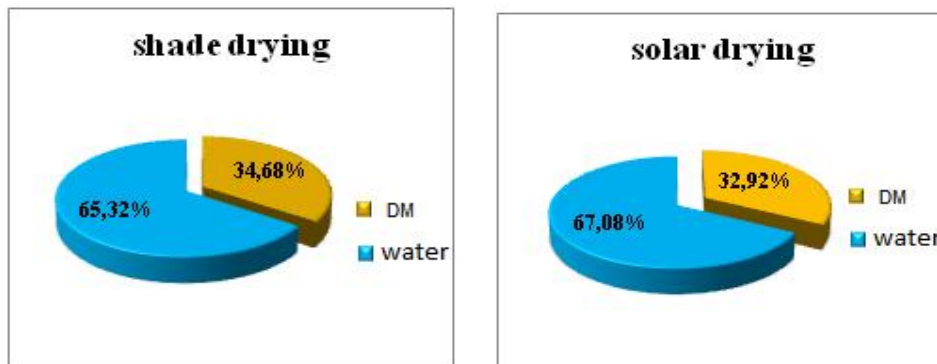


Fig. 12. Humidity fresh rhizome *Equisetopsida Asterales*

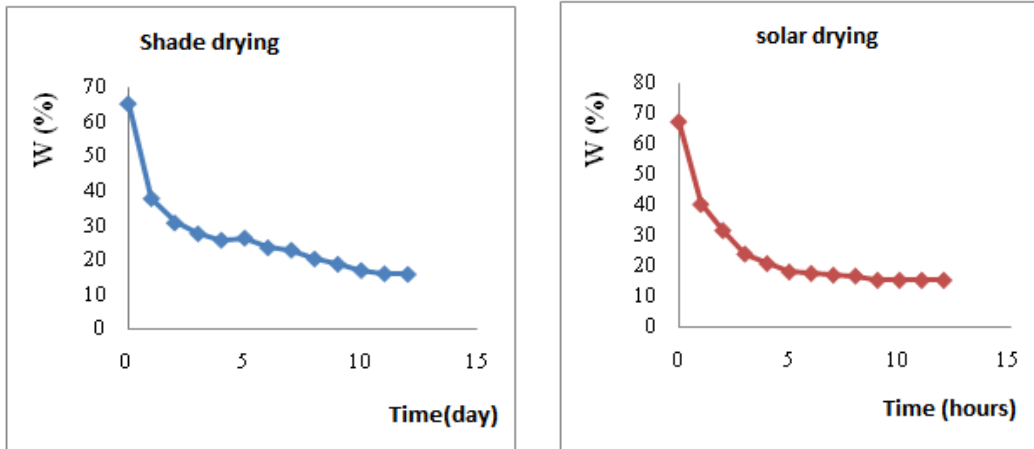


Fig.13. Evolution of water content of *Equisetopsida Asterales* rhizomes according drying time

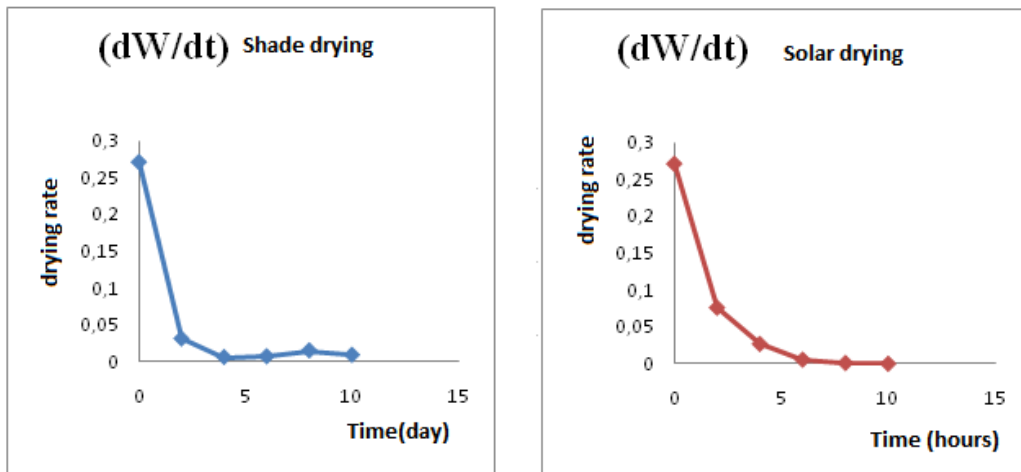


Fig. 14. Evolution of drying rate of *Equisetopsida Asterales* rhizomes according drying time

We compared the color and smell of rhizome powder (SD) and (SE), it was observed that the powder shade drying lost the original color (becomes darker) and the characteristic odor of the rhizome by against SE powder kept the color and odor characteristic of the rhizome, indicating that the drying method affects the quality of the dried product [17].

From these results we see that the traditional method of drying (drying in the shade) required a long exposure time (10 days), drying is sometimes interrupted due to adverse weather conditions, rehydration nights and contamination multiple, resulting in very poor quality; against drying by solar need a short drying time (10 hours), with a better quality of dried product.

### 3.2 Essential Oil Yield

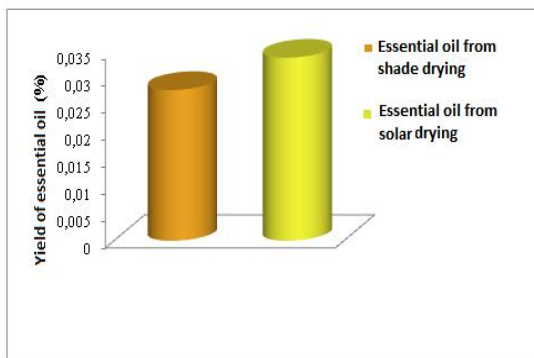
Extraction of essential oils from *Equisetopsida Asterales* rhizomes SD and SE was performed by steam distillation oils.

Returns based on the total weight of crushed (100 g) indicate that the content of the rhizomes in shade drying essential oils is 0.028% against 0.034% for SE (Fig. 15).

The essential oil content found in the dried rhizomes of *Equisetopsida Asterales* solar energy is slightly higher than that found by drying in the shade, the two values are considered low but comparable to the values obtained by Sarolgu et al. (2006) some who were low, 0.012% for *A. Anthemis* species tomentosa,



0.028% for *A.weneri*, 0.047% for *A.altissima*, 0.011% for *A.* and 0.096% for *melanolepis A.tinctoria*. so the performance of *A. altissima* is 0.05% (Javidnia et al. 2004).



**Fig. 15. Yield of essential oil according drying mode**

Relatively higher values by [18] from different parts of *A. xylopoda*: a yield of 0.54% from the flowers, 0.38% from the leaves. These differences are due to several factors: the technique for obtaining the environmental factors, the plant species itself, the plant organ, the time and place of collection, stage of growth and culture conditions plant [19] According [11], the essential oil content of *Thuja Barbary* leaves increases significantly with method and drying time.

### 3.2.1 Performance lyophilisate

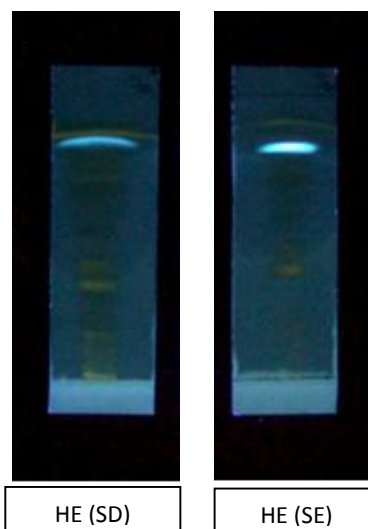
Extraction of polar fraction under reflux condenser given to a yield of 32% of lyophilized N and 42% for the SE. Both values are higher than those obtained by [12] who worked on *Tamarindus indica*, the value is estimated at 23%.

## 3.3 Analysis of Essential Oils

### 3.3.1 Thin Layer Chromatography (TLC)

The development of the method for TLC begins with the choice of the mobile phase, three solvent systems were used, after revelation by  $KMnO_4$  was selected that gave a better separation: hexane / ether / formic acid (100/50/1).

The revelation of the task is performed by observation under UV light at 365 nm (Fig.16), and revelation by  $KMnO_4$  (Fig. 17).



**Fig. 16. Revelation under UV light at 365 nm**



**Fig. 17. Revelation by  $KMnO_4$**

In a TLC compounds appear as round or oval tasks, 7 spots are obtained for both ET.

Tasks A, B, C, D, E being close could correspond to pure compounds, the F task is phosphorescent under ultraviolet light at 365 nm, which confirms the presence of phenolic acids, and the task J Dark yellow color seen its size may correspond to a set of compound.

### 3.3.2 Gas Chromatography coupled to Mass Spectrometry (GC / MS)

Essential oils (SD) and (SE) were analyzed by GC / MS to meter showed a possible difference

in the quality and quantity between the two terms.

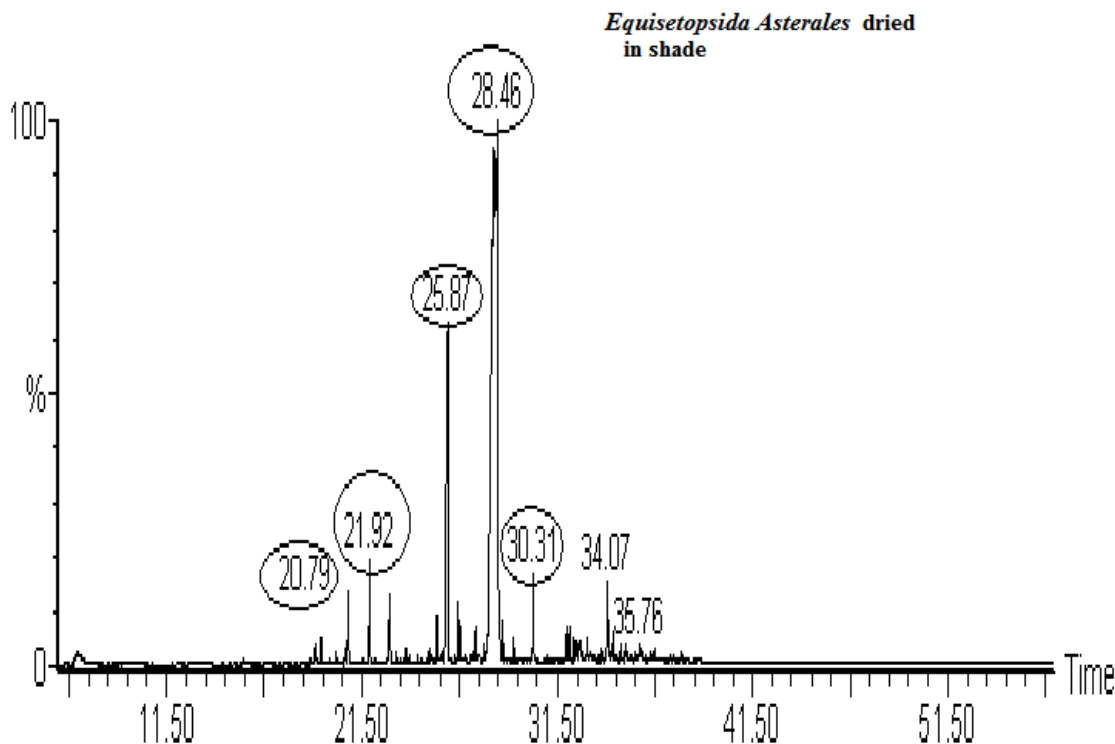
*Equisetopsida Asterales* essential oils obtained by steam distillation from the rhizome of *Equisetopsida Asterales* dried in the shade and the dried by solar energy, are represented through the chromatograms illustrated in Fig.18 and Fig. 19.

As shown in Fig. 18 and Fig. 19, chromatographic profiles of the essential oils (SD) and essential oils (SE) look different from one another. We note first the presence of a larger number of peaks in essential oils (SE), which reflects its relative wealth of volatile compounds. Drying by solar would thus better impact on the quality of the oil as it seems to best preserve its constituents. Drying in the shade rather promote a considerable loss of these. This could be attributed to by a drying time (10 days to dry in the shade and 10 hours for drying by solar energy), and secondly to drying conditions (temperature, humidity) [20,19].

On the chemical composition of the two oils, this one was made by comparing the mass spectra of the chromatographic peaks considered, with those of the NIST 89 library. We were able to take a formal decision on the identity of all the peaks appearing in the chromatograms, except for those listed in Table 3.

Both oils are characterized mainly by the presence of such compounds *sesquiterpenes*: *b-Caryophyllene* (-) *caryophyllene oxide*, *b-b-farnesene* and *selinene*, these compounds are known in many species belonging to the *Asteraceae* family, such as *Chrysothamnus pulchellus* in the *sesquiterpenes* which represent 16.2% of the essential oil of the aerial part.

Recall that a large number of sesquiterpenes are common constituents of essential oils of plants and, as such, they may be involved in the pharmacological properties attributed to these volatile fractions [21].



**Fig. 18. Chromatogram of *Equisetopsida Asterales* essential oils dried in the shade**

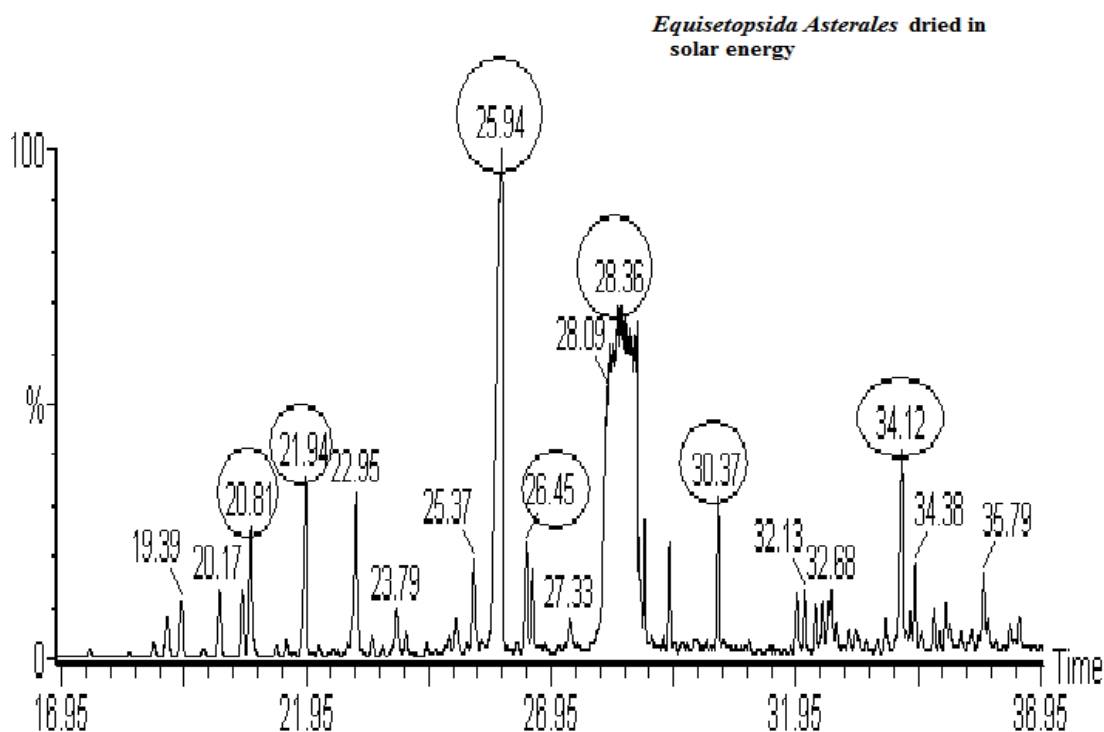


Fig. 19. Chromatogram of *Equisetopsida Asterales* essential oils dried by solar energy

### 3.4 Evaluation of the Antimicrobial Activity of Lyophilized *Equisetopsida Asterales*

For the evaluation of the antimicrobial potential of freeze-dried rhizome of *Equisetopsida Asterales* dried in the shade and solar energy, they were tested against several microorganisms, the results obtained are shown in Table 4.

From the results obtained, we note that:

- *E. coli* 11 mm <math>\varnothing</math> <math><14</math> mm SD, so sensitive 17 mm <math>\varnothing</math> <math><19</math> mm SE, very sensitive
- *P. aeruginosa*, *K. ornithinolytica*, *K. pneumoniae*, *S. faecalis*, *Fusarium sp* have a diameter of inhibition: 9 mm <math>\varnothing</math> <math><14</math> mm, so sensitive to both freeze-dried (SD and SE).
- *S. aureus* 9 mm <math>\varnothing</math> <math><14</math> mm N, so sensitive 15 mm <math>\varnothing</math> <math><19</math> mm SE, very sensitive
- *S. cerevisiae*: 9 mm <math>\varnothing</math> <math><14</math> mm SE therefore sensitive

Based on the above results, we note that the antimicrobial potency of lyophilized SE is higher than that obtained with the lyophilized SD for all strains tested. Overall, all strains tested were

sensitive to both freeze-dried used irrespective of the mode of drying done. However, with regard to the species *S. aureus*, there is a remarkable difference in the inhibitory activity of two freeze-dried according to the type of drying.

Table 2. Reports frontal spots obtained by the solvent system hexane / ether / formic acid

Task	Essential oils(SD)	Essential oils (SE)
	RF	
A	0,29	0,24
B	0,39	0,40
C	0,48	0,48
D	0,68	0,60
E	0,79	0,70
F	0,73	0,74
G	0,84	0,87

Indeed, it has a very sensitive profile when the rhizome is dried by solar energy. According to the literature, the sensitivity of *Staphylococcus aureus* strain ATCC can probably be explained by the sensitivity of Gram (+) to external environmental changes such as temperature, pH, and natural extracts, due to the absence the outer membrane.

**Table 3. Majority components of *Equisetopsida Asterales* essential oils**

Essential oils (SD)		Essential oils(SE)	
Compounds	Extraction time	Compounds	Extraction time
Trans caryophyllene	20,79	Caryophyllene	20,81
Trans B-Farnesene	21,92	Bicyclo	21,94
(-) Caryophyllene oxide	25,87	(-) Caryophyllene oxide	25,94
9, 12,15- Octatrien-1-ol (Z,Z,Z)	28,46	9,12,15- Octatrien-1-ol (Z,Z,Z)	28,09
B-Selinene	30,31	B-Selinene	30,37

**Table 4. Test results of the antimicrobial activity of freeze-dried SE and SD of *Equisetopsida Asterales***

Microorganismes ( $\emptyset$ )	Lyophilisat SD	Lyophilisat SE
	Inhibition diameter	Inhibition diameter
<i>E. coli</i>	11± 0,00	17 ± 0,00
<i>S. aureus</i>	13,5± 2,12	16,5± 2,12
<i>P. aeruginosa</i>	11± 0,00	12,5± 0,70
<i>K. ornithinolytica</i>	10,5± 0 ,70	11,5± 0,70
<i>K. pneumoniae</i>	10,5± 0 ,70	13± 1,41
<i>S. faecalis</i>	11,5± 0,70	12,5± 0,70
<i>Fusarium sp</i>	12± 0,00	13± 0,00
<i>S. cerevisiae</i>	/	11± 0,00

The results of the antifungal activity showed the ineffectiveness of lyophilized SD against mold *Saccharomyces cerevisiae* but with a zone of inhibition with interesting lyophilisate (SE), this difference can be explained by the multiple benefits of drying probably preserves product quality safeguarding large numbers of active principles extracted from the drying in the shade as shown by [22]. On the other hand, the method used to evaluate the antibacterial activity can affect the results. Thus, found that the diffusion method from wells on agar is more appropriate to study the activity of aqueous and organic extracts of *Euphorbia fusiformis* and hydro-ethanolic *Rhus coriaria* *Zataria multiflora* and that the method of agar diffusion. The lyophilisate of *Equisetopsida Asterales* is very active on the bacterial strain *Staphylococcus aureus* (diameter 16.5 lyophilisate SD) compared to other bacteria.

#### 4. CONCLUSION

The yield of dried *Equisetopsida Asterales* rhizomes essential oil in shade is 0.028% and by solar energy (0.0348%) is significant and can be profitable on an industrial scale.

*Equisetopsida Asterales* rhizomes are used in Algeria as a traditional cream, which contributes to the disappearance of scars generated by burning [23], not found in national or international market; and it available for non-dried plant just at May month, we see that burning can exist all time and all situation, so accessibility of this product was be necessary. From the results of our work, we have shown that solar drying gave a powder of better quality and quantity than that dried in the shade, which will promote the availability of this product in Algerian market and that can be marketed at a good price and even exported in its three forms: dry plant, powder and essential oil, as long as it is dried by means of a practical and available at the farm, operating with a free and clean energy (solar energy) and with solar drying time 24 parts less than SD time (time is money) [24]. By referencing to other work with other research that showed that the oil extracted from a process of drying, gives a better performance in chemical composition than those extracted from a plant not dried [22] and in our experience, we have inferred that the method of solar drying gave even better performance than any other mode. Antimicrobial activity has been demonstrated by both essential oils extracted by both methods and was more important to that obtained from solar drying than in the shade.

#### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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