



ISSN  
2217-5369  
(print version ceased in 2023)  
2217-5660 (online)

www.foodandfeed.fins.uns.ac.rs

# FOOD AND FEED RESEARCH

Journal of the Institute of Food Technology – FINS  
University of Novi Sad



UDK

Original research paper

<https://doi.org/10.5937/ffr0-63027>

## **PELARGONIUM GRAVEOLENS (L'HÉR) ESSENTIAL OIL, ANTIOXIDANTS, ANTICYANOBACTERIAL AND ANTIALGAL COMPOUNDS: A SOLAR-BASED EXTRACTION TECHNIQUE AND YIELD PREDICTION USING LINEAR REGRESSION**

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**Abstract:** Solar hydro-distillation (SHD) is presented as a new green technique to effectively extract the various phytochemicals from food by-products. To better understand the procedure, results, and advantages of such a green and sustainable source, the goal of this study was to compare the efficiency of SHD to extract essential oils from *Pelargonium graveolens* (L'Hér) while simultaneously releasing antioxidant compounds like polyphenols and flavonoids in the remaining phase of the solar still. The yields of the essential oils were 0.64 % and 0.60 % for SHD and conventional hydro-distillation (CHD), respectively. By using GC-MS analysis, 52 volatile components were identified. Citronellol (27.54 %–26.51 %), citronellyl formate (13.63 %–11.33 %), geraniol (11.94 %–10.97 %) and geranyl formate (8.31 %–5.84 %) represented the main components for SHD and CHD oils, respectively. For the extracts produced by SHD and maceration, total phenolic compounds (TPC), total flavonoid compounds (TFC), and antioxidant activity (AA) based on 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging activity were evaluated. The results showed that the SHD extract produced an extremely good extraction yield in TPC, TFC, and IC<sub>50</sub> (249.72 mg EAG.g<sup>-1</sup> DM, 205.88 mg EQ.g<sup>-1</sup> DM, and IC<sub>50</sub>=6.54 µg.mL<sup>-1</sup>, respectively). Moreover, HPLC-UV analyses showed conservation in the extract after the SHD process of some identified compounds such as tyrosol (31.71 mg.g<sup>-1</sup> DM), gallic acid (24.67 mg.g<sup>-1</sup> DM), protocatechuic acid (22.59 mg.g<sup>-1</sup> DM), and ferulic acid (21.04 mg.g<sup>-1</sup> DM). On the other hand, the essential oil and the extract prepared by SHD showed an important anti-cyanobacterial/anti-algal activity against the bacterial/algal strain tested *Microcystis aeruginosa* and *Chlorella sp.*, with growth inhibition diameters of 15.43 ± 0.32 mm, 16.73 ± 0.40 mm and 17.13 ± 0.35 mm, 25.96 ± 0.15 mm respectively for essential oil and extract. A positive linear correlation was observed between antioxidants, polyphenols, flavonoids and anti-cyanobacterial/anti-algal activity for solar extracts. Results showed that SHD is a good alternative for recovering bioactive compounds from the *Pelargonium graveolens* (L'Hér.) with potent antioxidant, and anti-cyanobacterial/anti-algal activity.

**Key words:** geranium, solar hydro-distillation, essential oil, total phenolics/flavonoids, antioxidant activity, *Microcystis aeruginosa*, *Chlorella*.

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## INTRODUCTION

*Pelargonium graveolens* is native to South Africa. It is a widespread plant and cultivated in Spain, Italy, Morocco, Reunion, Egypt and China; these last two countries are the greatest producers (Juliani et al. 2006). It is grown in many Mediterranean and subtropical regions where it is distilled for its essential oil (EO). *P. graveolens* is known for its beneficial properties for human health, due to its richness in essential oil and phenolic compounds (Ferraz, Pastorinho, Palmeira-de-Oliveira & Souza, 2022). The content of total phenolic compounds (TPC) in *P. graveolens* varies between 51.60 mg EAG g<sup>-1</sup> dry matter (DM) (Sompaga, Jyoth, Chekuri, Baburao, & Anupalli, 2016) and 142.71 mg EAG g<sup>-1</sup> DM (Ben ElHadj Ali et al. 2020) using methanolic maceration extract. It can even exceed 381.25 mg EAG g<sup>-1</sup> DM (El Aanachi et al. 2020).

Traditional maceration (Riahi et al. 2020) and supercritical extraction with CO<sub>2</sub> (Machalova, Sajfrtova, Pavela, & Topiar, 2015; Ponomareva & Molohova 2017) have been used in previous studies to extract essential oils and antioxidants from *P. graveolens* leaves. These methods have proven to be innovative and practical. Additionally, using a Clevenger-style apparatus, the volatiles produced by *P. graveolens* were traditionally recovered by hydro-distillation (Ali, Hassan, & Elgimabi, 2018; Ćavar & Maksimović, 2012). However, according to our knowledge, essential oils and antioxidants were extracted from *P. graveolens* leaves using the solar technique for the first time.

In 1986, Wolfgang Scheffler tested his collector for the first time in Kenya and India (Scheffler 2006), using a second reflector, a solar still, and a condenser to extract essential oils. Because the payback period for such a system is not more than two years, using it today presents an economically interesting opportunity (Jayasimha, 2006). The solar distillation method was previously employed to deodorize rosemary leaves using the same solar distillation device that was used in this work (Hilali et al. 2018). It was also used to extract orange peels' essential oils before applying an organic solvent to extract the bio-active components (Hilali et al., 2019).

The objective of this work was the use of solar hydro-distillation (SHD) for essential oils ex-

traction and deodorization of *P. graveolens* leaves by utilizing a solar-powered, environmentally friendly, and efficient process, and then measure the amount of total phenols, total flavonoid compounds and antioxidant activity in the remaining phase of the solar still, and to test the anti-algal/cyanobacterial potential of our essential oil and solar extract in solid medium on two strains (*Microcystis aeruginosa* and *Chlorella* sp.). Another objective was to compare the results obtained with those of a conventional system (CHD) based on butane gas and with the results of maceration of *P. graveolens* leaves with methanol.

## MATERIALS AND METHODS

### Plant materials

In 2023, *P. graveolens* leaves has been cultivated in Marrakech (31° 37' 46 N, 7° 58' 52 W), a city in the center of Morocco at an altitude of 466 meters. The plant was identified under number MARK-14579. To achieve constant mass, leaves were rinsed in water and allowed to air dry for 22 days at room temperature (37 °C) and atmospheric pressure (1atm) in the shade. After air-drying, the leaf's moisture content was 82.90%. The leaves were crushed, completely dried, and stored (under the same conditions) in plastic bags in a dry, dark area until needed.

(Moisture content %) = [(Initial mass of the plant – Final mass of the plant) / Initial mass of the plant] × 100

### Chemical products

The chemicals utilized in this study were the entire analytical grade. The Folin-Ciocalteu reagent, sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>), gallic acid, methanol, aluminium trichloride (AlCl<sub>3</sub>), sodium hydroxide (NaOH), and sodium nitrite (NaNO<sub>2</sub>) were obtained from Sigma-Aldrich, Darmstadt, Germany. Gallic acid, quercetin, and 2,2-diphenyl-1-picrylhydrazyl (DPPH) were procured from the Somaprole company in Casablanca, Morocco.

### The solar distillation system

The solar distillation system utilized in our earlier work was installed at the national center for water and energy studies and research (CNEREE) at Cadi Ayyad University in Marrakech, Morocco (31° 37' 46 N, 7° 58' 52

W) (Ezzarrouqy, Hejjaj, Idlimam, Ait, & Laila, 2022). This solar thermal equipment consists of a 10-m<sup>2</sup> fixed focus Scheffler concentrator, a secondary reflector, and a solar still (Fig. 1). To align the rotational axis of the concentrator and the earth, the rotating axes are adjusted to the relative local angle of latitude (31° 37' 46").

The available solar intensity, together with the optical and thermal performance of the solar distillation system, determines how much energy can be used for distillation.

The performance of the solar distillation system is based on solar still absorption and the optical efficiency of the primary and secondary

reflectors (Afzal, Munir, Ghafoor, & Alvarado, 2017). Glass mirrors with an 85 % reflectivity make up the surface of the primary reflector. A small electronic photovoltaic (PV) plate and a daily and seasonal mechanical solar control system are installed within 10 m<sup>2</sup> parabolic reflectors.

A data logger was utilized to connect thermocouples and pyranometer, which were used to measure temperature and solar irradiance, respectively, to a computer. When daily solar radiation is low due to dense cloud cover, a conventional heating system can be used, such as a butane injector placed at the bottom of the solar still.

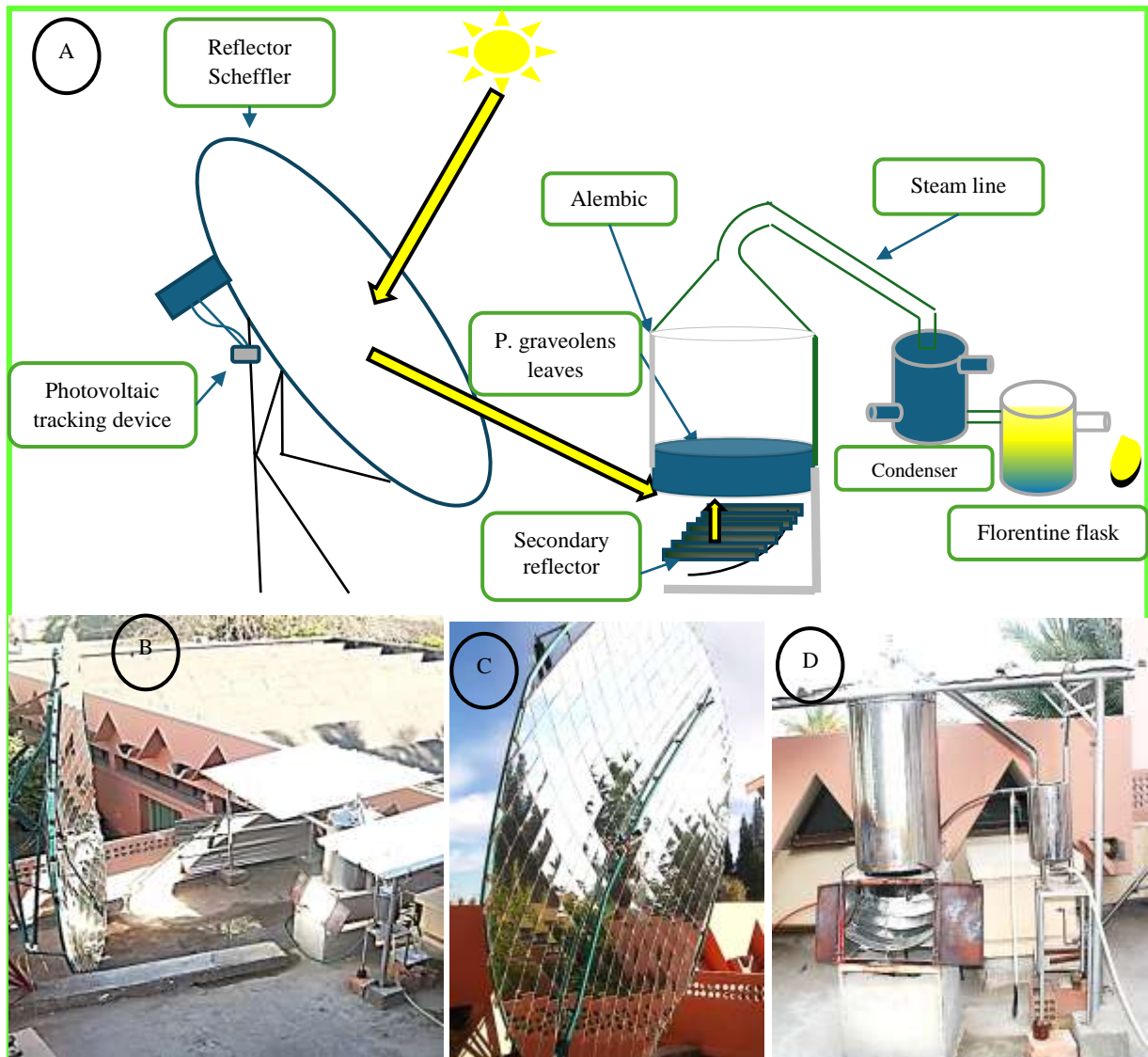


Figure 1. System for solar distillation. (A) The schematic apparatus; (B) A solar reflector; (C) A solar device; and (D) A distillation unit

## Experimental methods

### Solar hydro-distillation

500 grams of powdered *P. graveolens* leaves were distilled for four hours in 10 L of water. Boiling water releases steam that is loaded with essential oils, which are collected after condensation. When there is no more essential oil left to retrieve, the extraction process is finished. After being dried with anhydrous sodium sulfate, the essential oil was stored at 4 °C. The experiment was repeated three times.

### Conventional hydro-distillation

The conventional system was operated under identical conditions for an accurate evaluation. In this experiment, butane gas was used to heat the water, and the resulting steam was charged with essential oil before being transformed into liquid in the condenser and collected in a Florence flask. When there is no more essential oil left to retrieve, the extraction process is finished. The essential oil was dried with anhydrous sodium sulfate and then kept at 4 °C. The experiment was repeated three times.

### Preparation of extracts using maceration

One gram of crushed and dried *P. graveolens* leaves were macerated in 20 milliliters of 96% methanol or distilled water for a duration of four hours. A rotary evaporator with a lower pressure setting was used to remove the extraction solvents from the filtrate. The concentrated extracts were kept in a freezer at 4 °C until they were needed. The experiment was repeated three times.

### Total phenolic compounds (TPC) determination

Using gallic acid as a reference, to determine each extract's total phenolic compound (TPC) concentration, the Folin-Ciocalteu reagent was utilized (Box, 1983). To 0.1 mg.mL<sup>-1</sup> of the extract, 0.5 mL of the Folin reagent was added. 2 mL of Na<sub>2</sub>CO<sub>3</sub> (20 %) were added after 5 minutes. After that, the mixture was let to sit at room temperature in the dark for 30 minutes.

Utilizing a Selecta spectrophotometer, the absorbance at 750 nm was determined. The total phenolic component content was expressed as milligrams of gallic acid equivalent per gram of dry vegetable matter (mg EAG g<sup>-1</sup> DM). The experiment was repeated three times.

### Total flavonoid compound (TFC) determination

The aluminum trichloride method was used to determine each extract's total flavonoid compound (TFC) concentration (Dewanto, Xianzhong, Adom, & Liu, 2002). 250 µL of the diluted extract (0.1 mg mL<sup>-1</sup>) were mixed with 75 µL of a 7 % NaNO<sub>2</sub> solution. The combination was then given 150 µL of a fresh solution of 10 % AlCl<sub>3</sub> after 6 minutes of room temperature incubation. After 5 minutes at room temperature, 0.5 mL of sodium hydroxide (NaOH, 1 M) was added to the mixture.

With distilled water, the volume was completed to 2.5 mL. At 510 nm, the preparation's absorbance was quantified. The same operating conditions were used to create a calibration curve utilizing quercetin. TFC contents were given as milligram equivalent of quercetin per gram of dry vegetable matter (mg EQ g<sup>-1</sup> DM). The experiment was repeated three times.

### Total antioxidant capacity (TAC) determination

When in its reduced form, the free radical DPPH (2,2-diphenyl-1-picrylhydrazyl) is yellow, and when in its oxidized form, it is violet (Parejo et al. 2002). The DPPH radical is reduced and turns yellow in the presence of antiradical agents, ascorbic acid was used as positive control, and methanol as negative control. To calculate the percentage of DPPH radical inhibition, the extract's antiradical power was measured using its absorbance at 517 nm (Brand-Williams, Cuvelier, & Berset, 1995). Typically, 25 mg of DPPH were dissolved in 100 mL of methanol and then diluted 1:10 using the same solvent.

The extract was added to 3.9 mL of methanolic DPPH solution in a 100 mL container. The mixture was kept out of direct sunlight and at room temperature for half an hour. The percentage of antioxidant activity (I) is calculated according to the following formula:

$$I (\%) = [(Abs \text{ blank} - Abs \text{ simple}) / Abs \text{ blank}] \times 100$$

where Abs blank: is the DPPH absorbance at time zero prior to the addition of sample;

Abs simple is the test sample's absorbance following a 30-minute incubation period.

## Essential oil analysis

The volatile components of essential oils were determined using gas chromatography linked to mass spectrometry (GC-MS) investigation conducted on an Agilent 7890A gas chromatography connected to a 5975C mass spectrometer utilizing the Wiley 6, NIST 02, and ERIMI databases. The HP5 nonpolar column (30 m × 0.25 mm × 0.25 μm) was employed in this analysis. The following parameters were used to obtain the MS spectra: helium carrier gas with a flow rate of 1.25 mL/min; a split of 1:100; an injection volume of 0.1 μL; and an injection temperature of 256 °C.

The temperature was programmed at 50 °C for three minutes, then 4 °C/min until 100 °C, held for two minutes; then 6 °C/min until 265 °C, then 15 °C/min up to 300 °C, maintained for five minutes.

The solvent used for rinsing was ethyl acetate/acetone. The Kovats retention index used to characterize the acquired chromatographic peaks and computed based on the C7 to C30 n-alkanes. By contrasting their mass spectra with those of standards chemicals, the volatile content of *P. graveolens* essential oil was identified. The experiment was repeated three times.

## Identification of phenolic compounds in each extract by HPLC-UV

According to the IOOC method (Class, 2009). The high-performance liquid chromatography method was utilized to identify and quantify the phenolic components present in the *P. graveolens* extract. Before being subjected to HPLC (UV-vis) analysis, the extracts (10 mg) were diluted in 1 mL of 80% methanol and filtered through a 0.45 μm filter. A C18HG Wakosil (5 μm, 4.6\*150 mm) was used to separate the phenolic components at 40 °C.

Elution was done in gradient mode using a binary solvent mixture consisting of 50/50 methanol/acetonitrile (solvent B) and acidified water containing 0.2% phosphoric acid (solvent A). After rebalancing for 12 minutes, a linear gradient was passed for 40 minutes, going from 96% (A) and 4% (B) to 50% (A) and 50% (B); for 5 minutes, it was passed to 40% (A) and 60% (B); and for 15 minutes, it was passed to 0% (A) and 100% (B). The injection volume for each sample was 20 μL,

and the flow rate of the mobile phase was 1 mL/min. By contrasting the retention durations and UV spectra of each phenolic compound with those of reference compounds, all phenolic compounds were found. Utilizing peak regions in comparison to standards, quantitative measures were made. The experiment was repeated three times.

## Antibacterial activity testing

### *Microcystis aeruginosa* strain

The strain of *M. aeruginosa* MCAUt, a non-axenic, monoclonal, colonial, and toxin producing strain, isolated from the Lalla Takerkoust eutrophic lac reservoir in 2015 was used as a model of harmful cyanobacteria (El Amrani Zerrifi et al., 2019). After isolation and identification, the strain was maintained in culture in liquid BG11 medium under controlled conditions of light (2500 lux, photoperiod 15:9 light: dark) and temperature (25 °C ± 2 °C).

### *Chlorella* sp. strain

A non-axenic strain of *Chlorella* sp., isolated in 2017 from an artificial pond in the biology department of the Faculty of Sciences Semlalia (Marrakech), was used as a model of beneficial algae of the freshwater ecosystem (El Amrani Zerrifi et al., 2019). After isolation, the strain was maintained in culture in liquid Z8 medium under the same conditions (2500 lux, 15:9 light: dark, 25 °C ± 2 °C).

### Anti-cyanobacterial and anti-algal assay

A qualitative assessment of the anti-algal/cyanobacterial potential of our essential oils and extracts in solid medium was performed during this experiment.

A modified double-layer agar plate method was used to obtain a homogenous distribution of the microorganisms in solid medium according to the method previously described by Tazart et al. (2021). BG11 medium was used for the cyanobacterium and Z8 medium for the microalgae.

Briefly, soft-agar medium made of 2 mL of microorganism suspension at the exponential growth phase (30 × 10<sup>9</sup> cell/L (OD<sub>600</sub>=0.8853 at 1 cm) for *Chlorella* sp.; 100 × 10<sup>9</sup> cell/L (OD<sub>600</sub>=3.3631 at 1 cm) for *Microcystis aeruginosa* MCAUt) and 3 mL of 1% agar medium was poured onto 20 mL of basal

**Table 1.**

The minimum inhibitory concentration of the teste pelargonium essential oil and copper sulfate against *M. aeruginosa* and *Chlorella* sp.

	Minimum inhibitory concentration (µg/ml)	
	<i>M. aeruginosa</i>	<i>Chlorella</i> sp.
Pelargonium essential oil	12.500	12.500
Copper sulfate	3.125	3.125

2% agar medium. Paper disks of 6 mm, soaked with 10 µl of the essential oils or extract and copper sulfate used as positive control (30 mg/mL), sterile culture medium used as negative control, were placed onto the center of the plates, immediately put at 4 °C for 2 hours. The double-layer agar plates were cultivated for 10 days under the previously described culture conditions. The anti-cyanobacterial and anti-algal activity was expressed as mm of a clear zone around the paper disk on the double-layer agar plates. Each treatment was performed in triplicate.

The minimum inhibitory concentration (MIC) of the teste essential oils was determined using the broth microdilution method, following the NCCLS M7-A4 guidelines (NCCLS 1997). A 96-well plate was prepared, with each well containing 100 µL of the corresponding culture in the exponential growth phase ( $3 \times 10^6$  cells/mL) and 100 µL of essential oil dissolved in 1% DMSO, yielding a final concentration range of 3.125 to 6400 µg/mL. Copper sulfate and 1% DMSO were included as positive and negative controls, respectively, at corresponding concentrations (Table 1). The plates were incubated for five days under the previously described culture chamber conditions. All tests were conducted in triplicate using BG11 medium for the cyanobacterium and Z8 medium for the microalgae.

### Statistical analysis

#### Relative linear importance

The Pearson correlation coefficient has been used to analyze the linear relationship degree between the output variables (essential oil yield, antioxidant activity, content of total phenolic compounds, content of total flavonoid compounds, antibacterial activity of essential oil, and antibacterial activity of solar extract) and the other input variables under investigation, such as the quantity of *Pelargonium* ( $Q_{pel}$ ), Quantity of water ( $Q_w$ ), solar radiation intensity ( $G_b$ ), and temperature at the focal point ( $T_f$ ). The strength of the linear rela-

tionship between two quantitative variables, X and Y, is expressed by the Pearson correlation coefficient (r), a statistical measure that goes from -1 to +1. A p-value ( $p < 0.05$ ) was also used to assess the level of significant linear correlation.

#### Linear regression

Linear regression analyses were performed to evaluate the relationships between the biochemical parameters (TPC, TFC) and the biological activities (IC50, EO, TAE, TAEO). Before modelling, Pearson correlation coefficients were computed to identify the strongest explanatory variables and to assess potential multicollinearity among predictors. Because of this strong multicollinearity and the limited number of experimental points ( $n = 9$ ), multiple linear regression was not statistically appropriate, as it would produce unstable or non-interpretable coefficients.

Therefore, the analysis was restricted to simple linear regression models of the form:

$$Y_i = \beta_0 + \beta_1.TPC + \epsilon_i$$

where  $Y_i$  represents each response variable (EO, IC50, TAE, TAEO, TFC). For each model, the regression equation, coefficient of determination ( $R^2$ ), and p-value of the slope were calculated. All analyses were conducted in R (version 3.5.2), and results were visualized using an open-source data visualization package `ggplot2` (<https://r-graph-gallery.com/ggplot2-package.html>).

$$R^2 = 1 - \frac{\sum(y_o - y_t)^2}{\sum(y_t)^2}$$

where  $y_o$  is the expected value, and  $y_t$  is the real value. The model is optimal if  $R^2$  is near one.

## RESULTS AND DISCUSSION

### Solar hydro-distillation

On a particular day (July 14, 2023) in the summer, the energy was calculated; it was discovered that the useful energy was 5.25

kWh in 4 hours of operation. Figure 2 shows that a high essential oil volume is collected every 30 minutes after each high beam radiation value; this had an immediate impact on the extraction process. Since the solar reflector only uses direct sunlight, the daily solar energy and cloud cover density have an impact on the efficiency of solar extraction.

More significantly, it has been demonstrated that the temperature at the fixed focal point is below 350 °C due to problems with solar radiation concentration, while it can reach 500 °C. In any case, the temperature observed was significantly higher than what is required for water to boil, which could have the benefit of accelerating the extraction of essential oils from *P. graveolens* leaves.

According to Fig. 2, it was possible to reach 0.64 % as yield in the essential oil extracted by SHD process of *P. graveolens* leaves; this value is still higher than the other studies cited before. The essential oils yields found by (Rana, Puram, Adhoiwala, & Dun, 2003; Boukhris et al. 2012; Boukhatem, Kameli, & Saidi, 2013; Boukhris et al. 2015; Ben ElHadj Ali et al. 2020) were 0.22 %, 0.19 %, 0.18 %, and 0.15 % respectively, using a Clevenger apparatus for hydro-distillation of the *P. graveolens* leaves. Also, the yield found in this work is higher than the 0.49 % obtained by Riahi et al. (2020) using the maceration method, but still slightly below the 0.7 % reported by Čavar and Maksimović (2012) using a Clevenger apparatus for hydro-distillation of the *P.*

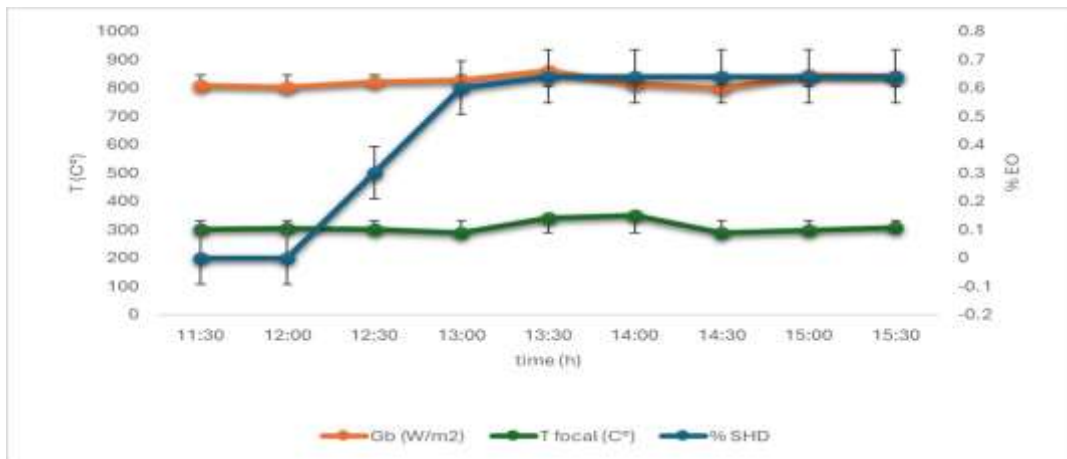


Figure 2. Temperature at the focal point, essential oil yield, and solar flux as functions of time using SHD method.

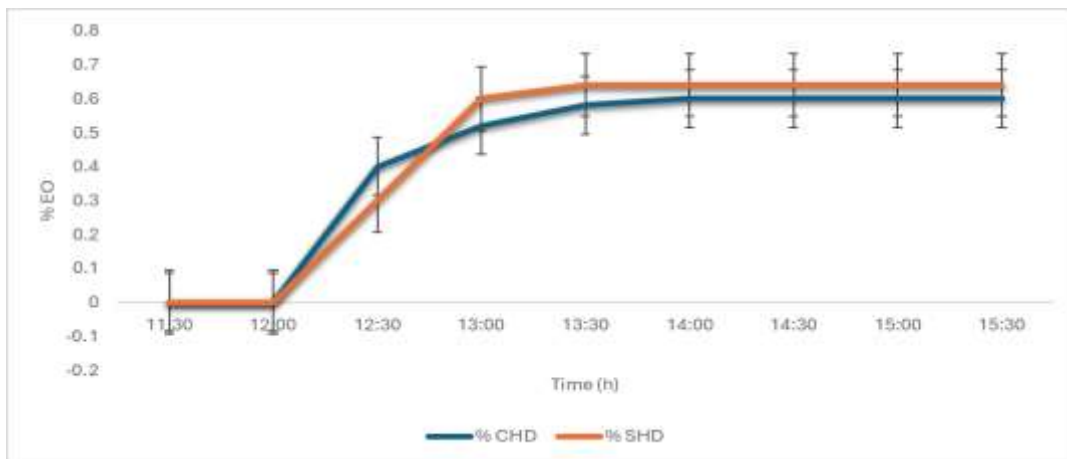


Figure 3. Essential oil yield for SHD and CHD as a function of time

*graveolens* leaves. These fluctuations and changes in the yields can be linked to a variety of factors in addition to the plant's origin. The crucial ones are:

**Plant spacings.** Rao (2002) recommends 60x30 cm spacing and preferably intercropping for better yield, also weed control has a positive effect on yield increase, Mineral amendments help the plants grow larger, which increases the amount of oil produced by using organic fertilizers in combination with nitrogen (160 Kg/ha).

**Drying time.** This tendency is mentioned by the work of Gomes, Mata, and Rodrigues (2004) who reported in a comparative study that the EO distilled from dry plants and yellow foliage is the best qualitatively and quantitatively.

**Extraction method.** Studies had shown, on the one hand, the influence of the extraction technique and, on the other hand, the influence of the vegetative cycle on the yield and quality of the EO (Gomes, Mata, & Rodrigues, 2004).

Fig. 2 also shows the impact of direct irradiation collected by the Scheffler dish on the temperature recorded at the focal point, and thus on the maintenance of the state of evaporation of water in the alembic to extract the EOs.

### **Solar and conventional hydro-distillation kinetics**

The kinetics of the distillation systems (solar and conventional) are composed of three steps (Fig. 3). For zero yields, we have a plateau in the first step, corresponding to the heating phase of water and *P. graveolens*. The second step corresponds to a marked increase in the quantity of EO recovered (30-120 min). Finally, during the third step, the curve tends towards a second level, which corresponds to the maximum possible yield to be reached.

As shown in Fig. 3, the SHD and CHD systems were able to extract almost the same amount of EO. Moreover, the solar system has more characteristics that are positive, because solar energy is a source that is free while traditional sources utilize up to 3.2 kWh, or 2560 g of CO<sub>2</sub> during the extraction process. As long as SHD can achieve approximately the same yield in 2 h rather than 3 h, the extraction time was also reduced by 37 %. The solar

system can be considered an effective, environmentally friendly, and economical technique for extracting EOs.

From Fig. 3, it can be seen that our matrix belongs to the family of fragrant pelargonium where the secretory structures (which contains the EO) are superficial, the outer membrane or cuticle which is the only barrier to the release of the EO is quickly broken at boiling. The quick evaporation of the volatile chemicals during extraction accounts for the brief time it takes to achieve the cumulative peak. Before contacting water or its vapor, essences that are subcutaneous must first diffuse through the thickness of the plant tissue. They will then be slowly evaporated compared to the superficial secretory structures.

### **Essential oil composition of *P. graveolens* leaves**

The principal compounds found by GC-MS in *P. graveolens* EO are listed in Table 2. Overall, essential oils extracted by SHD and CHD units had equivalent quantities of essential oil compounds. Citronellol was the most common abundant component, which accounted for 27.54% for the SHD process and 26.51% for the CHD process followed by citronellyl formate and geraniol with a value of 13.63% and 11.94% respectively for the SHD process; 11.33% and 10.97% respectively for CHD process.

Consequently, in the solar process, solar radiation increased the distillation time by 36.8 % without significantly altering the oil's volatile composition, and this result was confirmed by Hilali et al. (2019). The results obtained in this study regarding essential oils are generally consistent with those reported previously (Atailia & Djahoudi 2015; Baldin et al. 2015; Szutt, Dołhańczuk-Sródka & Sporek, 2019), which showed that the chemical profile of *P. graveolens* essential oils includes numerous monoterpenes and oxygenated monoterpenes like  $\alpha$ -pinene, eucalyptol, l-menthone and geraniol.

Table 2 also shows that the molecule identification rate is slightly higher for SHD (94.98 %) than for CHD (94.88 %) due to some molecules (alloaromadendrene oxide, citronellyl oleate, heneicosane, dibenzyl ketoxime, dodecane, squalene, and verbenol) that are extracted by the SHD process and not by the CHD process (Fig. 4).

**Table 2.**  
Principal chemical compounds of *P. graveolens* EO extracted by SHD and CHD

N°	Retention time	Compounds	SHD (%)	CHD (%)
1	02.77	<i>Trans</i> - $\alpha$ -Ocimene	0.02 $\pm$ 0.001	0.02 $\pm$ 0.001
2	03.15	$\alpha$ -Pinene	0.03 $\pm$ 0.001	0.03 $\pm$ 0.001
3	03.64	Eucalyptol	0.09 $\pm$ 0.001	0.26 $\pm$ 0.010
4	04.48	Linalool	0.68 $\pm$ 0.011	1.27 $\pm$ 0.051
5	04.70	<i>Cis</i> -Rose oxide	1.19 $\pm$ 0.032	1.46 $\pm$ 0.033
6	05.38	Citronellal	0.29 $\pm$ 0.021	0.01 $\pm$ 0.001
7	05.54	l-Menthone	5.20 $\pm$ 0.341	3.37 $\pm$ 0.167
8	06.10	Levomenthol	0.12 $\pm$ 0.051	0.18 $\pm$ 0.044
9	06.22	$\alpha$ -Terpineol	0.09 $\pm$ 0.001	0.08 $\pm$ 0.001
10	06.85	Citronellol	27.5 $\pm$ 1.471	26.5 $\pm$ 1.138
11	07.21	$\alpha$ -Citral	0.60 $\pm$ 0.230	0.36 $\pm$ 0.101
12	07.45	Geraniol	11.9 $\pm$ 1.019	10.9 $\pm$ 1.986
13	07.92	Citronellyl formate	13.6 $\pm$ 1.656	11.3 $\pm$ 1.876
14	08.58	Geranyl formate	8.31 $\pm$ 1.991	5.84 $\pm$ 1.987
15	09.75	Neric acid	0.11 $\pm$ 0.101	0.07 $\pm$ 0.001
16	09.82	Citronellol acetate	0.16 $\pm$ 0.021	0.21 $\pm$ 0.011
17	09.92	$\alpha$ -Cubebene	0.25 $\pm$ 0.061	0.16 $\pm$ 0.011
18	10.09	$\alpha$ -Guaiene	0.12 $\pm$ 0.011	0.05 $\pm$ 0.011
19	10.63	Geranyl acetate	2.09 $\pm$ 0.201	0.98 $\pm$ 0.101
20	10.92	$\alpha$ -Bourbonene	0.90 $\pm$ 0.021	1.02 $\pm$ 0.071
21	11.85	Caryophyllene	0.53 $\pm$ 0.051	1.26 $\pm$ 0.011
22	12.21	Citronellyl iso-valerate	0.18 $\pm$ 0.021	0.21 $\pm$ 0.013
23	12.41	$\alpha$ -Muurolene	0.08 $\pm$ 0.001	0.23 $\pm$ 0.015
24	12.49	$\alpha$ -copaene	0.09 $\pm$ 0.001	0.14 $\pm$ 0.021
25	12.59	Aromandendrene	0.26 $\pm$ 0.034	0.47 $\pm$ 0.018
26	12.75	Humulene	0.18 $\pm$ 0.012	0.37 $\pm$ 0.013
27	12.96	Alloaromadendrene	0.56 $\pm$ 0.014	0.50 $\pm$ 0.018
28	13.06	Geranyl propionate	0.40 $\pm$ 0.032	0.53 $\pm$ 0.012
29	13.48	Germacrene D	0.67 $\pm$ 0.122	3.55 $\pm$ 0.154
30	13.64	Eudesma-4(14),11-diene	0.17 $\pm$ 0.012	0.50 $\pm$ 0.021
31	13.86	Ledene	1.19 $\pm$ 0.111	2.16 $\pm$ 0.132
32	13.95	$\alpha$ -Muurolene	0.15 $\pm$ 0.071	0.01 $\pm$ 0.001
33	14.05	4- <i>Epi-cis</i> -Dihydroagarofuran	0.07 $\pm$ 0.001	0.12 $\pm$ 0.021
34	14.13	Geranyl isobutyrate	0.24 $\pm$ 0.031	0.19 $\pm$ 0.013
35	14.37	Cubebol	0.40 $\pm$ 0.012	0.34 $\pm$ 0.056
36	14.57	<i>cis</i> -Calamenene	0.70 $\pm$ 0.013	1.34 $\pm$ 0.340
37	15.27	Alloaromadendrene oxide	0.49 $\pm$ 0.041	0.35 $\pm$ 0.056
38	15.91	Ledene oxide-(II)	0.18 $\pm$ 0.021	0.07 $\pm$ 0.001
39	16.08	Spathulenol	0.29 $\pm$ 0.019	0.22 $\pm$ 0.019
40	16.18	2-Phenylethyl tiglate	1.61 $\pm$ 0.161	1.64 $\pm$ 0.171

**Table 2.** Continued

41	16.26	Caryophyllene oxide	1.55 ± 0.165	1.20 ± 0.191
42	16.57	Geranyl isovalerate	0.18 ± 0.018	0.11 ± 0.017
43	17.08	<i>Epi</i> -cubenol	0.28 ± 0.065	0.43 ± 0.087
44	17.24	ç-Eudesmol	5.12 ± 0.980	7.68 ± 0.819
45	17.63	Agarospirol	0.23 ± 0.016	0.32 ± 0.045
46	17.87	Cubenol	0.40 ± 0.076	0.61 ± 0.019
47	18.04	Globulol	1.20 ± 0.371	1.92 ± 0.565
48	18.26	Citronellyl tiglate	0.36 ± 0.016	0.45 ± 0.017
49	19.20	Geranyl angelate	2.10 ± 0.419	2.30 ± 0.401
50	19.61	<i>trans</i> -Geranylgeraniol	0.17 ± 0.076	0.21 ± 0.018
51	20.28	Isoaromadendrene epoxide	0.13 ± 0.001	0.06 ± 0.001
52	23 30	Aromadendrene oxide	0.17 ± 0.001	0.05 ± 0.001
<b>Total</b>			94.98 ± 4.089	94.88 ± 3.903

Moreover, the amount of energy accessible at the alembic's bottom after four hours of distillation was  $E_{\text{bot}}=11.92$  kWh and the thermal efficiency of alembic was  $\eta_{\text{still}} = 94.61$  %, thus the useful energy was 11.28 kWh exploitable by water and *P. graveolens*. Given that it is a renewable and free source, energy consumption has been reduced with the SHD approach, the CHD method consume approximately 11.28 kWh of butane in 4 hours which stands for 9.54 kg of CO<sub>2</sub> (Hilali et al. 2018), SHD is a reasonable solution for the economical, efficient, and environmental extraction of EO from *P. graveolens*.

#### Analysis of the total polyphenol content and flavonoid

To evaluate the impact of SHD and CHD on the extraction of TPC and TFC in *P. graveolens* extract, a comparison was made after the deodorization by two processes. The TPC obtained in SHD and CHD extracts were 249.72 mg EAG g<sup>-1</sup> DM and 209.48 mg EAG g<sup>-1</sup> DM, respectively. In the same context, the TFC values obtained in SHD and CHD extracts were 205.88 mg EQ g<sup>-1</sup> DM and 147.05 mg EQ g<sup>-1</sup> DM respectively.

Polyphenols are organic molecules containing one or more aromatic nuclei and one or more hydroxyl groups attached. Because of this, they have a high molecular weight, and unlike essential oils, they cannot be charged with water steam. It has been observed that the *P. graveolens* subjected to SHD treatment had higher TPC and TFC values than the CHD

process; probably there is more conservation of total phenols after using SHD (Fig. 5).

Figure 5 demonstrates that, in comparison to the SHD extract (249.72 mg EAG g<sup>-1</sup> DM) and TFC (205.88 mg EQ g<sup>-1</sup> DM), the extraction by maceration using methanol 96 % yielded the maximum amount of TPC (399.92 mg EAG g<sup>-1</sup> DM) and TFC (352.94 mg EQ g<sup>-1</sup> DM). Distilled water is a non-toxic and relatively cheap bio-solvent that was used in distillation as it is a good extracting agent due to its polarity (Guinda et al. 2015) and to contrast the amount achieved in TFC and TPC with the amount obtained with methanol. The TPC and TFC obtained by maceration with distilled water were 192.43 mg EAG.g<sup>-1</sup> DM) and 117.65 mg EQ.g<sup>-1</sup> DM, respectively. Besides that, the extraction of phenolic compounds is enhanced by using polar solvents. The SHD extract gives high TPC and TFC values of 249.72 EAG g<sup>-1</sup> DM and 205.88 EAG g<sup>-1</sup> DM, respectively against 209.48 mg EAG g<sup>-1</sup> DM and 147.06 mg EQ g<sup>-1</sup> DM for the CHD extract. Solar heating has mechanically affected the ultrastructure of the *P. graveolens*. Hilali et al. (2018) investigated the processes used to extract metabolites from rosemary leaves, which were induced by the same Scheffler parabola. In our situation, a rise in porosity was carried on by the 105 °C temperature inside the alembic. Such severe deformation will enhance both the release of cellular substances and the diffusion of the solvent in the plant matrix. SHD is a green technique as a result since it makes use of solar energy.

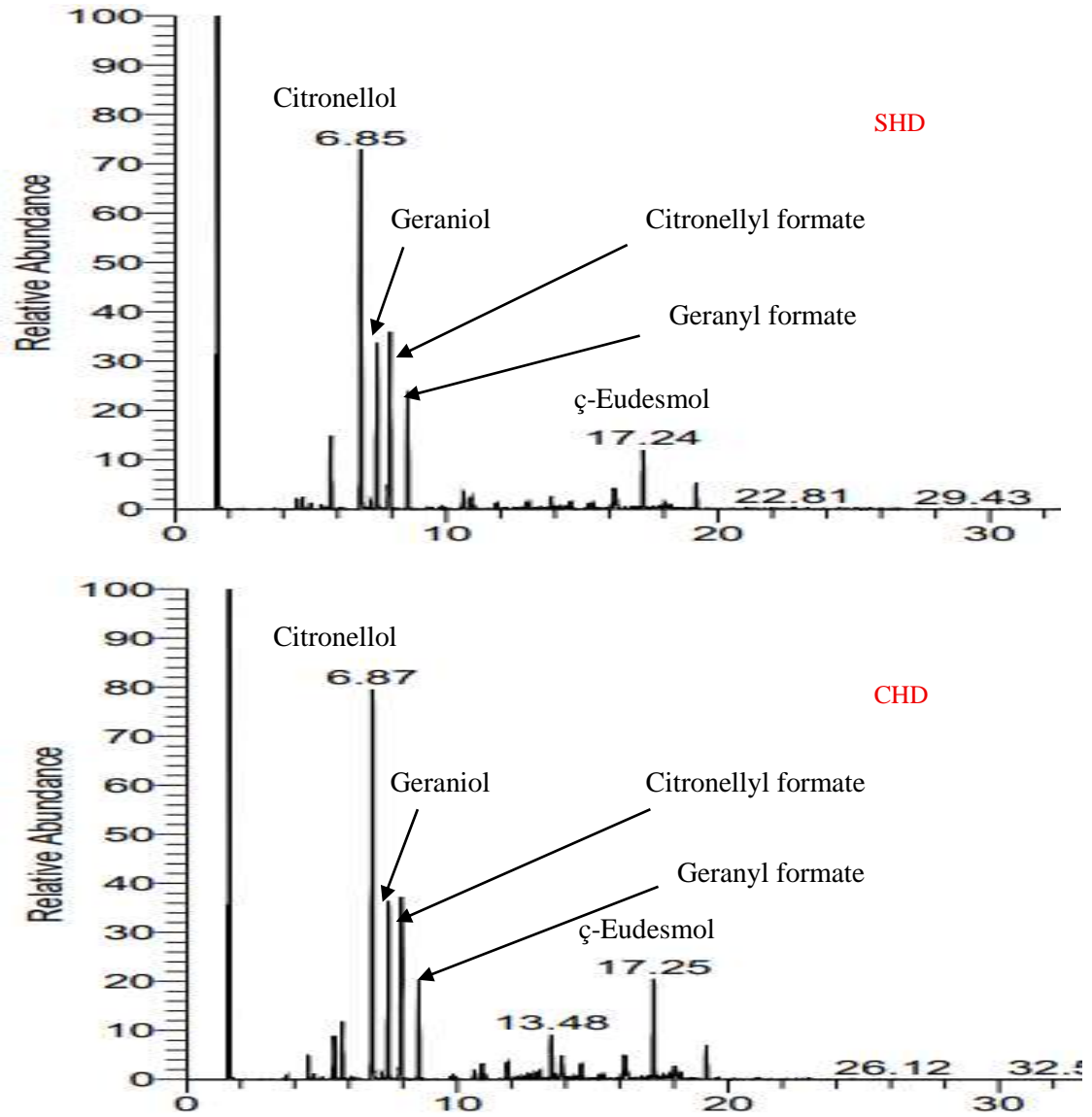


Figure 4. GC-MS chromatograms of *P. graveolens* essential oils extracted by SHD and CHD.

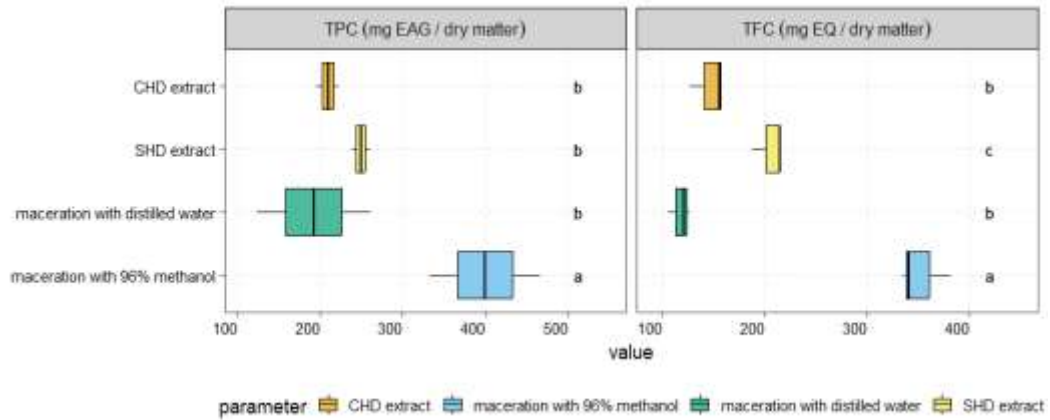


Figure 5. TPC and TFC of *P. graveolens* extracts using CHD, SHD, 96% methanol, and distilled water.

**Table 3.**

Antioxidant activity (AA) and IC<sub>50</sub> of different extracts of *P. graveolens*

Extract	AA (%)	IC <sub>50</sub> (µg.mL <sup>-1</sup> )
Maceration with methanol 96 %	91.07 ± 3.165	2.55 ± 0.651
SHD extract	82.65 ± 3.018	6.54 ± 0.787
CHD extract	82.44 ± 3.618	7.78 ± 0.765

The results obtained in the SHD extract in this investigation are comparable with previous research. The TPC and TFC values are higher than 142.71 mg EAG g<sup>-1</sup> DM and 52.32 mg EQ g<sup>-1</sup> DM respectively reported by (Ben ElHadj Ali et al. 2020) using maceration with methanol. Also higher than 105.62 mg EAG g<sup>-1</sup> DM and 48.55 mg EQ g<sup>-1</sup> DM reported by Riahi et al. (2020), using the maceration method with ethanol 80 %. (El Aanachi et al. 2020), on the other hand, reported values of 381.25 mg EAG g<sup>-1</sup> DM for TPC and 330.08 mg EQ g<sup>-1</sup> DM for TFC using maceration with methanol. Therefore, these last results are higher compared to those obtained in the SHD extract but are also lower than those obtained using 96 % methanol as maceration solvent in this study.

#### Analysis of the antioxidant activity of *P. graveolens*

The antioxidant activity of various *P. graveolens* extracts produced in different ways was tested using DPPH scavenging activity. It should be noted that the IC<sub>50</sub> decreases with increasing antioxidant activity. Three methods were used (Table 3), in the last method, an organic solvent was used. In general, organic solvents are expensive, can be harmful to the environment and employees' health, and need handling concerns. On the other hand, water has been suggested as an extraction solvent wherever possible due to its affordability and environmental friendliness (Da Rosa, Vanga, Garipey, & Raghavan, 2019). Our work's IC<sub>50</sub> was less than the 1.5 µg.mL<sup>-1</sup> obtained by Ali et al. (2018), who modified the growth and productivity of AA and essential oils of *P. graveolens* utilizing moringa leaf extract as a substitute and eco-friendly plant growth stimulant (Ali et al. 2018).

In addition, Boukhris et al. (2015) demonstrates that, depending on the phenological stage, the evaluated samples were capable of converting the stable violet DPPH radical to the yellow DPPH-H with a range of IC<sub>50</sub> values between 1 and 2 µg mL<sup>-1</sup>.

Other results were reported for the AA of *P. graveolens* with IC<sub>50</sub> of 21.11 - 31.03 µg mL<sup>-1</sup> employing methanolic extracts to inhibit tyrosinase and urease (El Aanachi et al. 2020). The result obtained in this work were higher than 12.24 µg mL<sup>-1</sup> and 14.68 µg mL<sup>-1</sup> reported by Ben ElHadj Ali et al. (2020) using methanolic and ethanolic extracts, respectively. Al-Saffar et al. (2016) showed a value of 484 µg.mL<sup>-1</sup> using methanol as a solvent of maceration. As for the comparison between the AA of SHD extract and the AA of CHD extract, the AA obtained by SHD is greater than that obtained by CHD (IC<sub>50</sub> = 6.54 µg mL<sup>-1</sup>) than for the CHD extract (IC<sub>50</sub> = 7.78 µg mL<sup>-1</sup>).

#### Characterization of phenolic compounds from *P. graveolens* extracts by HPLC-UV

Figure 6 shows the identification of the phenolic components in each extract. This is the first time HPLC-UV has been used to determine the compounds of extracts obtained by SHD and CHD of *P. graveolens* leaves using water as polar solvent. The major compounds identified are pyrogallol, gallic acid, syringic acid, tyrosol, catechin, protocatechuic acid, caffeic acid, coumaric acid, ferulic acid, hesperidin, salicylic, quercetin and kaempferol. Several factors such as the sampling period, the cultivar, age and climatic changes affect the composition of the plant (Souilem et al. 2017).

The SHD extract showed that tyrosol (31.71 mg g<sup>-1</sup> DM), gallic acid (24.67 mg g<sup>-1</sup> DM), protocatechuic acid (22.59 mg g<sup>-1</sup> DM) and ferulic acid (21.04 mg g<sup>-1</sup> DM) were major compounds in *P. graveolens* extracts. Besides, when this extraction was carried out by the CHD method, it was found that the tyrosol (14.82 mg.g<sup>-1</sup> DM), and gallic acid (22.58 mg g<sup>-1</sup> DM) contents decreased, when the amount of protocatechuic acid (37.51 mg g<sup>-1</sup> DM) increased. In addition, the major compound in this case is salicylic with a high amount of 66.97 mg g<sup>-1</sup> DM. Moreover, the identification of phenolic compounds by HPLC has never been done previously.

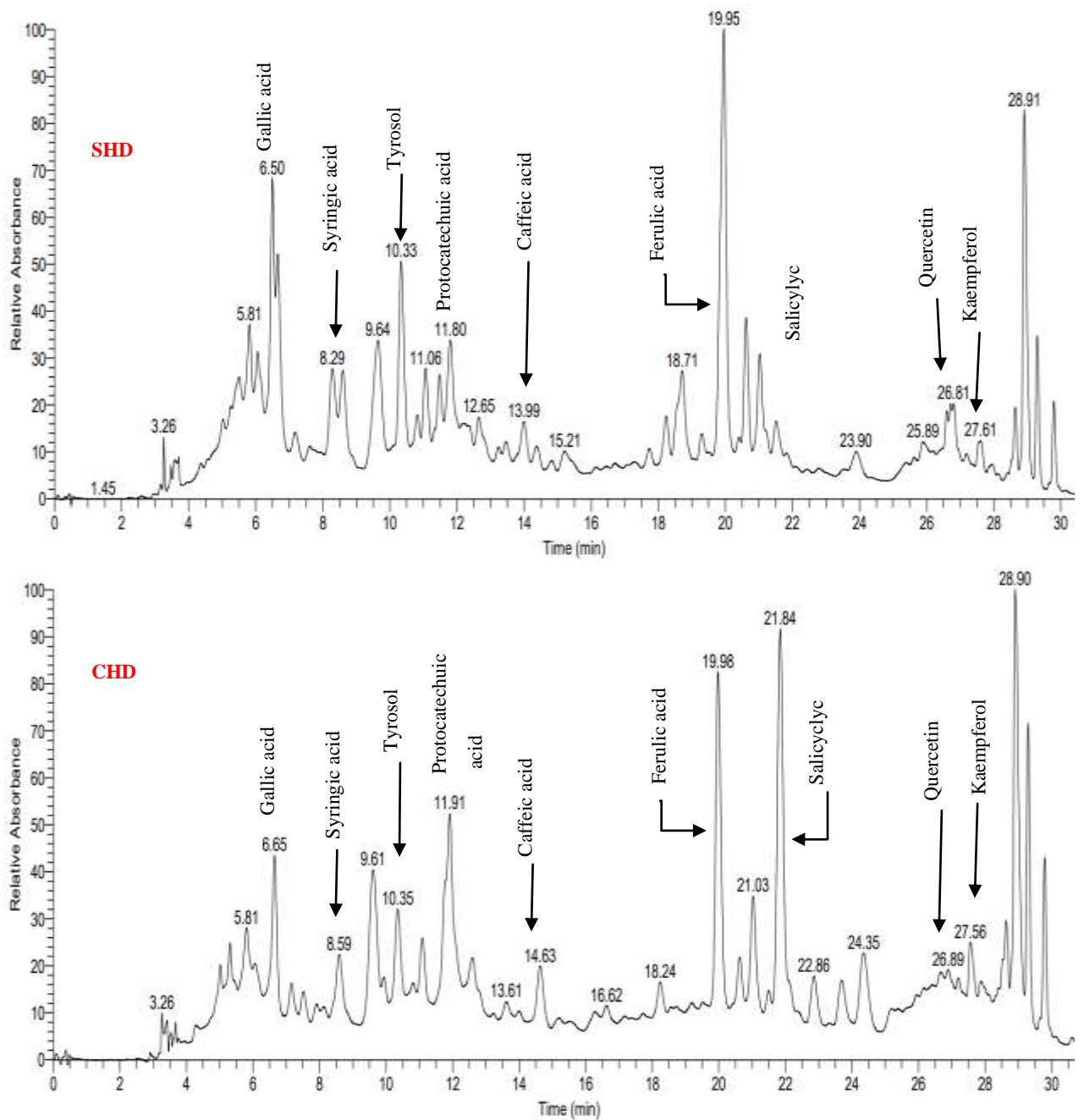


Figure 6. Identified phenolic compounds in *P. graveolens* extracts using CHD and SHD methods

### Anti-cyanobacterial/anti-algal test of *P. graveolens* essential oil and extract

Table 4 displays the zones of inhibition following a 7-day incubation period. Copper sulfate was used as a positive control, whereas DMSO was used as a negative control. Copper sulfate demonstrated strong algicidal activity against both microalgae tested ( $23.73 \pm 0.66$  and  $25.56 \pm 0.87$  mm against *M. aeruginosa* and *Chlorella sp.*, respectively). In contrast,

SHD essential oil of *P. graveolens* showed moderate algicidal action against *M. aeruginosa* and *Chlorella sp.*, with growth inhibition diameters of  $15.43 \pm 0.32$  mm and  $16.73 \pm 0.40$  mm, respectively. While CHD essential oil showed moderate activity against *M. aeruginosa* and *Chlorella sp.* with inhibition zones measuring  $14.40 \pm 0.34$  mm and  $14.03 \pm 0.30$  mm, respectively. The anti-cyanobacterial/anti-algal activity of the essential oil of SHD is similar in comparison with

that extracted by CHD. These results are confirmed by Atailia and Djahoudi (2015) when they studied the anti-cyanobacterial/anti-algal activity of an essential oil rich in citronellol (19.22%) on a bacterial population composed of one hundred and thirty strains, and shows that the average value of minimum inhibitory concentrations (MIC) is 1%.

Another study by Boukhatem et al. (2013) showed that *S. aureus* and *E. faecalis* (21.17 mm) were the most sensible strains to EO from *pelargonium* leaves, followed by *B. subtilis* (20.5 mm) and *S. epidermidis* (16.17 mm). Ben ElHadj Ali et al. (2020) showed that the zones of inhibition of essential oil-sensitive strains of *P. graveolens* were between 21 mm for leaf oils and 22.5 mm for flower oils, respectively, for *Pseudomonas aeruginosa* and *Bacillus cereus* strains.

On the other hand, the SHD *P. graveolens* extract showed algicidal action against *M. aeruginosa* and *Chlorella* sp., with growth inhibition diameters of  $17.13 \pm 0.35$  mm and  $25.96 \pm 0.15$  mm, respectively.

In a recent study, the essential oil of *P. graveolens* showed very low activity against *M. luteus* (3 mm) and *S. aureus* (4 mm) strains (Rathore, Mukhia, Kumar, & Kumar, 2023). Our results obtained with essential oils are in agreement with that of Al-Mijalli et al. (2022) who showed that the best antibacterial effect against a panel of microorganisms was ob-

tained with an essential oil extracted at the stage of full bloom with zones of inhibition of diameter between  $11.00 \pm 0.17$  mm and  $17.30 \pm 0.17$  mm. In another study (Sompaga et al. 2016) found that methanolic extract of *P. graveolens* showed better zone of inhibition on *K. pneumonia* (13 mm) than ethyl acetate extract of *P. graveolens* on *E. coli* (12 mm) was determined at the concentration of 20 ug/mL.

Ben ElHadj Ali et al. (2020) further stated that the various *P. graveolens* extracts showed high activity against *B. cereus* and *P. aeruginosa*, with the strongest inhibition zones ranging from 13.5 to 22.5 mm, respectively.

The methanol extract of *P. graveolens* showed the largest zones of inhibition against all strains tested ranging from  $9.43 \pm 0.40$  to  $14.27 \pm 0.31$  mm against *P. aeruginosa* and *Listeria innocua*, respectively (El Aanachi et al. 2020).

*Pelargonium graveolens* essential oil's chemistry, which is high in alcohols and terpene phenols ( $\beta$ -citronellol, geraniol, linalool, epi- $\gamma$ -eudesmol), is primarily responsible for its strong anti-cyanobacterial/anti-algal activity. Numerous writers have examined the antibacterial properties of essential oils' primary constituents, arranging them in the following sequence: alcohols, hydrocarbons, ethers, ketones, aldehydes, and phenols (M'hamdi et al., 2024).

**Table 4.**

Algicidal activity of SHD essential oil, CHD essential oil and SHD extract of *P. graveolens* leaves against *M. aeruginosa* and *Chlorella* sp.

Tested species	Diameter of inhibition zone (mm)		
	<i>P. graveolens</i> extract	CHD <i>P. graveolens</i> oil	SHD <i>P. graveolens</i> oil
<b><i>Chlorella</i> sp.</b>			
Test 1	26.10	14.30	17.10
Test 2	25.80	14.10	16.30
Test 3	26.00	13.7	16.80
<b>Average</b>	25.97	14.03	16.73
<b>SD</b>	0.15	0.31	0.40
<b><i>M. aeruginosa</i></b>			
Test 1	16.80	14.20	15.30
Test 2	17.50	14.80	15.80
Test 3	17.10	14.20	15.20
<b>Average</b>	17.13	14.40	15.43
<b>SD</b>	0.35	0.35	0.32

### Relative linear importance

As was previously noted, the strength of the linear relationship between each pair of variables under investigation was evaluated using the Pearson coefficient.

A correlation matrix (Fig. 7a) provides an illustration of the findings, blue indicates a positive correlation, while red indicates a negative association.

A range of correlations, from weak (r-Pearson < 0.7 or r-Pearson > -0.7) to high (r-Pearson >

0.7 or r-Pearson < -0.7), can be seen. Consequently, IC<sub>50</sub> on the one hand and TPC and TFC on the other have a highly significant negative relationship of r=-0.88 each, while these two last variables and TAE have a high correlation of r=0.86 ± 0.011 and r=0.78 ± 0.021, respectively.

Also, there is a good positive correlation between EO and TAEO with r=0.71 ± 0.023 and between EO and TPC (r=0.92 ± 0.011), TFC (r=0.91 ± 0.017), and IC<sub>50</sub> (r=-0.91 ± 0.013).

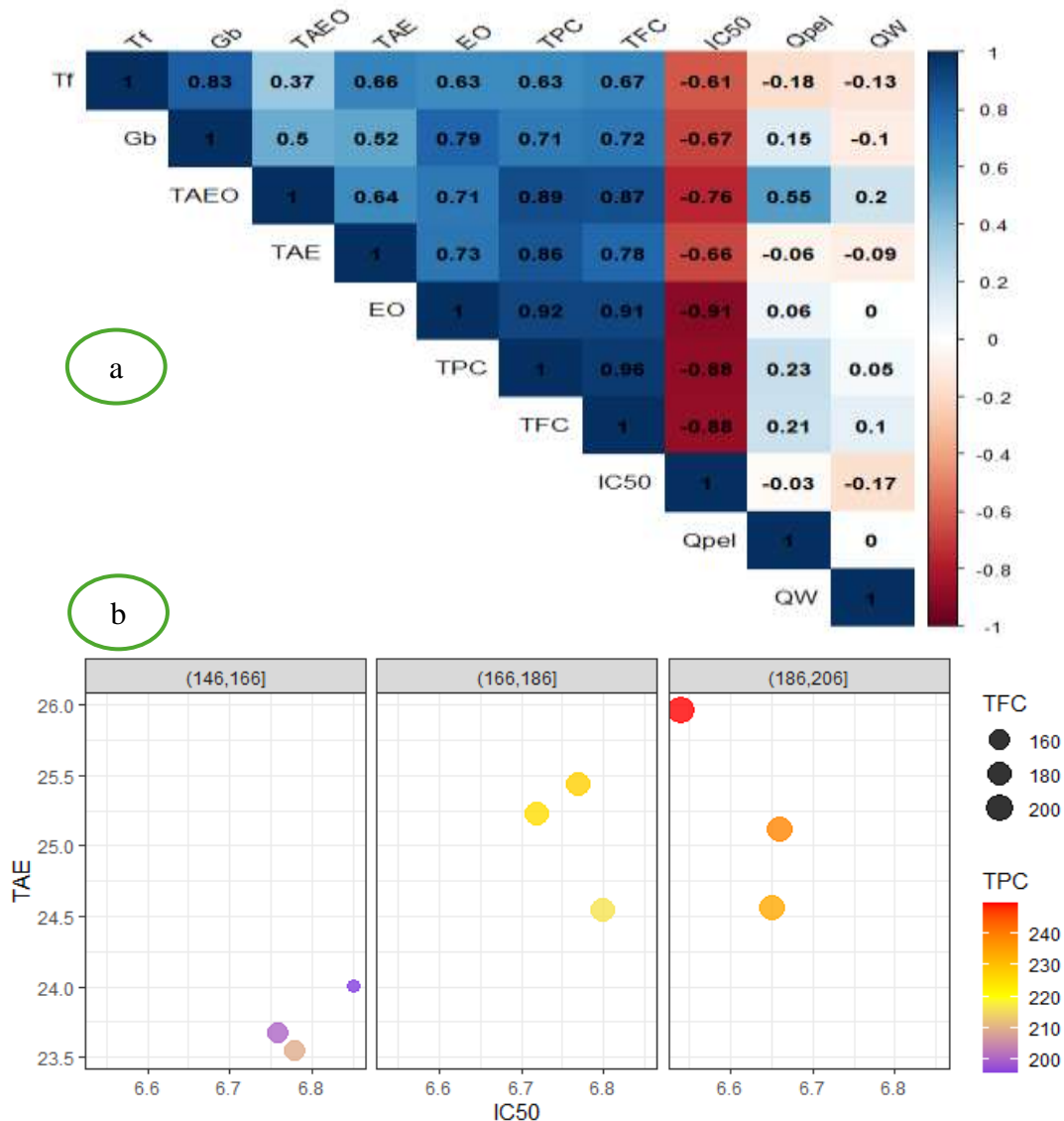


Figure 7. **a)** Analyzing the linear relationship between input variables and output variables; **b)** Analyzing the linear relationship between output variables (IC<sub>50</sub>: The median inhibitory concentration; TPC: Total phenolic compounds; TFC: Total flavonoids compounds; TAE: Test antibacterial of extract) using a correlation matrix diagram

Concerning the input variables,  $Q_w$  and  $Q_{pel}$  have no relationship with output variables. On the other hand, the Gb variable influences almost all the other parameters, with Pearson correlation coefficients of  $r=0.83 \pm 0.061$  on  $T_f$ ,  $r=0.79 \pm 0.018$  on EO,  $r=0.71 \pm 0.015$  on TPC, and  $r=0.72 \pm 0.015$  on TFC. This matrix exhibited also a substantial and positive correlation between TPC and TFC ( $r=0.96 \pm 0.021$ ).

This statistical analysis has never been done for the *P. graveolens* plant. Fig. 7b shows a good contribution by TPC and TFC to the antioxidant activity of the solar extract (low  $IC_{50}$ ), giving good antibacterial activity against the two bacterial strains tested.

The simple linear regression analyses revealed that TPC was the main explanatory variable for all biochemical and biological responses evaluated in this study (Fig. 8). A strong and significant positive linear relationship was observed between TPC and the TFC, with an excellent model fit ( $R^2 = 0.919$ ,  $p < 0.05$ ), indicating that flavonoids represent a major

fraction of the total phenolic compounds. TPC also showed strong associations with anti-cyanobacterial/anti-algal activities.

Specifically, the solar extract inhibitory activity against cyanobacterial/algal (TAE) increased significantly with TPC ( $R^2 = 0.734$ ,  $p < 0.05$ ), while antibacterial activity of the essential oils (TAE) exhibited a similarly strong dependence on TPC ( $R^2 = 0.791$ ,  $p < 0.05$ ).

The antioxidant response ( $IC_{50}$ ) was also significantly explained by TPC, displaying a negative linear trend indicative of stronger antioxidant activity with increasing phenolic content ( $R^2 = 0.780$ ,  $p < 0.05$ ). Essential oil yield (EO) showed a positive relationship with TPC ( $R^2 = 0.840$ ,  $p < 0.05$ ), although the slope was close to zero, reflecting lower sensitivity of this variable to phenolic composition.

Collectively, these results confirm that TPC is the dominant biochemical parameter governing the antioxidant, and anti-cyanobacterial/anti-algal properties of the extracts in this experimental system.

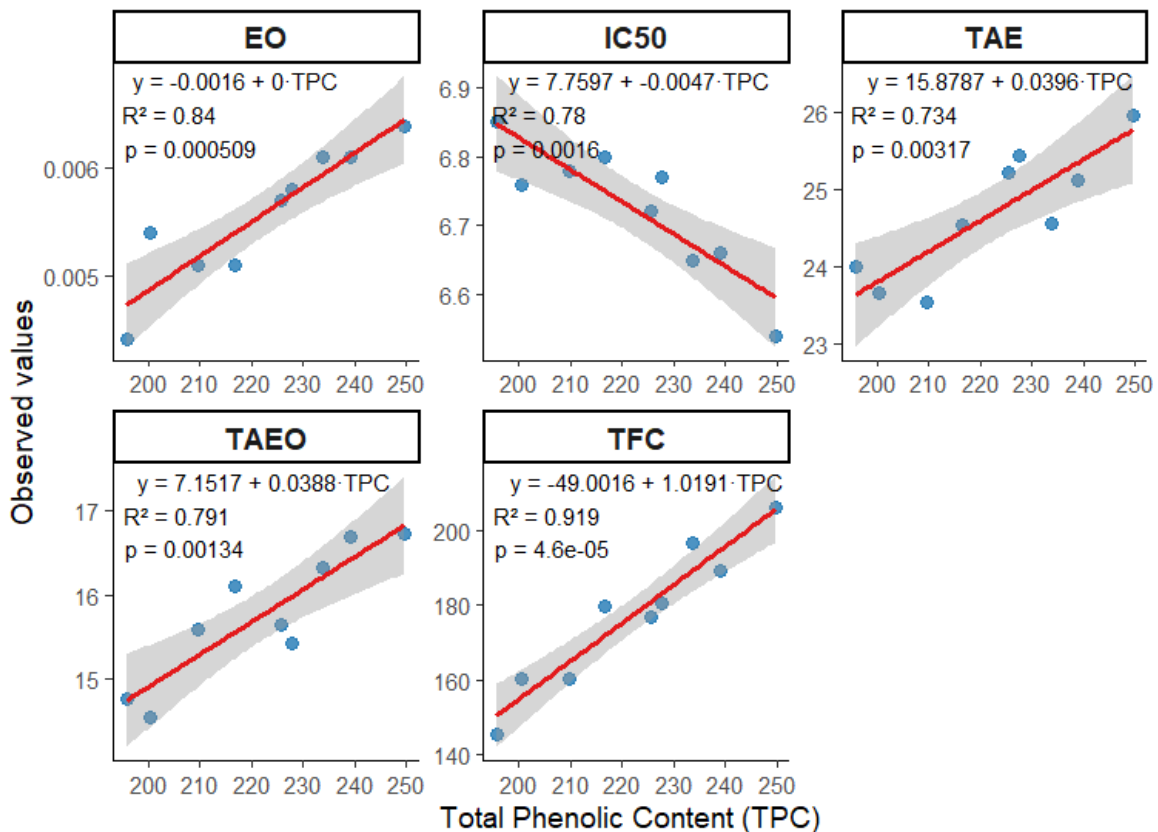


Figure 8. Linear models of output variables

## CONCLUSIONS

The impact of solar thermal processing on the essential oil, phenolic content, and antioxidant capacity of *P. graveolens* leaves has never been studied before. This process increases phenolic content and antioxidant capacity. The novelty of this work concerns also its contribution to the identification of some phenolic compounds of *P. graveolens* leaves using HPLC-UV analysis, such as tyrosol (31.71 mg g<sup>-1</sup> DM), gallic acid (24.67 mg g<sup>-1</sup> DM), protocatechuic acid (22.59 mg g<sup>-1</sup> DM), and ferulic acid (21.04 mg.g<sup>-1</sup> DM) which are the major compounds. The primary substances found in the essential oil by GC-MS in the SHD extract are citronellol, citronellyl formate, and geraniol with a value of 27.54 %, 13.63 %, and 11.94 % respectively. Moreover, the results showed that the SHD extract provided a very satisfactory extraction yield in TPC, TFC, and IC<sub>50</sub> (249.72 mg EAG g<sup>-1</sup> DW, 205.88 mg EQ g<sup>-1</sup> DW and IC<sub>50</sub>=6.54 µg mL<sup>-1</sup> respectively), which was higher than that attained by CHD process (209.48 mg EAG.g<sup>-1</sup> DW, 147.06 mg EQ.g<sup>-1</sup> DW and IC<sub>50</sub>=7.78 µg.mL<sup>-1</sup> respectively). Also, the essential oil and the extract prepared by SHD showed a medium antibacterial activity of extract against the two bacterial strains tested *M. aeruginosa* and *Chlorella* sp. SHD could be considered an interesting alternative way to intensify the extraction process of essential oil and phenolic compounds from *P. graveolens* leaves. The good functioning of SHD depends mainly on the efficiency of direct radiation in the work environment. Additionally, compared to conventional methods, solar energy has a noticeable impact on extraction kinetics; SHD can be considered a more effective process because it uses green energy. As a result, this study found a linear association between AA, TPC, TFC, and antibacterial activity of extract. Furthermore, for the SHD extract, there is a positive linear correlation (coefficient r = 0.86 and 0.78, respectively) between antibacterial activity of extract and TPC or TFC. These findings revealed that the AA and antibacterial activity of extract of the examined *P. graveolens* leaves were strongly influenced by the TPC and TFC.

## AUTHOR CONTRIBUTIONS

Conceptualization, K.E.; Methodology, K.E.; Investigation, formal analysis, K.E., S.S. and Y.S.; validation, K.E. and L.M.; Writing-

original draft preparation, K.E.; Writing-review and editing, K.E.; Supervision, A.H., A.I., B.O. and L.M.

## DATA AVAILABILITY STATEMENT

Data contained within the article.

## ACKNOWLEDGEMENTS

The authors would like to thank the National Center for Studies and Research on Water and Energy (Cadi Ayyad University-Morocco) for the technical and financial support of this research.

## CONFLICT OF INTEREST

The authors declare no conflict of interest.

## REFERENCES

- Afzal, A., Munir, A., Ghafoor, A., & Alvarado, J. L. (2017). Development of hybrid solar distillation system for essential oil extraction. *Renewable Energy*, 113, 22–29. <https://doi.org/10.1016/j.renene.2017.05.027>
- Al-Mijalli, S. H., Mrabti, H. N., Assagaf, H., Attar, A. A., Hamed, M., Baaboua, A. E. L., Omari, N., El Menyiy, N., El Hazzoumi, Z., Sheikh, R. A., Zengin, G., Sut, S., Dall'Acqua, S., & Bouyahya, A., (2022). Chemical profiling and biological activities of pelargonium graveolens essential oils at three different phenological stages. *Plants*, 11(17), 1–16. <https://doi.org/10.3390/plants11172226>
- Al-Saffar, A. Z., Al-Shanon, A. F., Al-Brazanc, S. L., Sabry, F. A., Hassan, F., & Hadi, N. A. (2016). Phytochemical analysis, antioxidant and cytotoxic potentials of *pelargonium graveolens* extract in human breast adenocarcinoma (MCF-7) Cell Line. *Asian Journal of Biochemistry*, 12(1), 16–26. <https://doi.org/10.3923/ajb.2017.16.26>
- Ali, E. F., Hassan, F. A. S., & Elgimabi, M. (2018). Improving the growth, yield and volatile oil content of *Pelargonium graveolens* L. Herit by foliar application with moringa leaf extract through motivating physiological and biochemical parameters. *South African Journal of Botany*, 119, 383–389. <https://doi.org/10.1016/j.sajb.2018.10.003>
- Atailia, I., & Djahoudi, A. (2015). Composition chimique et activité antibactérienne de l'huile essentielle de géranium rosat (*Pelargonium graveolens* L'Hér.) cultivé en Algérie. *Phytotherapie*, 13(3), 156–162. <https://doi.org/10.1007/s10298-015-0950-2>
- Baldin, E. L. L., Aguiar, G. P., Fanela, T. L. M., Soares, M. C. E., Groppo, M., & Crotti, A. E. M. (2015). Bioactivity of *Pelargonium graveolens* essential oil and related monoterpenoids against sweet potato whitefly, *Bemisia tabaci* biotype B. *Journal of Pest Science*, 88(1), 191–199. <https://doi.org/10.1007/s10340-014-0580-8>
- Ben ElHadj Ali, I., Tajini, F., Boulila, A., Jebri, M. A., Boussaid, M., Messaoud, C., & Sebaï, H. (2020). Bioactive compounds from Tunisian *Pelargonium graveolens* (L'Hér.) essential oils and extracts: α-

- amylase and acetylcholinesterase inhibitory and antioxidant, antibacterial and phytotoxic activities. *Industrial Crops and Products*, 158(May), 112951. <https://doi.org/10.1016/j.indcrop.2020.112951>
- Boukhatem, M. N., Kameli, A., & Saïdi, F. (2013). Essential oil of Algerian rose-scented geranium (*Pelargonium graveolens*): Chemical composition and antimicrobial activity against food spoilage pathogens. *Food Control*, 34(1), 208–213. <https://doi.org/10.1016/j.foodcont.2013.03.045>
- Boukhris, M., Bouaziz, M., Feki, I., Jemai, H., El Feki, A., & Sayadi, S. (2012). Hypoglycemic and antioxidant effects of leaf essential oil of *Pelargonium graveolens* L'Hér. in alloxan induced diabetic rats. *Lipids in Health and Disease*, 11, 1–10. <https://doi.org/10.1186/1476-511X-11-81>
- Boukhris, M., Hadrich, F., Chtourou, H., Dhoub, A., Bouaziz, M., & Sayadi, S. (2015). Chemical composition, biological activities and DNA damage protective effect of *Pelargonium graveolens* L'Hér. essential oils at different phenological stages. *Industrial Crops and Products*, 74, 600–606. <https://doi.org/10.1016/j.indcrop.2015.05.051>
- Box, J. D. (1983). Investigation of the Folin-Ciocalteu phenol reagent for the determination of polyphenolic substances in natural waters. *Water Research*, 17(5), 511–525. [https://doi.org/10.1016/0043-1354\(83\)90111-2](https://doi.org/10.1016/0043-1354(83)90111-2)
- Brand-Williams, W., Cuvelier, M. E., & Berset, C. (1995). Use of a free radical method to evaluate antioxidant activity. *LWT - Food Science and Technology*, 28(1), 25–30. [https://doi.org/10.1016/S0023-6438\(95\)80008-5](https://doi.org/10.1016/S0023-6438(95)80008-5)
- Ćavar, S., & Maksimović, M. (2012). Antioxidant activity of essential oil and aqueous extract of *Pelargonium graveolens* L'Her. *Food Control*, 23(1), 263–267. <https://doi.org/10.1016/j.foodcont.2011.07.031>
- Class, A. (2009). Determination of biophenols in olive oils by HPLC. *International Olive Council*, 29, 1–8. <http://www.internationaloliveoil.org>
- Da Rosa, G. S., Vanga, S. K., Garipey, Y., & Raghavan, V. (2019). Comparison of microwave, ultrasonic and conventional techniques for extraction of bioactive compounds from olive leaves (*Olea europaea* L.). *Innovative Food Science and Emerging Technologies*, 58, 102234. <https://doi.org/10.1016/j.ifset.2019.102234>
- Dewanto, V., Xianzhong, W., Adom, K. K., & Liu, R. H. (2002). Thermal processing enhances the nutritional value of tomatoes by increasing total antioxidant activity. *Journal of Agricultural and Food Chemistry*, 50(10), 3010–3014. <https://doi.org/10.1021/jf0115589>
- El Aanachi, S., Gali, L., Nacer, S. N., Bensouici, C., Dari, K., & Aassila, H. (2020). Phenolic contents and in vitro investigation of the antioxidant, enzyme inhibitory, photoprotective, and antimicrobial effects of the organic extracts of *Pelargonium graveolens* growing in Morocco. *Biocatalysis and Agricultural Biotechnology*, 29(June), 101819. <https://doi.org/10.1016/j.bcab.2020.101819>
- El Amrani Zerrifi, S., Tazart, Z., El Khalloufi, F., Oudra, B., Campos, A., & Vasconcelos, V. (2019). Potential control of toxic cyanobacteria blooms with Moroccan seaweed extracts. *Environmental Science and Pollution Research*, 26(15), 15218–15228. <https://doi.org/10.1007/s11356-019-04921-9>
- Ezzarrouqy, K., Hejjaj, A., Idlimam, A., Ait, F., & Laila, N. (2022). Study of the energetic, exergetic, and thermal balances of a solar distillation unit in comparison with a conventional system during the distillation of rosemary leaves. *Environmental Science and Pollution Research*, 29(17), 25709–25722. <https://doi.org/10.1007/s11356-021-17612-1>
- Ferraz, C. A., Pastorinho, M. R., Palmeira-de-Oliveira, A., & Sousa, A. C. A. (2022). Ecotoxicity of plant extracts and essential oils: A review. *Environmental Pollution*, 292(PB), 118319. <https://doi.org/10.1016/j.envpol.2021.118319>
- Gomes, P. B., Mata, V. G., & Rodrigues, A. E. (2004). Characterization of Portuguese-grown geranium oil (*Pelargonium* sp.). *Journal of Essential Oil Research*, 16(5), 490–495. <https://doi.org/10.1080/10412905.2004.9698779>
- Guinda, Á., Castellano, J. M., Santos-Lozano, J. M., Delgado-Hervás, T., Gutiérrez-Adán, P., & Rada, M. (2015). Determination of major bioactive compounds from olive leaf. *LWT - Food Science and Technology*, 64(1), 431–438. <https://doi.org/10.1016/j.lwt.2015.05.001>
- Hilali, S., Fabiano-Tixier, A. S., Elmaataoui, M., Petitcolas, E., Hejjaj, A., Aitnouh, F., Idlimam, A., Jacotet-Navarro, M., Bily, A., Mandi, L., & Chemat, F. (2018). Deodorization by solar steam distillation of rosemary leaves prior to solvent extraction of rosmarinic, carnosic, and ursolic acids. *ACS Sustainable Chemistry and Engineering*, 6(8), 10969–10979. <https://doi.org/10.1021/acssuschemeng.8b02347>
- Hilali, S., Fabiano-Tixier, A. S., Ruiz, K., Hejjaj, A., Ait Nouh, F., Idlimam, A., Bily, A., Mandi, L., Chemat, F. (2019). Green extraction of essential oils, polyphenols, and pectins from orange peel employing solar energy: toward a zero-waste biorefinery. *ACS Sustainable Chemistry and Engineering*, 7(13), 11815–11822. <https://doi.org/10.1021/acssuschemeng.9b02281>
- Jayasimha, B. (2006). Application of Scheffler reflectors for process industry. In *Proceedings of the International Solar Cooker Conference*, 8, (pp. 1–2). Pune, India. [http://www.solare-bruecke.org/infoartikel/Papers\\_from\\_SCI\\_Conference\\_2006/Jayasimha\\_Rathod.pdf](http://www.solare-bruecke.org/infoartikel/Papers_from_SCI_Conference_2006/Jayasimha_Rathod.pdf)
- Juliani, H. R., Koroch, A., Simon, J. E., Hitimana, N., Daka, A., Ranarivelo, L., & Langenhoven, P. (2006). Quality of geranium oils (*Pelargonium* species): Case studies in Southern and Eastern Africa. *Journal of Essential Oil Research*, 18, 116–121. <https://doi.org/10.1080/10412905.2006.12067131>
- M'hamdi, Z., Bouymajane, A., Riffi, O., Rhazi Filali, F., Ettarchouch, M., ELhourri, M., & Amechrouq, A. (2024). Chemical composition and antibacterial activity of essential oil of *Pelargonium graveolens* and its fractions. *Arabian Journal of Chemistry*, 17(1), 105375. <https://doi.org/10.1016/j.arabjc.2023.105375>
- Machalova, Z., Sajfrtova, M., Pavela, R., & Topiar, M. (2015). Extraction of botanical pesticides from *Pelargonium graveolens* using supercritical carbon dioxide. *Industrial Crops and Products*, 67, 310–317. <https://doi.org/10.1016/j.indcrop.2015.01.070>
- Parejo, I., Viladomat, F., Bastida, J., Rosas-Romero, A., Flerlage, N., Burillo, J., & Codina, C. (2002). Comparison between the radical scavenging activity and antioxidant activity of six distilled and non-

- distilled Mediterranean herbs and aromatic plants. *Journal of Agricultural and Food Chemistry*, 50(23), 6882–6890. <https://doi.org/10.1021/jf020540a>
- Ponomareva, E. I., & Molohova, E. I. (2017). Evaluation of the efficiency of supercritical carbon dioxide extraction for *Pelargonium graveolens* L'Her essential oil production. *Russian Journal of Physical Chemistry B*, 11(8), 1270–1275. <https://doi.org/10.1134/S1990793117080097>
- Rana, V. S., Puram, K., Adhoiwala, L., & Dun, D. (2003). Chemical composition of the volatile oil of *Ageratum conyzoides* aerial parts. *International Journal of Aromatherapy*, 13(4), 203–206. [https://doi.org/10.1016/S0926-4562\(03\)00080-8](https://doi.org/10.1016/S0926-4562(03)00080-8)
- Rao, B. R. R. (2002). Biomass yield, essential oil yield and essential oil composition of rose-scented geranium (*Pelargonium* species) as influenced by row spacings and intercropping with cornmint (*Mentha arvensis* L.f. piperascens Malinv. ex Holmes). *Industrial Crops and Products*, 16(2), 133–144. [https://doi.org/10.1016/S0926-6690\(02\)00038-9](https://doi.org/10.1016/S0926-6690(02)00038-9)
- Rathore, S., Mukhia, S., Kumar, R., & Kumar, R. (2023). Essential oil composition and antimicrobial potential of aromatic plants grown in the mid-hill conditions of the Western Himalayas. *Scientific Reports*, 13(1), 1–13. <https://doi.org/10.1038/s41598-023-31875-3>
- Riahi, L., Cherif, H., Miladi, S., Neifar, M., Bejaoui, B., Chouchane, H., Masmoudi, A. S., & Cherif, A. (2020). Use of plant growth promoting bacteria as an efficient biotechnological tool to enhance the biomass and secondary metabolites production of the industrial crop *Pelargonium graveolens* L'Hér. under semi-controlled conditions. *Industrial Crops and Products*, 154(June), 112721. <https://doi.org/10.1016/j.indcrop.2020.112721>
- Scheffler, W. (2006). Introduction of the revolutionary design of Scheffler reflectors. In *Proceedings of the International Solar Cooker Conference*. Aislingen, Germany.
- Sompaga, S., Jyothi, B., Chekuri, S., Baburao, N., & Anupalli, R. (2016). Organic extracts of *Pelargonium graveolens*: phenol content, anti-oxidant and anti-bacterial activities. *European Journal of Medicinal Plants*, 17(1), 1–8. <https://doi.org/10.9734/ejmp/2016/29040>
- Souilem, S., Fki, I., Kobayashi, I., Khalid, N., Neves, M. A., Isoda, H., Sayadi, S., & Nakajima, M. (2017). Emerging technologies for recovery of value-added components from olive leaves and their applications in food/feed industries. *Food and Bioprocess Technology*, 10(2), 229–248. <https://doi.org/10.1007/s11947-016-1834-7>
- Szutt, A., Dołhańczuk-Sródka, A., & Sporek, M., (2019). Evaluation of chemical composition of essential oils derived from different *Pelargonium* species leaves. *Ecological Chemistry and Engineering S*, 26(4), 807–816. <https://doi.org/10.1515/eces-2019-0057>
- Tazart, Z., Manganelli, M., Scardala, S., Buratti, F. M., Di Gregorio, F. N., Douma, M., Mouhri, K., Testai, E., & Loudiki, M., (2021). Remediation strategies to control toxic cyanobacterial blooms: Effects of macrophyte aqueous extracts on microcystis aeruginosa (growth, toxin production and oxidative stress response) and on bacterial ectoenzymatic activities. *Microorganisms*, 9(8), 1782. <https://doi.org/10.3390/microorganisms9081782>

## ETERIČNO ULJE *PELARGONIUM GRAVEOLENS* (GERANIJUMA), ANTIOKSIDANSI I ANTI-CIJANOBAKTERIJSKI/ANTI-ALGIJSKI SASTOJCI: SOLARNA TEHNIKA EKSTRAKCIJE I PREDVIĐANJE PRINOSA POMOĆU LINEARNE REGRESIJE

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**Sažetak:** Solarna hidrodestilacija (SHD) je predstavljena kao nova zelena tehnika za efikasnu ekstrakciju različitih fitohemikalija iz nusproizvoda hrane. Da bi bolje razumeli postupak, učinke i prednosti ovakvog zelenog i održivog izvora, cilj ove studije bio je da se uporedi efikasnost SHD-a u ekstrakciji eteričnih ulja iz *Pelargonium graveolens* (L'Hér), uz istovremeno oslobađanje antioksidativnih jedinjenja poput polifenola i flavonoida u preostaloj fazi solarnog destilatora. Prinos eteričnog ulja bio je 0,64% i 0,60% za SHD i konvencionalnu hidrodestilaciju (CHD), respektivno. Korišćenjem GC-MS analize identifikovane su 52 isparljive komponente. Citronelol (27,54 %–26,51 %), citronelil formiat (13,63 %–11,33 %), geraniol (11,94 %–10,97 %) i geranil formiat (8,31 %–5,84 %) predstavljali su glavne komponente za SHD i CHD ulja, respektivno. Za ekstrakte proizvedene SHD i maceracijom, određeni su ukupni fenolni jedinjenja (TPC), ukupni flavonoidni jedinjenja (TFC) i antioksidativna aktivnost (AA, %) na osnovu aktivnosti uklanjanja 2,2-difenil-1-pikrilhidrazil (DPPH) radikala. Rezultati su pokazali da je po sadržaju TPC, TFC i IC50 (249,72 mg EAG g<sup>-1</sup> DM, 205,88 mg EQ g<sup>-1</sup> DM i IC50=6,54 µg mL<sup>-1</sup>, respektivno), SHD ekstrakt proizveo izuzetno dobar prinos ekstrakcije. Štaviše, HPLC-UV analize su pokazale očuvanje u ekstraktu nakon SHD procesa nekih identifikovanih jedinjenja kao što su tirozol (31,71 mg g<sup>-1</sup> DM), galna kiselina (24,67 mg g<sup>-1</sup> DM), protokatehinska kiselina (22,59 mg g<sup>-1</sup> DM) i ferulinska kiselina (21,04 mg g<sup>-1</sup> DM). S druge strane, eterično ulje i ekstrakt pripremljen SHD-om pokazali su značajnu antibakterijsku aktivnost protiv dva testirana soja bakterija i alge *Microcystis aeruginosa* i *Chlorella* sp., sa prečnicima inhibicije rasta od 15,43 ± 0,32 mm, 16,73 ± 0,40 mm i 17,13 ± 0,35 mm, 25,96 ± 0,15 mm, respektivno, za eterično ulje i ekstrakt. Primećena je pozitivna linearna korelacija između antioksidanata, polifenola, flavonoida i antibakterijske aktivnosti za solarne ekstrakte. Rezultati su pokazali da je SHD dobra alternativa za očuvanje antioksidanata, polifenola, eteričnih ulja i antibakterijskih sredstava iz biljke *Pelargonium graveolens* (L'Hér).

**Ključne reči:** geranijum, solarna hidrodestilacija, eterično ulje, ukupni fenoli/flavonoidi, *Microcystis aeruginosa*, *Chlorella*

**Received:** 26 November 2025 / **Received in revised form:** 26 March 2026/19 May 2026 / **Accepted:** 19 May 2026

**Available online:** June 2026



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