Journal of Materials and Environmental Science
ISSN : 2028-2508
CODEN : JMESCN

J. Mater. Environ. Sci., 2020, Volume 11, Issue 10, Page 1689-1698

http://www.jmaterenvironsci.com

Using Anise (*Pimpinella anisum* L.) Essential Oils As Natural Herbicide

Kowthar G. El-Rokiek[1], Mohamed E. Ibrahim[2*], Samia A. Saad El-Din[1], Salma A. El-Sawi[3]

[1]Botany Department, National Research Centre, 12622 Dokki, Egypt, [2]Medicinal and Aromatic Plants Research Department, National Research Centre, 12622 Dokki, Egypt, [3]Pharmacognosy Department, National Research Centre, 12622 Dokki, Egypt

Received 26 Aug 2020,
Revised 01 Oct.2020,
Accepted 02 Oct 2020

Keywords
- *Essential oil,*
- *Pimpinella anisum,*
- waste oil,
- oil seed,
- weed growth ,
- wheat.

melsayed491@yahoo.com
Phone: +2o1224392974;

Abstract
Recycling of agricultural waste plays an important role in the disposal of plant residues and reduction of environmental pollution, with improving the economic situation in Egypt. This work represents new and unconventional methods of producing anise oil from dry post-harvest agricultural residues.The results of anise oil analysis showed that the anethole <trans> is the main compound in both oil extracted from seeds and waste, respectively. Also, all oils were rich in oxygenated monoterpenes (OM) compared to other terpene groups. In this investigation, allelopathic effects of essential oil extracted from seeds and waste of *Pimpinella anisum* L. seeds was tested against the two broad leaf weeds *Anagalis arvensis* and *Malva parviflora* that infested with wheat (*Triticum aestivum* L.) cv. Giza168 in pot experiment. This investigation was carried out in the greenhouse of National Research Centre, Dokki Egypt in the two successive winter seasons, 2017/2018 and 2018/2019. The pots were sprayed with *Pimpinella anisum* waste and seed essential oil at concentrations,1, 2 and 3 % for each. The results indicated suppression in growth of the two weeds, *Anagalis arvensis* and *Malva parviflora* by essential oil of both waste and seeds. The essential oils have high inhibitory effect against the two weeds at high concentrations specially with that of oil seeds. On the other hand, weed control by the studied essential oils were accompanied by increases in wheat growth as well as yield and yield components. Although seed oil of *P. anisum* induced more reduction in weed growth, the authors suggested using waste oil. The authors suggested commercial uses of waste oil as a bioherbicide.

1. Introduction
Weeds are unwanted plants that can grow in cultivated fields. These weeds compete with the main plants for the resources that plants need such as soil nutrients, water and space for growth, so, reduced growth and crop production [1-2]. The continuous use of herbicides resulted in the herbicidal resistance of weeds in addition to several consequences related to environment and human health. These concerns lead many workers to find out alternatives for chemicals (herbicides) that posses' good control and environmentally safe [3-5]. Essential oils are important natural resources that were found to possess phytotoxic activity [6-11].

Anise (*Pimpinella anisum* L.), is an annual important spice and medicinal plant related to the family Apiaceae (Umbelliferae) and native to the Mediterranean region. The fruits of *Pimpinella anisum* contain about 2 to 6% of an essential oil [12]. Anise seeds are an important natural raw material which is used for pharmaceutics, perfumery, food and cosmetic industries [13]. In addition, anise has antimicrobial, antifungal, insecticidal and anticorrosiove activities [14-18]. Concerning the effect on weed, in general the allelopathic effects of some Apiaceae (Umbelliferae) seeds such as fennel, cumin, caraway, celery, dill, anise and coriander are reported [19]. Azizi *et al.* [20] recorded suppression in germination of *Bromus tectorum*, *Centura ovina* and *Descurainia sophia* by *Bunium persicum* and *Cuminum cyminum* essential oils especially at high concentration. Also, it has been found that essential oils of *Coriandrum sativum* as well as *Cuminum cyminum* reduced seed germination of two weeds, *Lathyrus annuus* and *Vicia villosa*, furthermore, radicle growth was stopped after 7 days, and seedlings did not appear [21] Dhima *et al.* [22] concluded that *Pimpinella anisum* essential oil possesses strong allelopathic suppression against barnyard grass. De Almeida *et al.* [8] as well recorded reduction in germination and radicle elongation of *Raphanus sativus*, *Lactuca sativa* and *Lepidium sativum* by essential oil extracted from anise. Furthermore, Shokouhian *et al.* [23] reported that essential oil (25-50%) of *Pimpinella anisum* reduced seed germination of *Lactuca sativa*, *Piper longum* and *Solanum lycopersicum*.

The current work aims to control *Anagalis arvensis* and *Malva parviflora* associated wheat plants by natural materials present in essential oil extracted from anise (*Pimpinella anisum)* seeds.

2. Materials and methods

2.1 Plant material and isolation of essential oils*:*

The dry seeds samples of the anise plant growing in Assiut Egypt were taken in May (mature seeds stage) and the dry waste was taken after harvest. The essential oils of the dry plant materials were extracted by hydro-distillation for 3 hr. by Clevenger apparatus [24]. The essential oils were dehydrated over anhydrous sodium sulfate and subjected to GC/MS analysis.

2.2. Chemical analysis

2.2.1. Gas chromatography: GC analysis was performed using a Shimadzu GC- 9A gas chromatograph equipped with a DB5 fused silica column (30 m x 0.25 mm i.d., film thickness 0.25 μm). The oven temperature was held at 40°C for 5 min and then programmed until 250°C at a rate of 4°C/min. Injector and detector (FID) temperature were 260°C; helium was used as a carrier gas with a linear velocity of 32 cm/s.

2.2.2. Gas chromatography- mass spectrometry:

GC-MS analyses were carried out on a Varian 3400 system equipped with a DB-5 fused silica column (30 m x0.25 mm i.d.); Oven temperature was 40 to 240°C at a rate of 4°C/min, transfer line temperature 260°C, injector temperature 250°C, carrier gas helium with a linear velocity of 31.5 cm/s, split ratio 1/60, flow rate 1.1 ml/ min, Ionization energy 70 eV; scan time 1 s ; mass range 40-350 *m/z*..

2.2.3. Qualitative and quantitative analysis of essential oil:

Identifications were made by library searches [25] combining MS and retention data of authentic compounds by comparison of their GC retention indices (RI) with those of the literature or with those of standards available in our laboratories. The retention indices were determined in relation to a homologous series of n-alkanes (C8–C22) under the same operating conditions. Further identification was made by comparison of their mass spectra with those stored in NIST 98 and Wiley5 Libraries or with mass spectra from literature. Component relative concentrations were calculated based on GC peak areas without using correction factors.

2.3. Pot experiment:

The essential oils isolated from anise waste and seeds were dissolved in distilled water with the help of ethanol. The concentrations of the essential oils isolated from anise were prepared at1, 2 & 3(v/v) for waste and seeds [26].

Pot experiments were conducted in the greenhouse of the National Research Centre, Egypt for two winter seasons 2017/2018 and 2018/2019. Wheat cv. Giza168 was obtained from the Agricultural Research Centre, Egypt. The pots, 30 cm in diameter and 30 cm in height, contained equal amounts of sieved soil (2: 1 v/v clay and sand). Wheat grains were selected for uniformity by choosing those of equal size and with the same colour. Grains were sown 2 cm deep (8 grains in each pot) and allowed to germinate. All pots (except weed free treatment) were infested with the same weight of weed (0.03 g) of both *Anagalis arvensis* and *Malva parviflora* seeds and mixed thoroughly at a depth of 2 cm in the soil. Wheat grains and both weeds were sown at the same time. The cultivated wheat grains were thinned two weeks after sowing so that three homogeneous seedlings were left per pot. Irrigation and routine fertilizers were carried out. The experiment consisted of eight treatments including: two untreated controls, wheat only, wheat with *A. arvensis* and *M. parviflora* (unweeded treatment). The other six treatments were *Pimpinella anisum* oil at concentrations 1,2&3% (v/v) for waste and seeds. Each treatment was represented by 6 pots. The pots were distributed in a complete randomized design. Different concentrations (1-3% for each) of *Anise*oil were sprayed on the pots contained wheat plants and the two weed species at the rate of 60 ml /pot. The treatments were applied three times during three weeks starting from two weeks old plants. The data were taken at 45 days after sowing and at harvest.

Weeds data: In each season, weed samples were taken from each of the three pots 45 days after sowing and at harvest (all weed samples in each pot were pulled up). The fresh weights of *A. arvensis* and *M. parviflora* were recorded then were oven dried at 60°C for determination of dry weight (g/pot).

Wheat data:Three plants in each pot were taken for recording, plant height, number of leaves, as well as fresh and dry weight (g/plant) were recorded 45 days after sowing. At harvest, spike length, number of spikelets/spike, grain yield (g/plant) and 1000- grain weight (g) were determined**.**

2.4. Statistical analysis:

The obtained data were subjected to analysis of variance (ANOVA) by using completely randomized design and the Least Significant Difference (LSD) at the 5% probability level were calculated [27]**.**

3. Results and discussion

3.1. Essential oil percentage: The dry anise seeds gave the highest percentage of anise oil (1.15 %) while, the waste recorded 1.12 %.

3.2. Oil constituents (Table 1)**:** Trans-anethole was found as the main constituents of anise dry seed essential oil which recorded (65.56 %). Also, some other significant compounds were detected, i.e., estragole, isopentyl N-butyrate, eugenyl acetate, linalool, propyl butyrate and anethole <cis->. The main group in the dry anise seed oil was (OM). The total percentage of OM in this respect recorded (81.2%) against 1.11, 0.07, 0.31 and 10.31% for (MH), (SH), (OS) and (VC) group, respectively. Among the new compounds of anise seed oil identified in this work are methyl butyrate, propyl butyrate, isopentyl N-butyrate, myrtenal, p-anisaldehyde, methyl eugenol , cuparene , nuciferol <E-> and eugenyl acetate.

Table 1: Oil constituents of anise Seeds and waste oils

Compound	**KI**	**Seeds**	**Waste**	**Groups**
		%	**%**	
Methyl butyrate	724	0.03	-	VC
Propyl butyrate	896	3.01	-	VC
α–Pinene	939	0.06	0.03	MH
Sabinene	976	0.03	0.05	MH
Myrecene	991	0.02	0.03	MH
Limonene	1031	0.97	-	MH
Benzyl alcohol	1032	-	-	VC
Isopentyl, N-butyrate	1060	3.69	2.98	VC
Terpinene <gamma->	1062	0.03	0.02	MH
Linalool	1098	3.29	4.42	OM
Dihydro terpineol <cis.beta->	1136	-	0.02	VC
Myrtenal	1193	0.03	0.07	OM
Terpineol <alpha>	1189	0.02	-	OM
Myrtenol <cis>	1194	-	1.08	OM
Estragole	1195	10.83	11.01	OM
Cinnamaldehyde <Z->	1214	-	-	VC
p-Anisaldehyde	1252	0.14	-	VC
Anethole <cis>	1265	1.47	0.2	OM
Anethole <trans>	1283	65.56	62.69	OM
Methyl eugenol	1403	0.03	-	VC
Anisyl acetate <para->	1416	-	0.03	OM
Humulene <beta->	1440	-	0.07	SH
Humulene <alpha->	1454	-	0.04	SH
Ethyl cinnamate <E->	1462	-	0.13	VC
Germacrene D	1480	0.03	0.09	SH
Cuparene	1502	0.02	-	SH
Germacrene B	1556	0.02	0.05	SH
Cadinene <delta>	1524	-	-	SH
Eugenyl acetate	1525	3.41	2.31	VC
Santalol < Z, alpha-}	1678	-	-	OS
Coniferyl alcohol <E>	1729	-	-	VC
Nuciferol <E->	1758	0.31	-	OS
Total		**93.00**	**85.32**	

MH= Monoterpene hydrocarbons OM= Oxygenated monoterpenes SH = Sesquterpenes hydrocarbons VC= Various compounds OS= Oxygenated sesquterpenes

At the same time, anethole <trans> was found as the main constituent of anise waste oil which recorded 62.69%. Estragole , linalool, eugenyl acetate, isopentyl N-butyrate and myrtenal <cis-> were found as the main constituents of waste oil. Four chemical groups. Were recorded in anise waste oil. These groups were (MH), (OM), (SH) and (VC). β–humulene and α–humulene were found as new compounds in the anise waste oil.

Table 2 : Percentage of different chemical groups in anise seed and waste oils

Group	Seeds	Waste
Monoterpene hydrocarbon	**1.11**	**0.13**
Oxygenated monoterpenes	**81.20**	**79.5**
Sesquterpenes hydrocarbons	**0.07**	**0.25**
Oxygenated sesquterpenes	**0.31**	**0.00**

3.3. Weed growth: Table 3 show significant reduction in *Anagalis arvensis* weed fresh and dry weight by spraying anise waste oil or seed oil at 1-3% in comparison to the unweeded treatment. The applied waste oil at 3% controlled about 56% of the weed, *A. arvensis*. In addition, the seed oil of anise at 3% controlled about 70% of the same weed after 45 days from sowing. In respect to *M. parviflora*, the reduction in weed growth reached 70% of the unweeded control by waste oil at 3%. Seed oil of anise realized 80% reduction as corresponding result. At the end of the season, the treatment of waste oil at 3% controlled about 57% of *A. arvensis* and about 70 with seed oil. Waste and seed oil at 3% for each controlled about 70 and 80% *M. parviflora* as maximum in comparison to the unweeded control.

Table 3: Effect of anise seed oil on the growth of the two broadleaved weeds *Anagalis arvensis* and *Malva parviflora associated* wheat plants. (Average of the two seasons)

Treatments		Concentration (Percentage)	Weed growth [weight (g / pot)] 45 days after sowing *Anagalis arvensis* Fresh weight	*Anagalis arvensis* Dry weigh	*Malva parviflora* Fresh weight	*Malva parviflora* Dry weight	At the end of the season *Anagalis arvensis* Dry weight	*Malva parviflora* Dry weight
Wheat only		0	0.00	0.000	0.00	0.000	0.000	0.000
Wheat +*A. arvensis*+*M. parviflora*		0	11.27	2.789	15.26	2.546	24.166	43.235
***P. anisum* Waste**	**Wheat + *A. arvensis* + *M. parviflora***	1	9.42	2.063	10.26	2.176	20.303	35.100
		2	5.64	1.311	7.18	1.123	13.250	22.200
		3	5.39	1.212	5.42	0.727	10.203	10.333
***P. anisum* seed**	**Wheat + *A. arvensis* + *M. parviflora***	1	7.66	1.732	9.79	1.795	17.336	23.935
		2	4.76	0.836	5.88	0.591	8.681	10.726
		3	3.37	0.795	3.84	0.474	4.780	5.793
LSD at 5%			0.79	0.086	1.19	0.046	1.109	1.765

3.4. Wheat growth: In general, the data in Table 4 indicate that spraying wheat plants with waste and seed oil of anise induced significant increases in the recorded growth parameters 45 days after sowing. Plant height as well as number of tillers increased significantly over untreated control with 3% of waste oil and all concentrations (1-3%) of seed oil. Number of leaves has significant increase over unweeded control by waste and seed oil of anise at all concentrations (1-3%). Both fresh and dry weight increased significantly over unweeded control by spraying the oil at all concentrations. The number of tillers increased significantly over unweeded control by using waste oil at 2 and 3%. Seed oil realized corresponding results with all concentrations.

Table 4: Effect of anise seed oil on the growth of wheat plants 45 days after sowing (average of the two seasons)

Treatments		Concentration (Percentage)	Plant height (cm)	Number of tillers /plant	Number of Leaves /plant	Fresh weigh (g/plant)	Dry weight (g/plant)
Wheat only		0	33.66	3.00	12.66	1.151	0.280
Wheat+*A. arvensis*+*M. parviflora*		0	27.00	2.66	9.00	0.858	0.174
***P. anisum* Waste**	Wheat + *A. arvensis* + *M. parviflora*	1	28.33	3.00	10.66	0.965	0.194
		2	29.00	3.00	11.00	1.381	0.262
		3	30.33	3.50	12.00	1.780	0.325
***P. anisum* seed**	Wheat + *A. arvensis* + *M. parviflora*	1	35.00	3.00	11.00	1.372	0.207
		2	38.33	3.66	13.33	1.392	0.346
		3	40.66	4.00	13.33	2.433	0.599
LSD at 5%			2.32	0.44	1.26	0.197	0.065

3.5.Wheat yield: Spraying wheat plants with waste oil and seed oil of anise induced significant increases in spike length, number of spikes/ plant and number of spikelets/spike at all concentrations (1-3%) used Table 5 in comparison to the unweeded control. Grain yield /plant as well as weight of 1000 grains revealed significant increases as well over unweeded control by using all concentrations. The grain yield/plant in pots treated with anise seed oil at 3% reached to 90% over its correspondence in unweeded pots which represented the maximum value, followed by that treated with anise waste oil at 3% (70% over unweeded pots). Weight of 1000 grains recorded corresponding maximum results, 95% increase and 75 %.

Table 5: Effect of anise seed oil on yield and yield components of wheat plants (average of the two seasons)

Treatments		Concentration (Percentage)	Spike length (cm)	No. spikes/plant	No. Spiklets/spike	Weight of grains/plant (g)	Weight of 1000 grains (g)
Wheat only		0	10.33	6.56	20.00	9.560	38.93
Wheat+*A. arvensis* +*M. parviflora*		0	6.04	4.93	15.00	5.860	23.75
***P. anisum* Waste**	**Wheat + *A. arvensis* + *M. parviflora***	1	10.22	5.13	16.66	9.478	37.48
		2	10.50	5.63	19.00	9.597	39.36
		3	10.66	5.96	18.33	10.05	41.68
***P. anisum* seed**	**Wheat + *A. arvensis* + *M. parviflora***	1	10.16	6.71	19.66	10.45	39.85
		2	10.83	6.92	23.33	10.87	44.95
		3	12.50	7.00	24.66	11.16	46.49
LSD at 5%			0.39	0.24	1.42	0.40	2.58

4. Discussion

Comparing the oils of the seeds and waste, it was found that, the main constituent was trans-anethole in the two oils. Its concentrations were the highest in the seeds oil followed by the waste oil. Similar results were reported by Ibrahim *et al.* [27] who stated that trans anethole was the main constituent of anise seeds oil. Some minor compounds found in anise seed oil as methyl butyrate, terpineol, p-anisaldehyde, methyl eugenol and nuciferol [E-] The composition of the anise waste oil was closer to that of the anise seed oil. This was also obvious in the percentage of the major group (OM) and that of (SH) which means that waste oil be similar to the seed oil to a great extent.

Natural products have the potential to provide efficient and safer herbicides for humans and the environment. Essential oils extracted from different plants are natural materials that have allelopathic effects on different species [17,11].

The results in the present study showed satisfactory control for the two weed species, *A. arvensis* and *M. parviflora* by spraying waste and seed oil of anise. Controlling these two weeds infested in wheat plants increased with increasing in concentrations of the two oils up to 3%. The inhibition in the two-weed growth was higher with anise seed oil Table 3. The inhibition of *M. parviflora* was higher than *A. arvensis*. Using essential oil as allelopathic materials that have herbicidal potential were documented by several workers [6,8&9]. The results in Table 3 that indicated growth inhibition of the two weeds, *A. arvensis* and *M. parviflora* came in accordance with that documented by De Almeida *et al.*[8] who recorded reduction in germination and radicle elongation of *Raphanus sativus*, *Lactuca sativa* and *Lepidium sativum* by essential oil extracted from anise. The results also coincided with that reported by Shokouhian *et al.*[23] that essential oil (25-50%) of *Anise* reduced seed germination of *Lactuca sativa*, *Piper longum* and *Solanum lycopersicum*.

The inhibition in weed growth in the current results may be explained on the bases of different kinds of monoterpenes present in plant essential oils. The main constituent was trans-anethole in the two oils Table 1 that is related to OM (oxygenated monoterpenes. It has been documented that monoterpenes have phytotoxic potential against germination and growth of plants [28]. Terpenes represent the largest and most diverse group of secondary metabolites, and the most abundant constituents of essential oils [29,30]]. Although The composition of the anise waste oil was closer to that of the anise seed oil, the inhibition of the two weed species caused by seed oil was higher, perhaps this may be attributed to the presence of the fraction limonene which present in seed oil only. Andrianjafinandrasana *et al.* [31] documented that Limonene oil at 1000µ l/L inhibited the germination (57 %) of garden cress (*Lepidium sativum*) compared to control. Sabinene and limonene oils at 1000µl/l completely prevented the germination of green gram (*Vigna radiata*). Sabinene and limonene oils at 1000µl/l completely prevented the germination of green gram and caused 100% mortality.

Furthermore, it is worthy to mention that trans-anethole, the main compound in anise essential oil has strong antifungal activity through the inhibition of the mycelial growth of a wide range of fungi [32,33,16].

The results in Table 1 and 2 show that the main group in the dry anise seed oil was oxygénâted monoterpene which came in accordance with the findings of several workers who documented that the herbicidal activity of essential oils has been linked to the presence of oxygénâted monoterpenes [29,34,35.36,37]. The results confirmed by Vasilakoglou *et al.*[38] who Stated that trans-anethole has high phytotoxic effect on rigid ryegrass (*Lolium rigidum*). The present results are in consistence with the Recent work by El-Sawi *et al.*[11] who attributed the inhibition of germination and growth of purslane by *Citrus senensis* essential oil to the presence of oxygenated monoterpenes. The difference in response of the two weeds to the essential oils of anise as the reduction in *M. parviflora* is higher than *A. arvensis* may be attributed to the selectivity of essential oils as have been reported by Vokou *et al.* [39]. The authors suggested the important of this feature, specially when used as a bioherbicide.

Controlling *M. parviflora* and *A. arvensis* by anise seed oil Table 3 increased the competition of wheat plants against these two weeds which make more nutrients absorbed by the target plant (wheat), consequently, the inhibition in the two weed growth was concomitant with an increase in wheat growth and yield Tables 4,5. In consistence, plant height, number of leaves / plant as well as dry weight / plant increased over the unweeded control. The increase in wheat growth in turn resulted in enhancement in wheat yield and its components that represented by spike length, number of spiklets /spike, weight of grains/ plant and weight of 1000 grains were attained increases. In general, a reduction in weed infestation increased crop yield [2,40].

Conclusion

The current results aim to utilize natural materials in order to control weed growth and produce an environment free from hazards. So, the present experiment was carried out for this purpose using anise seed oil and waste. The results revealed satisfactory control for *Anagalis arvensis* and *Malva parviflora* with anise seed oil and waste. So, the results suggested using anise seed and waste essential oil as bioherbicides. The authors suggested using of waste oil in weed control to realize double purposes (controlling weeds and getting rid of waste).

Acknowledgment-The authors are thankful to the National Research Centre, Egypt, for providing materials and facilitating this work.

References

1. M. Kolahi B. Peivasegan, I. Hadizadeh and S. M. Seyyednejad, . Inhibition of germination and seedling growth of wild oat by rice hull extracts, *J. Applied Sci*, 9 (2009) 2857–2860. DOI: 10.3923/jas.2009.2857.2860
2. C. B. Singh, M. Hashim, A. Kumar, R. Rishi and U. C. Pandey, Performance of improved wheat (Triticum aestivum) varieties under various planting techniques in north eastern plain zone of India. *Journal of Community Mobilization and Sustainable Development*, 11. (2016) 145-149.
3. I. M., K. H. Kim Chung, J. K. Ahn, S.C. Chun, C. S. Kim and S. H. Kim, Screening of allelochemicals on barnyardgrass (*Echinochola crus-galli*) and identification of potentially allelopathic compounds from rice (Oryza sativa) variety hull extracts. *Crop Protection*, 21 (2002) 913–920. https://doi.org/10.1016/S0261-2194(02)00063-7
4. M. M. Hegab, S. E. A. Khodary, O. Hammouda and H. R. Gharieb. 2008. Autotoxicity of chard and its alllopathic potentiality on germination and some metabolic activities associated with growth of weed seedlings. *Afr. J. Biotechnol*, 7 (2008) 884–892. DOI: 10.5897/AJB
5. K. G. El-Rokiek, S. A. Saad El-Din, A. N. Shehata and S. A. M. El-Sawi, 2016. A study on controlling *Setaria viridis* and *Corchorus olitorius* associated with *Phaseolus vulgaris* growth using natural extracts of Chenopodium album. *J. Plant Protec. Res.*, 56 (2016) 186-192. https://doi.org/10.1515/jppr-2016-0031.
6. S. Azirak and S. Karaman, 2008. Allelopathic effect of some essential oils and components on germination of weed species. *Acta Agriculturae Scandinavica, Section B — Soil & Plant Science*, 58 (2008) 88-92. https://doi.org/10.1080/09064710701228353
7. F. E. Dayan, C. L. Cantrell and S. O. Duke, Natural products in crop protection. *Bioorg. Med. Chem. Lett.*, 17(2009) 4022–4034. DOI: 10.1016/j.bmc.2009.01.046
8. R, L. De Almeida Fernando, F. Fernando, E. Mancini, V. de Feo, Phytotoxic activities of mediterranean essential oils. *Molecules* 15(2010) 4309–4323. doi:10.3390/molecules24061136
9. A. M. S. Soliman and M. M. Z. Masoud, Comparative study on composition and allelopathic effect of volatile oils extracted from two thymus species of the gebel akhder in libya. Int'l *Journal of Advances in Chemical Engg., & Biological Sciences (IJACEBS)*, 1 (2014) 67-70. http://dx.doi.org/10.15242/IJACEBS.C514052

10. M. D. Ibáñez and M. A. Blázquez, Phytotoxicity of essential oils on selected weeds: potential hazard on food crops. *Plants*, 7(2018) 79-93. Doi: 10.3390/plants7040079
11. S. A. El Sawi, M. E. Ibrahim, K.G. El-Rokiek and S. A.Saad El-Din , Allelopathic potential of essential oils isolated from peels of three citrus species. *Annals of Agricultural Science*, 64(2019)89-94. https://doi.org/10.1016/j.aoas.2019.04
12. J. G. Lee, Y. J. Known, H. J., Jang, J. J. Kwag, O. C. Kim and Y. H. Choi, A comparison of different extraction methods for volatile components of anise (*Pimpinella anisum* L.). *Agric. Chem. Biotechnology*, 40(1997) 144-147.
13. Ross IA. 2001. Medicinal plants of the world: chemical constitutes, traditional and modern medicinal uses, Volume 2. Humana press, Totowa, New Jersey, pp. 363-374.
14. B. Tepe ,AH. Akpulat , M.Sokmen , D. Daferera , O.Yumrutas , E.Aydin , M. Polissiou and M.Sokmen . Screening of the antioxidtaive and antimicrobial properties of the essential oil of *Pimpinella anisum* and Pimpinella flabellifolia from Turkey. *Food Chemistry*, 97 (2006) 719-724. https://doi.org/10.1016/j.foodchem.2005.05.045
15. C. R. Tirapelli, , C. R. Andrade, A. O. de Cassano, F. A. de Souza, S. R. Ambrosio, F. B. Costa and A. M. da Oliveria, Antispasmodic and relaxant effects of the hydroalcoholic extract of *Pimpinella anisum* (Apiaceae) on rat anococcygeous smooth muscle. Journal of Ethnopharmacology, 110 (2007) 23-29. https://doi.org/10.1016/j.jep.2006.08.031
16. AI. Hussain, F. Anwar, PS.Nigam, M. Ashraf and AH. Gilani, Seasonal variation in content, chemical composition and antimicrobial and cytotoxic activities of essential oils from four Mentha species. *J Sci Food Agric*, 90 (2010) 1827–1836. https://doi.org/10.1002/jsfa.10098
17. H. Abo El Makarem, S. E. El Kholy, A. Abdel-Latif and A. Seif, . Effect of some botanical oils on the granary weevil, *Sitophilus granarius* (L.) (*Coleoptera*: *Curculionidae*). *Egypt. J. Exp. Biol. (Zool.)*, 13 (2017) 273 – 282. 10.5455/egysebz.20171025013938
18. N. Lotfi, H. Lgaz, M. Belkhaouda, M. Larouj, R. Salghi, S. Jodeh, H. Oudda, B. Hammouti, Effect of anise oil as a green inhibitor on steel corrosion behavior, *Arab. J. Chem. Environ. Res.* 01 (2014) 13-23
19. M. Azizi and Y. Fuji, 2006. Allelopathic effect of some medicinal plant substances on seed germination of *Amaranthus retroflexus* and *Portulaca oleraceae*. *Acta Horticulturae*, 699 (2006) 61-67. DOI: 10.17660/ActaHortic.2006.699.5
20. M. Azizi, L. Alimoradee and M. H. Rashedmohasse, Allelopathic effects of *Bunium persicum* and *Cuminum cyminum* essential oils on seed germination of some weeds species. *Iranian Journal of Medicinal and Aromatic Plants*, 22 (2006)198-208.
21. A. R. Rahimi, S. J. Mousavizadeh, H. Mohammadi, A. Rokhzadi, M. Mohammad Majidi and S. Amini, Allelopathic effect of some essential oils on seed germination of *Lathyrus annuus* and Vicia villosa. *J. Bio. & Env. Sci.*, 3 (2013) 67-73.
22. K. Dhima, L. Vasilakoglou, V. Garane, C. Ritzoulis, V. Lianopoulou and L. Panou-Philotheou, Competitiveness and essential oil phytotoxicity of seven annual aromatic plants. *Weed Sci.*, 58 (4) (2010) 457-465. https://doi.org/10.1017/wsc.2019.61
23. A. Shokouhian, H. Habibi and K. Agahi, Allelopatic effects of some medicinal plant essential oils on plant seeds germination. *J. BioSci. Biotech*, 5 (1) (2016) 13-17.
24. J. F. Clevenger, "Apparatus for the Determination of Volatile Oil," *JAm Pharm Assoc.*, 17 (1928) 346-349.
25. R.P. Adams, Identification of Essential Oil Components by Gas chromatography Mass Spectroscopy. *Allured Publishing, Carol Stream, Illinois and U.S.A.*1995 ISBN:(0- 931710-42-1)

26. Ibrahim, M. E., M. A. Mohamed, H. E. Wahba and S. A. El-Sawi, Unconventional Source of Anise Volatile Oil. J. Mater. Environ. Sci., 8 (2017) 3968-3972
27. GW.Snedecor, and W. G. Cochran, Statistical Methods, 7th ed. The Iowa State University Press, Ames, Iowa, IA, USA, (1980)pp. 507.
28. F. E.Dayan, A. Hernandez, S. N. Allen, R. M. Moraes, J. A. Vroman, M. A. Avery and S. O. Duke, Comparative phytotoxicity of artemisinin and several sesquiterpenes analogues. *Phytochemistry*, 50 (1999) 15-24. https://doi.org/10.1016/S0031-9422(98)00568-8
29. J. Gershenzon and N. Dudareva, 2007. The function of terpene natural products in the natural world. *Nature Chemical Biology*, 3(2007) 408-414. http://dx.doi.org/10.1007/s10886-011-9919-2.
30. H.Yoshimura, Y. Sawai, S. Tamotsu and A. Sakai, 1,8-Cineole inhibits both proliferation and elongation of BY-2 cultured tobacco cells. *J Chem Ecol*, 39(2011) 320-328.
31. S. N. Andrianjafinandrasana, H. S. Andrianoelisoa, M. L. Jeanson, R. I. Ratsimiala and P. Danthu, . Allelopathic effects of volatile compounds of essential oil from Ravensara aromatica Sonnerat chemotypes. *Allelopathy J.*, 31(2013):\333-344
32. Shukla, H. S. and S. G. Tripathi, Antifungal substance in the essential oil of anise (*Pimpinella anisum* L.). *Agric. Biol. Chem*, 51 (1987) 1991–1993. https://doi.org/10.1080/00021369.1987.10868311
33. M. M. Özcan,. and J. C. Chalchat, Chemical composition and antifungal effect of anise (*Pimpinella anisum* L.) fruit oil at ripening stage. *Ann Microbiol*, 56 (2006) 353–358. https://doi.org/10.1007/BF03175031
34. R. .Kotan, R, A. Cakir, F. Dadasoglu, T. Aydin, R. Cakmakci, H. Ozer, S. Kordali, E. Mete and N. Dikbas, Antibacterial activities of essential oils and extracts of Turkish Achillea, Satureja and Thymus species against plant pathogenic bacteria. *J Sci Food Agric*, 90 (2010) 145-160. DOI: 10.1002/jsfa.3799
35. B.Teixeira, A. Marques, C. Ramos, C. Serrano, O. Matos, N. R. Neng, J. M. F. Nogueira, J. A. Saraiva and M. L. Nunes, Chemical composition and bioactivity of different oregano (Origanum vulgare) extracts and essential oil. *J Sci Food Agric*, 93(2013)2707–2714. DOI: 10.1002/jsfa.6089
36. B. L. Yan, P. Gauthier, K.K. Keefover-Ring and J. D. Thompson, Variable Phytotoxic Effects of *Thymus vulgaris* (Lamiaceae) Terpenes on Associated Species. *Int. J. Plant Sci*, 176 (2015) 20-30. https://doi.org/10.1086/678772
37. A. R. Ersilia, A., Sumalan, C. Danciu, D. Obistioiu, M. Negrea, M. Poiana, C. Rus, I. Radulov, G. Pop and C. Dehelean, Synergistic antifungal, allelopathic and antiproliferative potential of Salvia officinalis L., Thymus vulgaris L. essential oils. *Molocules*, 23 (2018) 1–15. DOI: 10.3390/molecules23010185
38. I. Vasilakoglou, K. Dhima, K. Paschalidis and C. Ritzoulis , Herbicidal potential on *Lolium rigidum* of nineteen major essential oil components and their synergy. *J. Ess. Oil Res.*, 25 (2013) 1-10. https://doi.org/10.1080/10412905.2012.751054
39. DVokou, P. Douvli, G. J. Blionis and J. M. Halley, Effects of monoterpenoids, acting alone or in pairs, on seed germination and subsequent seedling growth. *J Chem Ecol*, 29 (2003) 2281-230. https://doi.org/10.1023/A:1026274430898
40. D. Dey, D. Nath, P. B. Jamatia, Effect of raised bed planting method of maize under sandy loam soil of west Tripura. *Int. J. Appl. Res.* 1 (2015) 561-563.

(2020) ; http://www.jmaterenvironsci.com