Allelopathic Effect of Ecofriendly Botanical Extracts and Application of Vinasse as Alternative Source of Mineral Potassium Fertilizers on Yield and Oil Quality of Caraway Plant

Gehan F. Massoud¹, A.S. Dapor¹ and A.M. El-Shoura²

¹Medicinal and Aromatic Plants Research Department, Horticulture Research Institute, ARC, Giza, Egypt.
²Cross pollinated vegetables Research Department, Horticulture Research Institute, ARC, Giza, Egypt.

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ABSTRACT

A field trial was performed during two consecutive winter seasons of 2017 and 2018 at a private farm located in Kafr El Wekala, Sherbin, El Dakahlia Governorate, Egypt to evaluate the allelopathic effect of garlic and licorice extracts at 0, 1, 2 and 4 % and application of vinasse as organic fertilizer at V₁ (50%) and V₂ (100%) from the recommended dose of potassium fertilizer on growth of caraway plants, seed measurements, essential oil productivity as well as N, P, K, Ca, Mg and Fe contents of caraway seeds. The obtained results showed that garlic and licorice extracts under both the chemical fertilizer NPK and levels of vinasse possess the positive allelopathic effect on growth and the yield of caraway. Vinasse at two levels resulted in significant increments on all studied parameters compared to chemical fertilizer. Furthermore, the plants treated with the high rate of vinasse (100%) in combinations with licorice extracts at 2% in both seasons recorded the maximum means values for growth characters, plant dry weight, seed weight per plant subsequently seed yield per fed. and essential oil production. Moreover, Vinasse (100%) in combinations with garlic extracts at 4%, significantly increased the chemical constituents of caraway seeds compared to the control and other treatments in both seasons. GCM analyses for oil showed that a total of 10 compounds, the main component was carvone which constitutes the maximum percentages in plants treated with vinasse (100%) in combination with garlic foliar application at all levels. The obtained data revealed that the best growth, essential oil and chemical composition were obtained from treated plants with vinasse (100%) from the recommended dose of K in combination with licorice extracts at 2% and garlic extract at 4% which had attained the uppermost values of yield and quality of caraway compared to either control or other treatments. Therefore, we recommend spraying caraway plants with eco-friendly, organic and costless extract of licorice at 2% and replacement of the mineral K fertilizers by vinasse (100%) as an organic potassium source without adverse effects on caraway yield and oil quality.

Keywords: Carum carvi L., vinasse, allelopathic effect, garlic, licorice extracts, yield, quality of oil

Introduction

The production of safe and healthy foods using sustainable and environmentally friendly agricultural practices plays a vital role in determining their market value and nutritional benefits. For increasing productivity, a new strategy is using organic fertilizers and growth stimulating compounds that should be safe and inexpensive.

Caraway (Carum carvi L., Apiaceae) is one of the earliest cultivated herbs in Africa, Europe and Asia (Kamenik J., 1996). It was used in folk medicine, recently caraway has good antioxidant activity (Samojlik et al. 2010), possesses anti fungal and antibacterial properties (Gupta et al. 2011). Also, it exerted antimicrobial, anticancer, anti diabetic, diuretic, gastrointestinal, bronchial relaxant effects and other pharmacological activities. It improves lactation in nursing mothers. In addition, it was used as a potato sprouting inhibitor (Şanli et al. 2010). The main criteria for determining the quality of caraway is its high content of essential oil, which is obtained by distillation of the dried fruit and gives its characteristic aroma (Sachan et al., 2010). Fruits are used as a spice in food due to its

Corresponding Author: Gehan, F. Massoud, Medicinal and Aromatic Plants Research Department, Horticulture Research Institute, ARC, Giza, Egypt. E-mail: gehan.fawzy75@gmail.com
mechanical and chemical properties of soil are presented in Table 1. Analysis and Improvement, using standard method designated by Page caraway at 30 cm depth from soil surface before planting were ana as well as N, P, K, Ca, Mg and Fe contents of caraway seeds. The soil samples supporting growth of recom 1, 2 and 4% under two levels of vinasse as organic fertilizer at V Longitudes 31° 20' and 32° E), Egypt to evaluate allelopathic effect of garlic and licorice extracts at 0, 1, 2 and 4% under two levels of vinasse as organic fertilizer at V1 (50%) and V2 (100%) from the recommended dose of K on growth of caraway plants, seed measurements, essential oil productivity as well as N, P, K, Ca, Mg and Fe contents of caraway seeds. The soil samples supporting growth of caraway at 30 cm depth from soil surface before planting were analyzed at Mansoura Center of Soil Analysis and Improvement, using standard method designated by Page et al. (1982). Some mechanical and chemical properties of soil are presented in Table 1.

Materials and Methods

To achieve the goal of this investigation, field trial was conducted during two successive winter seasons of 2017 and 2018 at a private farm situated in Kafr El Wekala, Sherbin, El Dakahlia Governorate which is located in northeast of the Nile Delta region (Latitudes 30° 30’ and 31° 30’ N; Longitudes 31° 20’ and 32° E), Egypt to evaluate allelopathic effect of garlic and licorice extracts at 0, 1, 2 and 4% under two levels of vinasse as organic fertilizer at V1 (50%) and V2 (100%) from the recommended dose of K on growth of caraway plants, seed measurements, essential oil productivity as well as N, P, K, Ca, Mg and Fe contents of caraway seeds. The soil samples supporting growth of caraway at 30 cm depth from soil surface before planting were analyzed at Mansoura Center of Soil Analysis and Improvement, using standard method designated by Page et al. (1982). Some mechanical and chemical properties of soil are presented in Table 1.
Healthy seeds of caraway were attained from Medicinal and Aromatic Plants Dept. of Hort. Res. Inst., Agric. Res. Center. Seeds were sown on 17th and 19th October on two seasons, respectively in hills (30 cm) in between and on rows (60 cm) apart. The experimental unit area was 4.2 m². Plants were thinned to one plant per hill. The Experimental design was a factorial experiment (3 X 7) in split plot design with three replicates. The main plots were assigned to a different application of chemical and vinasse fertilizers at 50% and 100% vinasse of recommended dose of K fertilizer, sub plots were devoted to the foliar spray of natural extracts of Glycyrrhiza glabra and Glycyrrhiza glabra plants at (0, 1, 2 and 4%). The climatic conditions of the meteorological station within the experimental region were analyzed, maximum and minimum values were noted daily then recorded as mean / month (Table 2).

Control treatment was supplemented with mineral NPK fertilizer according to the recommendations of the Ministry of Agriculture; 200 kg/fed calcium superphosphate (15.5 % P₂O₅) during seed-bed preparation, nitrogen fertilizer 200 kg/fed ammonium sulfate (20.5 % N) which was added in two portions, half with all amounts of K fertilizer 50 kg/fed potassium sulfate (48 % K₂O) after thinning of plants and the second after 45 days of the first. All treatments were fertilized by a recommended dose N and P fertilizers, while K fertilizer was added from vinasse at 50 % and 100 % of the potassium recommended dose of caraway.

Table 1: Some mechanical and chemical properties of the experimental soil.

| Parameters | Clay | Silt | Sand | Organic matter (%) | CaCO₃ (%) | pH | E.C. (mmols/cm) |
|------------|------|------|------|-------------------|-----------|----|----------------|
| Soil fractions (%) | 2017 | 2018 | 2017 | 2018 | 2017 | 2018 | 2017 | 2018 |
| Clay | 44.48 | 45.12 | 1.47 | 1.66 |
| Silt | 31.02 | 32.88 | 2.31 | 2.54 |
| Sand | 23.60 | 21.04 | 8.1 | 7.87 |
| Soil texture | Clay loamy | Clay loamy | Na⁺ | 0.18 | 0.17 |
| N | 43.60 | 47.32 | Ca²⁺ | 3.02 | 3.00 |
| P | 9.03 | 11.00 | Mg²⁺ | 0.25 | 0.26 |
| K | 230.00 | 239.00 | HCO₃⁻ | 0.50 | 0.70 |
| Fe | 3.17 | 3.11 | Cl⁻ | 0.40 | 0.36 |
| Zn | 1.27 | 1.28 | SO₄²⁻ | 0.45 | 0.38 |
| Mn | 1.31 | 1.32 | CO₃⁻ | - | - |

Table 2: The climatic records of the meteorological station at El-Mansoura city during the two growing seasons (2017 and 2018).

| Month | 1st season (2017-2018) | 2nd season (2018-2019) |
|-------|------------------------|------------------------|
|       | Temp. C | Rh % | Ev mm/day | Temp. C | Rh % | Ev mm/day |
| Oct.  | Max | Min. | Mean | Max | Min. | Mean | Max | Min. | Mean | Max | Min. | Mean | Max | Min. | Mean | Max | Min. | Mean |
| Nov.  | 22.6 | 13.3 | 18.0 | 94.2 | 41.9 | 68.1 | 3.1 | 23.0 | 17.1 | 20.1 | 83.4 | 49.2 | 66.3 | 4.2 |
| Dec.  | 20.2 | 11.9 | 17.0 | 90.5 | 46.8 | 68.7 | 2.5 | 22.0 | 14.8 | 18.4 | 82.2 | 51.7 | 67.0 | 2.8 |
| Jan.  | 18.0 | 8.0 | 13.0 | 86.0 | 37.9 | 62.0 | 2.3 | 19.7 | 11.1 | 15.4 | 93.8 | 50.5 | 72.2 | 2.5 |
| Feb.  | 18.5 | 8.2 | 13.4 | 87.6 | 34.1 | 60.9 | 2.1 | 22.7 | 11.8 | 17.3 | 93.1 | 45.3 | 69.2 | 2.6 |
| Mar.  | 22.7 | 8.8 | 15.8 | 87.9 | 29.7 | 58.8 | 2.7 | 23.7 | 12.9 | 18.3 | 93.1 | 42.4 | 67.8 | 3.3 |
| Apr.  | 26.8 | 14.6 | 20.7 | 93.2 | 34.8 | 64.0 | 4.6 | 26.6 | 13.4 | 20.0 | 89.6 | 38.7 | 64.2 | 4.3 |
| May.  | 27.5 | 15.9 | 21.7 | 93.3 | 31.0 | 62.2 | 4.8 | 29.5 | 17.4 | 23.5 | 83.6 | 38.7 | 61.2 | 5.4 |
| Jun.  | 32.1 | 20.1 | 26.1 | 90.7 | 28.4 | 59.6 | 5.5 | 33.7 | 22.5 | 28.1 | 83.0 | 37.6 | 60.3 | 6.2 |

Temp.: temperature Rh: Relative humidity Ev: Evaporation

Vinasse treatments were sprayed two weeks after planting on soil surface three times along the period of plant growth after (2, 4 and 6 weeks) from planting as 375 L fed⁻¹ (50%) and 750 L fed⁻¹ (100%) from the recommended dose. Entirely agricultural practices were performed according to the recommendations of Egyptian Ministry of Agriculture. Caraway required from 5 to 7 irrigations till the umbels were filled with mature seeds. Vinasse was obtained from the integrated industries and...
Foliar treatments with licorice root and garlic extracts were applied three times at 30, 45 and 60 days from planting. The aqueous extract of licorice roots was prepared by blending (10, 20 or 40g) in one litre of distilled water 50°C for 24 hours. The solution filtered by wringing using a mutton cloth. The obtained extract re-filtered through Whatman No. 2 filter paper and completed by distilled water to one litre. Egyptian licorice roots, according to Morsi et al. (2008) contains K (480.2), Ca (984.0), Mg (473.5), Fe (25.27) and Mn (1.25) as well as total phenols (405.0) and total flavonoids (114.9) mg/100g dry weight. The 1, 2 and 4% garlic aqueous extract was prepared by blending 10, 20 and 40 g of fresh mature cloves in one litre of distilled water, frozen and thawed two times, and then filtered and diluted by distilled water to one litre (El-Desouky et al., 1998). Garlic bulbs as mentioned by Duke (1992) contain minerals as K (3,730-13,669), Mg (240-1,210), Fe (15-129), Ca (180-4,947), B (3-6) and Cr (2.5-15)ppm; amino acids such as Arginine (6,340-11,736), Methionine (760-1,824), Phenylalanine (1830-4,392) and Proline (1,000-2,400)ppm; vitamin such as ascorbic acid (100-788) ppm; terpenes and their derivatives as well as growth regulators such as gibberellin A3.

The following parameters were recorded as average of plant height (cm), number of branches per plant, plant dry weight (g), number of umbels per plant and number of umbelet per umbels. Some seed measurements; Dry weight of seeds (g plant⁻¹), Yield of dry seeds (kg fed⁻¹), thousand seed weight (g), harvest index (the ratio of seed yield per plant to the total above ground according to Ullah et al., 2006). The harvest took place in 20th and 25th June for both seasons manually by cutting plants then drying them up. Ripe caraway was threshed in a small plot harvester. For the chemical analyses, representative samples of seeds each were digested in a mixture of concentrated sulfuric acid and perchloric acids. Total nitrogen was determined in dried samples (A.O.A.C., 1990). Phosphorus was determined colorimetrically according to Murphy and Riley (1962). K, Mg, Ca and Fe were determined according to Chapman and Pratt, 1961. Essential oil determinations; the dried seeds of caraway were hydrodistilled in a Clevenger's type apparatus. Yellow coloured oil, with characteristic odour and the sharp taste was obtained. It was dried over anhydrous sodium sulphate to eliminate traces of moisture and stored in a refrigerator in the dark at 4°C until use. The essential oil percentage was calculated owing to the method described by British Pharmacopoeia (2000). Oil yield (ml plant⁻¹) and total yield (kg fed⁻¹) were calculated. The analysis of volatile oil of interaction treatments was performed separately with a Hewlett-Packard model 5890. A fused silica capillary column carbowax 20 M measuring 20 m x 0.32 mm internal diameter, the film thickness of 0.17µm. The temperature program adopted was maintained at 75°C for 5 min. with an increase of 4°C/min until 220°C (10 min.). The carrier gas was Helium and the working flow rate was 1.0 ml/min, the detector was 9144 HP. The temperature of the detector and injector were adopted at 280°C and 240°C, respectively. The identification of the separated compounds was achieved by matching their retention times with those of authentic samples injected under the same conditions. Statistical analysis was carried out according to Gomez and Gomez (1984) using CoStat. The obtained data were subjected to analysis of variance (ANOVA) and the means were compared using the least significant differences (L.S.D) at 5%.

Results and Discussion

1- Vegetative growth characters

Regarding to impact of NPK as chemical fertilization, vinasse as organic fertilization and allelopathic effect of garlic and licorice extracts on dry weight and some vegetative growth parameters of caraway, obtained results in Table (4) indicated that all applied treatments stimulated...
Table 4: Effect of vinasse and natural extracts of garlic and licorice on some vegetative growth characters and plant dry weight of *Carum carvi* L. during the two seasons of 2017/2018 and 2018/2019.

| Fertilizer(F) | Extracts(E) | Plant height(cm) | Branches No. / plant | Umbel No. / plant |
|---------------|-------------|------------------|----------------------|-------------------|
|               | NPK         | Vinasse 50%      | Vinasse 100%         | Mean              | NPK       | Vinasse 50% | Vinasse 100% | Mean     |
| **First season (2017/2018)** |             |                  |                      |                   |           |            |            |          |
| 0             | 88.90       | 100.87           | 101.93               | 97.23             | 11.93      | 16.93       | 17.33       | 15.40    |
| 1             | 92.83       | 107.27           | 109.80               | 103.30            | 12.30      | 17.40       | 17.83       | 15.84    |
| 2             | 94.63       | 110.80           | 117.90               | 107.78            | 12.47      | 18.33       | 19.47       | 16.76    |
| 4             | 95.73       | 112.07           | 120.13               | 109.31            | 12.93      | 18.93       | 20.07       | 17.31    |
| 1             | 89.47       | 100.87           | 110.60               | 100.31            | 12.37      | 17.47       | 18.43       | 16.09    |
| **Mean**      | 94.00       | 108.52           | 115.10               | 13.01             | 18.25      | 19.13       | 18.33       | 15.32    |
| **Second Season (2018/2019)** |             |                  |                      |                   |           |            |            |          |
| 0             | 73.90       | 95.43            | 96.80                | 88.71             | 11.43      | 13.90       | 14.87       | 13.40    |
| 1             | 73.90       | 97.07            | 99.90                | 90.29             | 11.50      | 16.00       | 17.87       | 15.12    |
| 2             | 79.87       | 105.70           | 113.80               | 99.79             | 12.30      | 18.67       | 19.83       | 16.93    |
| 4             | 83.30       | 108.07           | 114.67               | 102.01            | 12.43      | 19.40       | 20.33       | 17.39    |
| 1             | 76.17       | 99.57            | 102.47               | 92.73             | 12.07      | 17.07       | 18.50       | 15.88    |
| **Mean**      | 80.03       | 104.30           | 110.19               | 107.04            | 13.50      | 19.67       | 21.40       | 18.19    |
| **LSD(0.05)** |             |                  |                      |                   |           |            |            |          |
| F             | 2.983       |                  |                      | 0.285             |           |            |            | 0.176    |
| E             | 3.615       |                  |                      | 0.574             |           |            |            | 0.304    |
| F*E           | 6.261       |                  |                      | 0.994             |           |            |            | 0.527    |
Table 4: Continued

| Extracts(E) | Fertilizer(F) | Umbelets No. / umbel | Dry weight of plant |
|-------------|---------------|----------------------|---------------------|
|             |               | NPK                  | Vinasse 50%         | Vinasse 100% | Mean   | NPK      | Vinasse 50% | Vinasse 100% | Mean   |
| 0           | 15.33         | 17.43                | 18.10               | 16.96        | 89.47   | 108.77   | 109.40      | 102.54         |
| 1           | 15.33         | 18.30                | 19.03               | 17.56        | 92.10   | 109.93   | 111.97      | 104.67         |
| 2           | 15.97         | 19.07                | 22.33               | 19.12        | 97.37   | 112.10   | 114.03      | 107.83         |
| 4           | 16.43         | 19.07                | 22.37               | 19.29        | 100.33  | 112.73   | 114.40      | 109.16         |
| 1           | 15.33         | 18.97                | 19.03               | 17.78        | 93.53   | 111.10   | 112.03      | 105.56         |
| 2           | 16.83         | 19.07                | 22.33               | 19.41        | 103.40  | 113.77   | 115.17      | 110.78         |
| 4           | 17.10         | 19.23                | 22.17               | 19.50        | 106.20  | 113.93   | 115.20      | 111.78         |
| Mean        | 16.05         | 18.73                | 20.77               | 19.49        | 97.49   | 111.76   | 113.17      |                 |

| GA (%)      |               |                      |                     |
| 1           | 15.33         | 18.30                | 19.03               | 17.56        | 92.10   | 109.93   | 111.97      | 104.67         |
| 2           | 15.97         | 19.07                | 22.33               | 19.12        | 97.37   | 112.10   | 114.03      | 107.83         |
| 4           | 16.43         | 19.07                | 22.37               | 19.29        | 100.33  | 112.73   | 114.40      | 109.16         |
| Mean        | 16.05         | 18.73                | 20.77               | 19.49        | 97.49   | 111.76   | 113.17      |                 |

| Li (%)      |               |                      |                     |
| 1           | 15.33         | 18.97                | 19.03               | 17.78        | 93.53   | 111.10   | 112.03      | 105.56         |
| 2           | 16.83         | 19.07                | 22.33               | 19.41        | 103.40  | 113.77   | 115.17      | 110.78         |
| 4           | 17.10         | 19.23                | 22.17               | 19.50        | 106.20  | 113.93   | 115.20      | 111.78         |
| Mean        | 16.05         | 18.73                | 20.77               | 19.49        | 97.49   | 111.76   | 113.17      |                 |

| LSD(0.05)   | F             | 2.326                | 0.382               |
| E           | 0.994         |                      |                     |
| F*E         | 1.721         |                      |                     |

| Second Season (2018/2019) |               |                     |
|---------------------------|---------------|---------------------|
| 0            | 15.07         | 18.03                | 18.03               | 17.04        | 89.37   | 109.63   | 110.27      | 103.09         |
| 1            | 15.07         | 18.10                | 18.30               | 17.16        | 92.13   | 110.90   | 112.90      | 105.31         |
| 2            | 16.10         | 18.97                | 21.67               | 18.91        | 98.00   | 113.93   | 115.93      | 109.29         |
| 4            | 16.17         | 18.97                | 22.33               | 19.16        | 104.33  | 113.93   | 116.10      | 111.46         |
| 1            | 15.63         | 18.13                | 18.80               | 17.52        | 93.33   | 111.67   | 113.30      | 106.10         |
| 2            | 16.83         | 18.97                | 22.67               | 19.49        | 105.97  | 114.57   | 116.80      | 112.44         |
| 4            | 17.33         | 19.13                | 22.33               | 19.60        | 107.17  | 114.67   | 116.73      | 112.86         |
| Mean         | 16.03         | 18.61                | 20.59               | 19.60        | 98.61   | 112.76   | 114.57      |                 |

| LSD(0.05)   | F             | 0.909                | 0.514               |
| E           | 0.965         |                      |                     |
| F*E         | 1.678         |                      |                     |
considerably plant height, number of branches per plant, number of umbels per plant, number of umblets per umbel and dry weight of caraway as compared to control treatment in this respect in the two growing seasons. Foliar spraying of licorice extract at all levels led to significant increase in height of caraway plants, number of branches as well as number of umbels and umblets number. These findings are in harmony with those found by (Nasser et al., 2014 and Abd El -Azim et al., 2017), also foliar application of garlic extract at high rates significantly enhanced growth characters, these results are similar to those obtained by (El-Shayeb, 2009; EL-Saïd and Ali, 2013 and Al-Obady, 2015). Vinasse treatments at both two levels showed increments in the studied growth parameters, in this concern, Vadivel et al. (2014) stated that addition of various levels of vinasse in agriculture stimulated crop yields, increased a significant amount of nutrients and amended the soil quality of degraded land. These may be attributable to the positive effect of vinasse on encouraging microflora populations, and increase of microbial biomass thus improving the soil fertility status and enhanced crop production (Shang-Dong et al., 2013). It is obvious that the treatment of the high level of vinasse V2 (100%) in combinations with foliar spraying of garlic (4%) or licorice (2%) extracts significantly improved caraway growth traits and exceeded all other treatments in both branch and umbel numbers as well as umbletes number and dry weight of caraway plant. The other treatments recorded an intermediate values in both seasons.

2- Effect on seed measurements:

Table (5) clear that the highest seed yield per plant and per fed. were verified when the treatments of vinasse (V2) in combinations with foliar spraying of licorice (2%) were used followed by vinasse (V2) in combinations with foliar spraying of garlic (4%) applied without significance among them in both seasons. Also, the heaviest 1000 seeds and the highest harvest index percentage followed the same trend as in the highest seed yield per plant. Such effect was confirmed in the two successive seasons, this may be due to the positive allelopathic effect of garlic and licorice extract which rich in vitamins, amino acids and minerals so improves flowering, fruit yield and fruit quality. These results are in covenant with those of EL-Saïd and Ali, 2013; Fathel et al., 2015; Cheng et al., 2016 and Ding et al., 2016. Generally, data revealed that plants receiving a high concentration of vinasse (V2) recognized increments concerning the optimum yield of caraway as related to low concentration (V1), these due to better nutritional elements required for plant growth. Vinasse is a good source of available K when smeared to the soil and its application may reduce the quantity of fertilizers needed for crop yield (Arafat and Yassen, 2002). Also, vinasse may be used as soil conditioners, which improve the availability of the nutrients thus increase yield (Omar et al., 2000).

3- Essential oil productivity

Data presented in Table (6) showed that, vinasse fertilization level increased from V1 up to 100 % (V2) of the recommended dose, oil yield per plant and per fed. was increased. The highest oil yield was recorded in plants received V2 (100% of the recommended NPK dose) during both seasons. As for the effect of spraying natural extracts of licorice and garlic on caraway oil production, obtained results revealed that spraying plants with licorice and garlic extract at all tested concentrations enhanced essential oil production per plant and per fed. comparing to unsprayed control plants in the two seasons. The most effective treatment in this concern was spraying licorice extract at 2% (produced 2.9- 2.9 %, 1.51-1.62 ml and 31.80 - 33.92 L oil percentage, essential oil per plant and per fed. for both seasons, respectively) followed by spraying treatment of garlic extract at 4% (resulted 2.87- 2.9 %, 1.44- 1.58 ml and 30.24- 33.06 L for both seasons, respectively). When NPK chemical fertilization or vinasse at two levels interacted with extracts of licorice or garlic spraying, significant effects on oil production per plant and per fed. were detected as compared to unsprayed plants. The highest essential production/ plant was found in plants subjected to the treatment of vinasse at V2 dose in combination with spraying with licorice extract at 2%. These interaction effects on oil production/ plant were reflected on oil production/ fed and this confirmed in the two experimental seasons.
4- Constituents of essential oil

Ten compounds were detected and identified as shown in Table (7). All studied treatments showed that carvone is the main constituents essential oil (56.22-66.34%), followed by limonene which varied from (12.91 to 17.01%) then α-guaiene (2.13-5.49%) as well as linalool which ranged from (1.30 to 2.53 %). The other identified compounds were acoradien, estragole, α- gurjunene, β-caryophyllene, 2, 6- dihydroxyacetophenone, and α-pinene. These results are in harmony with those found by Olle and Bender, (2010).

5- Chemical Constituents

Caraway seeds are also a rich source of macro as well as micronutrients (Kluszczyńska 2002). A significant effect of vinasse fertilization alone or in combination with foliar spraying of licorice or garlic extract on the chemical composition of caraway seed was found (Table 8). All the different rates of vinasse fertilizer clearly increased the mean content of total N, P, K Ca, Mg and Fe in seeds. The highest average of total N and P contents were 3.61 and 0.58 % in the first season while, 3.63 and 0.60 % in the second season, respectively resulting from the fertilization treatment with the highest rates of vinasse (V2) interacted with highest rates of garlic extract (4%).

Regarding macro and microelements in caraway seeds, results in Table 8 showed steady increases during the two seasons as the level of vinasse fertilizer increased from 50% up to 100% of the recommended dose compared to NPK fertilizer. For the allelopathic effect of foliar spray of natural extracts of garlic and licorice on K, Ca, Mg and Fe % in caraway seeds, the obtained data showed that all tested extracts treatments increased the studied elements in seeds in the two seasons compared to unsprayed control treatment. Besides, the spray treatment of licorice extract at 4%, followed by the spray treatment of garlic extract at 4% under the utilization of the highest rate of vinasse (100% of the recommended dose) recorded the highest K percentages seeds for both seasons. On the other hand, the spray treatment of garlic extract at 4% under the utilization of the highest rate of vinasse (100% of the recommended dose) recorded the maximum percentages of Ca, Mg and Fe in seeds for both seasons. Respecting effect interaction treatments between NPK fertilization, vinasse fertilization and foliar spray of extracts of garlic or licorice on the studied elements in seeds, data in Table 8 showed that spraying tested extracts at certain rates under both tested fertilizer increased percentages of studied elements in caraway seeds as compared to the same fertilizer without spraying. Also, vinasse fertilization under any tested extract application increased the seed contents of the nutrients comparing to the same spray application with NPK fertilization. In general, the superior values of nutrients were found in plant seed of subjected plants to interaction treatments between full vinasse fertilizer dose and a foliar spraying of licorice extract at 2 or 4%. These results are in covenant with those of Shang-Dong et al., 2013; Al-Obady, 2015 and Seddik et al., 2016.

Conclusion

From the above mentioned results, it could be concluded that all the tested fertilization treatments and all tested natural extracts each alone or interacted with other significantly increased vegetative parameters and seed measurements of caraway plants. The combination of vinasse (100%) with foliar spraying of licorice extract at the rate of 2% was the most effective treatment in this respect. The results indicated that both garlic and licorice extracts under chemical fertilizer NPK and levels of vinasse as organic fertilizer possess a positive allelopathic effect on growth, the yield of caraway so biomass was stimulated with these treatments. Also, data emphasized that caraway plants produce the highest oil yield with the highest quality and the superior contents of nutrients in response to the same treatments, where the application of vinasse to the soil has increased the availability of macronutrients in the soil especially K and organic matter which improved crop yield. It could be concluded that the importance of the use of vinasse alone or in combination with natural extracts of garlic and licorice as alternative sources for K-fertilizers and beneficial cheap sources of K-fertilization for agriculture.
Table 5: Effect of vinasse and natural extracts of garlic and licorice on seed measurements of *Carum carvi* L. during 2017/2018 and 2018/2019.

| Extract | Fertilizer | Seed yield /plant (g) | Seed yield /fed. (kg) | Weight of 1000 seeds (g) | Harvest Index (%) |
|---------|------------|-----------------------|-----------------------|--------------------------|-------------------|
|         |            | NPK V | Vinasse 50% | Vinasse 100% | Mean | NPK V | Vinasse 50% | Vinasse 100% | Mean | NPK V | Vinasse 50% | Vinasse 100% | Mean | NPK V | Vinasse 50% | Vinasse 100% | Mean |
| 0       |            | 33.13 | 45.50 | 47.00 | 41.88 | 695.73 | 955.50 | 987.00 | 879.41 | 13.93 | 16.20 | 16.27 | 15.47 | 37.04 | 41.83 | 42.96 | 40.61 |
| 1       | GA (%)     | 40.33 | 47.80 | 49.20 | 45.78 | 847.00 | 1003.80 | 1033.20 | 961.33 | 15.03 | 16.43 | 17.10 | 16.19 | 43.79 | 43.48 | 43.94 | 43.74 |
| 2       | 40.67      | 50.03 | 50.23 | 46.98 | 854.00 | 1050.70 | 1054.90 | 986.53 | 15.10 | 17.53 | 19.47 | 17.37 | 40.53 | 44.38 | 43.91 | 42.94 |
| 4       | 40.33      | 48.47 | 49.90 | 46.23 | 847.00 | 1017.80 | 1047.90 | 970.90 | 15.03 | 16.73 | 17.37 | 16.38 | 43.12 | 43.62 | 44.54 | 43.76 |
| 1       | Li (%)     | 41.83 | 50.07 | 52.20 | 48.03 | 878.50 | 1051.40 | 1096.20 | 1008.70 | 15.27 | 18.17 | 20.37 | 17.93 | 40.46 | 44.01 | 45.33 | 43.27 |
| 2       | 43.40      | 50.13 | 50.27 | 47.93 | 911.40 | 1052.80 | 1055.60 | 1006.60 | 15.83 | 18.90 | 20.33 | 18.36 | 40.87 | 44.00 | 43.63 | 42.83 |
| Mean    |            | 39.98 | 48.85 | 49.85 | 839.49 | 1025.90 | 1046.90 | 1041.0 | 15.04 | 17.36 | 18.61 | 14.01 | 41.01 | 43.70 | 44.04 |          |

LSD (0.05) | F 0.621 | 0.492 | 0.754 |
|           | E 0.666 | 0.248 | 0.782 |
|           | F*E 1.153 | 0.429 | 1.354 |

Second Season (2018/2019)

| Extract | Fertilizer | Seed yield /plant (g) | Seed yield /fed. (kg) | Weight of 1000 seeds (g) | Harvest Index (%) |
|---------|------------|-----------------------|-----------------------|--------------------------|-------------------|
|         |            | NPK V | Vinasse 50% | Vinasse 100% | Mean | NPK V | Vinasse 50% | Vinasse 100% | Mean | NPK V | Vinasse 50% | Vinasse 100% | Mean | NPK V | Vinasse 50% | Vinasse 100% | Mean |
| 0       |            | 34.53 | 43.33 | 44.03 | 40.63 | 725.20 | 910.00 | 924.70 | 853.30 | 14.77 | 17.77 | 18.07 | 16.87 | 38.65 | 39.53 | 39.93 | 39.73 |
| 1       | GA (%)     | 39.03 | 44.07 | 43.98 | 49.80 | 819.70 | 925.40 | 1025.50 | 923.50 | 15.07 | 18.17 | 19.93 | 17.72 | 42.37 | 43.94 | 43.26 | 41.79 |
| 2       | 40.37      | 50.67 | 51.47 | 48.40 | 847.70 | 1064.00 | 1137.50 | 1016.40 | 15.80 | 20.13 | 21.03 | 18.99 | 41.20 | 44.47 | 46.72 | 44.13 |
| 4       | 41.17      | 52.57 | 54.27 | 48.90 | 864.50 | 1076.60 | 1139.60 | 1026.90 | 16.07 | 20.43 | 21.07 | 19.19 | 39.46 | 44.99 | 46.74 | 43.73 |
| 1       | Li (%)     | 39.87 | 44.97 | 49.33 | 44.72 | 837.20 | 944.30 | 1036.00 | 939.17 | 15.27 | 18.97 | 20.03 | 18.09 | 42.71 | 42.27 | 43.55 | 42.18 |
| 2       | 41.90      | 52.40 | 55.70 | 50.00 | 879.90 | 1100.40 | 1169.70 | 1050.00 | 16.33 | 20.70 | 21.13 | 19.39 | 39.54 | 45.74 | 47.69 | 44.32 |
| 4       | 42.70      | 53.93 | 54.33 | 50.32 | 896.70 | 1132.60 | 1141.00 | 1056.77 | 16.83 | 20.90 | 21.07 | 19.60 | 39.84 | 47.04 | 46.54 | 44.47 |
| Mean    |            | 39.94 | 48.66 | 51.52 | 838.70 | 1021.90 | 1082.00 | 1056.70 | 15.73 | 19.58 | 20.33 | 14.01 | 40.54 | 43.11 | 44.92 |          |

LSD (0.05) | F 0.425 | 0.240 | 0.490 |
|           | E 0.731 | 0.133 | 0.728 |
|           | F*E 1.266 | 0.231 | 1.261 |
Table 6: Effect of vinasse and natural extracts of garlic and licorice on essential oil productivity of *Carum carvi* L. during 2017 and 2018.

| Fertilizer (F) | Essential oil (%) | Essential oil yield /plant (ml) | Essential oil yield /fed. (L) |
|---------------|-------------------|--------------------------------|-----------------------------|
|               | NPK | Vinasse 50% | Vinasse 100% | Mean | NPK | Vinasse 50% | Vinasse 100% | Mean | NPK | Vinasse 50% | Vinasse 100% | Mean |
|               | NPK | Vinasse 50% | Vinasse 100% | Mean | NPK | Vinasse 50% | Vinasse 100% | Mean | NPK | Vinasse 50% | Vinasse 100% | Mean |
| 0 | 2.63 | 2.70 | 2.70 | 2.68 | 0.87 | 1.23 | 1.27 | 1.12 | 18.32 | 25.78 | 26.65 | 23.58 |
| 1 | 2.63 | 2.70 | 2.73 | 2.69 | 1.06 | 1.29 | 1.34 | 1.23 | 22.31 | 27.09 | 28.23 | 25.88 |
| 2 | 2.67 | 2.73 | 2.87 | 2.76 | 1.07 | 1.37 | 1.44 | 1.29 | 22.47 | 28.68 | 30.20 | 27.12 |
| 4 | 2.67 | 2.77 | 2.87 | 2.77 | 1.09 | 1.38 | 1.44 | 1.30 | 22.77 | 29.07 | 30.24 | 27.36 |
| GA (%) | 1 | 2.63 | 2.70 | 2.73 | 2.69 | 1.06 | 1.31 | 1.36 | 1.24 | 22.31 | 27.48 | 28.64 | 26.14 |
| 2 | 2.67 | 2.73 | 2.87 | 2.76 | 1.07 | 1.37 | 1.44 | 1.29 | 22.47 | 28.68 | 30.20 | 27.12 |
| 4 | 2.67 | 2.77 | 2.87 | 2.77 | 1.09 | 1.38 | 1.44 | 1.30 | 22.77 | 29.07 | 30.24 | 27.36 |
| Li (%) | 1 | 2.63 | 2.70 | 2.73 | 2.69 | 1.06 | 1.31 | 1.36 | 1.24 | 22.31 | 27.48 | 28.64 | 26.14 |
| 2 | 2.67 | 2.80 | 2.90 | 2.79 | 1.12 | 1.40 | 1.51 | 1.34 | 23.43 | 29.44 | 31.80 | 28.22 |
| 4 | 2.70 | 2.83 | 2.87 | 2.80 | 1.17 | 1.42 | 1.44 | 1.35 | 24.60 | 29.82 | 30.26 | 28.23 |
| Mean | 2.66 | 2.75 | 2.81 | 1.06 | 1.34 | 1.40 | 22.32 | 28.20 | 29.43 |

LSD (0.05)  
F 0.176 0.089 1.877  
E 0.044 0.028 0.587  
F*E 0.077 0.048 1.017

Second Season (2018/2019)

| Fertilizer (F) | Essential oil (%) | Essential oil yield /plant (ml) | Essential oil yield /fed. (L) |
|---------------|-------------------|--------------------------------|-----------------------------|
|               | NPK | Vinasse 50% | Vinasse 100% | Mean | NPK | Vinasse 50% | Vinasse 100% | Mean | NPK | Vinasse 50% | Vinasse 100% | Mean |
|               | NPK | Vinasse 50% | Vinasse 100% | Mean | NPK | Vinasse 50% | Vinasse 100% | Mean | NPK | Vinasse 50% | Vinasse 100% | Mean |
| 0 | 2.67 | 2.80 | 2.83 | 2.77 | 0.92 | 1.21 | 1.25 | 1.13 | 19.35 | 25.47 | 26.20 | 23.67 |
| 1 | 2.70 | 2.83 | 2.87 | 2.79 | 1.05 | 1.25 | 1.38 | 1.23 | 22.13 | 26.22 | 29.04 | 25.80 |
| 2 | 2.73 | 2.87 | 2.90 | 2.83 | 1.10 | 1.45 | 1.57 | 1.38 | 23.17 | 30.50 | 32.99 | 28.89 |
| 4 | 2.73 | 2.87 | 2.90 | 2.83 | 1.13 | 1.47 | 1.58 | 1.39 | 23.63 | 30.87 | 33.06 | 29.19 |
| GA (%) | 1 | 2.70 | 2.83 | 2.87 | 2.80 | 1.08 | 1.27 | 1.41 | 1.25 | 22.60 | 26.75 | 29.68 | 26.35 |
| 2 | 2.73 | 2.90 | 2.90 | 2.86 | 1.16 | 1.52 | 1.62 | 1.43 | 24.34 | 31.91 | 33.92 | 30.06 |
| 4 | 2.80 | 2.90 | 2.90 | 2.87 | 1.20 | 1.56 | 1.58 | 1.45 | 25.10 | 32.84 | 33.11 | 30.35 |
| Mean | 2.73 | 2.86 | 2.88 | 1.09 | 1.39 | 1.48 | 22.90 | 29.22 | 31.14 |

LSD (0.05)  
F 0.163 0.082 1.691  
E 0.063 0.044 0.917  
F*E 0.109 0.076 1.588
Table 7: Effect of NPK, vinasse and foliar application of garlic and licorice extracts on essential oil constituents (%) of *Carum carvi* L. in the second season of 2018/2019.

| Components          | NPK   | Vinasse (V₁) | V₁ (50%) + GA extract | V₁ (50%) + Li extract | Vinasse (V₂) | V₂ (100%) + GA extract | V₂ (100%) + Li extract |
|---------------------|-------|--------------|------------------------|------------------------|--------------|-------------------------|------------------------|
| α-pinene            | 0.19  | 0.07         | 0.08                   | 0.13                   | 0.18         | traces                  | 0.16                   | 0.17                   | 0.09 | 0.10 | 0.11 | 0.10 | 0.09 | 0.12 |
| 2, 6-dihydroxyacetophenone | 0.86  | 0.34         | 0.39                   | 0.42                   | 1.58         | 0.57                    | 1.11                   | 1.61                   | 1.75  | 0.73 | 1.89 | 2.03 | 0.81 | 2.04 | 2.35 |
| Acoradien           | 0.90  | 1.02         | 1.19                   | 1.71                   | 2.07         | 1.13                    | 1.93                   | 2.25                   | 1.20  | 1.32 | 1.25 | 1.60 | 1.66 | 2.27 | 2.43 |
| α-guaiene           | 5.00  | 2.13         | 3.81                   | 4.23                   | 5.02         | 2.39                    | 4.64                   | 5.08                   | 2.18  | 3.40 | 5.11 | 4.20 | 3.62 | 4.00 | 5.49 |
| Limonene            | 14.91 | 13.11        | 13.50                  | 13.77                  | 15.40        | 13.82                   | 16.25                  | 14.39                   | 14.51 | 14.51 | 17.01 | 15.00 | 13.92 | 15.27 | 12.91 |
| Linalool            | 2.39  | 1.38         | 1.38                   | 1.39                   | 2.38         | 1.30                    | 2.27                   | 2.39                   | 2.10  | 2.29 | 2.43 | 2.45 | 1.35 | 2.53 | 2.51 |
| β-caryophyllene     | 0.58  | 0.55         | 0.60                   | 1.09                   | traces       | 0.59                    | 1.18                   | 1.39                   | 0.67  | 0.82 | 1.42 | 1.63 | 1.05 | 1.70 | 1.63 |
| Carvone             | 59.39 | 60.00        | 60.39                  | 64.12                  | 63.03        | 60.00                   | 64.19                  | 63.00                   | 60.55 | 60.55 | 65.11 | 66.34 | 60.70 | 65.10 | 56.22 |
| Estragole           | 0.64  | 0.73         | 1.08                   | 1.92                   | 2.29         | 0.91                    | 2.17                   | 2.33                   | 1.23  | 1.58 | 2.56 | 2.60 | 1.73 | 3.00 | 2.71 |
| α-gurjunene         | 1.99  | 1.35         | 1.49                   | 2.00                   | 2.68         | 1.56                    | 2.52                   | 3.78                   | 1.87  | 2.40 | 2.60 | 2.61 | 1.98 | 2.75 | 3.79 |
| Unknown             | 13.15 | 19.32        | 16.09                  | 9.22                   | 5.37         | 17.73                   | 3.58                   | 3.61                   | 13.85 | 12.31 | 5.52 | 1.43 | 13.08 | 4.25 | 9.84 |
Table 8: Effect of vinasse and natural extracts of garlic and licorice on nitrogen, phosphorus, potassium, calcium, magnesium and iron contents (%) of *Carum carvi* L. during the two seasons of 2017/2018 and 2018/2019.

| Fertilizer(F) | Extracts(E) | N (%) | P (%) | K (%) | N (%) | P (%) | K (%) |
|---------------|-------------|-------|-------|-------|-------|-------|-------|
|               |             | First (2017/2018) |       |        | Second (2018/2019) |       |        |
|               |             | NPK 50% | Vinasse 50% | Vinasse 50% | mean | NPK 100% | Vinasse 100% | Vinasse 100% | mean |
| 0             | 1.607       | 1.707  | 1.681  | 0.097  | 0.133  | 0.173  | 0.134  | 1.203  | 1.867  | 2.103  | 1.724  |
| 1             | 1.607       | 1.777  | 1.781  | 0.097  | 0.177  | 0.203  | 0.159  | 1.247  | 2.16   | 2.547  | 1.984  |
| 2             | 1.607       | 2.263  | 2.462  | 0.103  | 0.220  | 0.497  | 0.273  | 1.307  | 2.843  | 3.807  | 2.652  |
| 4             | 1.613       | 3.217  | 2.814  | 0.117  | 0.337  | 0.583  | 0.346  | 1.367  | 3.413  | 4.073  | 2.951  |

### LSD(0.05)

|               | F   | 0.087 | 0.023 | 0.074 |
|---------------|-----|-------|-------|-------|
| E             | 0.054 | 0.013 | 0.086 |
| F*E           | 0.094 | 0.022 | 0.148 |

|               | F  | 0.087 | 0.026 | 0.046 |
|---------------|---|-------|-------|-------|
| LSD(0.05)     | 0.078 | 0.014 | 0.043 |
| F*E           | 0.134 | 0.025 | 0.074 |
Table 8: Continued

| Extracts (E) | Fertilizer (F) | Ca (%) | Mg (%) | Fe (%) |
|--------------|----------------|--------|--------|--------|
|              | NPK            | Vinasse 50% | Vinasse 100% | Mean | NPK            | Vinasse 50% | Vinasse 100% | Mean | NPK            | Vinasse 50% | Vinasse 100% | Mean |
| 0            | 0.503          | 0.543 | 0.547 | 0.531 | 1.26  | 1.767 | 2.083 | 1.703 | 0.917 | 1.040 | 1.113 | 1.023 |
| GA (%)       | 1.000          | 0.508 | 0.547 | 0.548 | 1.267 | 2.063 | 2.287 | 1.872 | 0.940 | 1.183 | 1.280 | 1.134 |
|              | 4.000          | 0.533 | 0.683 | 0.940 | 0.719 | 1.427 | 1.923 | 3.120 | 0.973 | 1.733 | 2.163 | 1.623 |
| Li (%)       | 1.000          | 0.513 | 0.563 | 0.593 | 0.557 | 1.310 | 2.150 | 2.031 | 0.957 | 1.217 | 1.377 | 1.183 |
|              | 2.000          | 0.537 | 0.753 | 0.870 | 0.720 | 1.527 | 3.707 | 4.270 | 3.168 | 1.987 | 2.080 | 1.693 |
| Mean         | 0.521          | 0.619 | 0.732 | 1.355 | 2.762 | 3.452 | 0.965 | 1.437 | 1.719 |

**LSD (0.05)**

|                | F   | E   | F*E  |
|----------------|-----|-----|------|
| F              | 0.100 | 0.013 | 0.022 |
| E              | 0.010 | 0.013 | 0.025 |
| F*E            | 0.038 | 0.032 | 0.056 |

**Second Season (2018/2019)**

| Extracts (E) | Fertilizer (F) | Ca (%) | Mg (%) | Fe (%) |
|--------------|----------------|--------|--------|--------|
|              | NPK            | Vinasse 50% | Vinasse 100% | Mean | NPK            | Vinasse 50% | Vinasse 100% | Mean | NPK            | Vinasse 50% | Vinasse 100% | Mean |
| 0            | 0.490          | 0.557 | 0.593 | 0.547 | 1.327 | 2.137 | 2.227 | 1.897 | 0.923 | 1.117 | 1.200 | 1.080 |
| GA (%)       | 1.000          | 0.513 | 0.663 | 0.596 | 1.330 | 2.247 | 2.626 | 1.947 | 0.943 | 1.277 | 1.470 | 1.230 |
|              | 4.000          | 0.537 | 0.787 | 0.973 | 0.766 | 1.757 | 2.887 | 3.927 | 2.857 | 0.990 | 1.957 | 2.220 | 1.722 |
| Li (%)       | 1.000          | 0.513 | 0.660 | 0.713 | 0.629 | 1.403 | 2.247 | 2.453 | 2.034 | 0.947 | 1.403 | 1.587 | 1.312 |
|              | 2.000          | 0.543 | 0.747 | 0.863 | 0.718 | 1.843 | 2.503 | 3.879 | 2.748 | 1.010 | 1.830 | 2.157 | 1.666 |
| Mean         | 0.526          | 0.702 | 0.794 | 1.567 | 2.494 | 3.169 | 0.981 | 1.627 | 1.842 |

**LSD (0.05)**

|                | F   | E   | F*E  |
|----------------|-----|-----|------|
| F              | 0.044 | 0.015 | 0.025 |
| E              | 0.042 | 0.015 | 0.072 |
| F*E            | 0.011 | 0.019 | 0.033 |
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