THE EFFECT OF INFECTION BY CALOGLYPHUS BERLESEI ON ORGANIC VOLATILE COMPOUNDS OF SOME STORED PRODUCTS

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SUMMARY

Mites are a major cause of qualitative and quantitative losses to several stored food and feed products. The present work aimed to study the effect of infection by Caloglyphus berlesei on chemical organic volatile compound of some stored products. Volatile compounds were evaluated for soybean, wheat, maize and fishmeal before and after infestation with mites after three months by GC/MS. Soybean infected by Caloglyphus berlesei caused to presence of different six compounds. Also, wheat infected caused to presence of different ten compounds. There were fifty compounds evaluated from stored maize by GC/MS. Maize infected presence of sixty six compounds, twenty of them were appeared different. There were four compounds present in stored maize uninfected but not detected in infected stored maize. Fishmeal infected by Caloglyphus berlesei after three months caused to presence of forty six compounds, twenty five of them were appeared different. There were six compounds present in stored fishmeal uninfected but not appeared in infected stored fishmeal. Volatile compounds in stored products (soybean, wheat, maize and fishmeal) appeared that 1-aminododecane and 10-Undecenal were related with Caloglyphus berlesei in all stored products under investigation.

Keywords: stored products, volatile compounds, soybean, wheat, maize and fishmeal.

INTRODUCTION

mites are a major cause of qualitative and quantitative losses to several stored products. The pest importance of stored product mites has been reviewed and three pest risks are suggested; (i) direct consumption on human food, animal feed or other products changing the quality of infested products, they can penetrate the hard grains and feed directly on the grain kernels, therefore they destroy their germination power, change the moisture contents of media, initiating growth and spread mold Gulati and Mathur, (1995); (ii) interaction to microorganisms leading to the transfer of mycotoxins production fungi or pathogenic bacteria; (iii) production of hazardous compounds among them the allergens are of the highest importance (Hubert,2011). The mites change the quality of infested food by the production of secretions and feces. The massive infestation by mites changes the smell of stored products. The stored-product mites cause hypersensitivity not only for stored grain and farm workers, millers and bakers, but they also seriously endanger the health of the city population Musken, et al. (2003). (Tuma, et al. 1990) reported mite specific volatiles decane, undecane, tridecane and perilen. Tridecane was detected in bin stored wheat infested by stored product mites. Ridgway, et al. (1999) observed that A. siro produced alkane undecane. The pheromones are produced mainly by opistosomal glands (Kuwahara, 2004). All the produced compounds probably change the smell of stored products. Although the compounds play an important role in mite communication. Curtis et al. (1981) found that the headspace above two strains of Acarus siro maintained on wheat germ/bran contain the hydrocarbons, decane, undecane and tridecane together with the furanoid terpene, perilen. When the bodies of the separated mites were extracted with diethyl ether a new compound was found in addition to those in the headspace. This compound has been identified as 2-hydroxy-6-methyl benzaldehyde. None of these compounds appear to be responsible for the ‘minty’ smell reportedly related to mite infestation. Some plants have been found to produce and emit volatile organic compounds (VOCs) such as ethylene, isoprene, mono and sesquiterpenes, alkanes, alcohols ,aldehydes, organic acids, ketones and others in response to an attack or an injury by external agents (Langenheim, 1994; Peñuelas et
These compounds represent an arsenal of defences ranging from chemical toxins to feeding deterrents (Langenheim, 1994). Some of these compounds emitted during phytophagous attack may be used in another defensive strategy namely the attraction and recruitment of the herbivores’ natural enemies: predators and parasites (Dicke et al., 1990; Turlings et al., 1990; Pallini et al., 1997). Among phytophagous animals, mites attack a large number of plants, and several studies have demonstrated that plants infested by spider mites initiate the release of VOCs that are attractive to predatory mites (Dicke, 1988; Dicke et al., 1990; Bruin et al., 1995; Koveos et al., 1995). This is a widely studied phenomenon for its great ecological interest and its possible application to agricultural pest control. Tuma et al. (1990) reported tridecane associated with three mite species, *Acarus siro* (L.), *Aeroglyphus robustus* Banks, and *Lepidoglyphus destructor* (Schrank), introduced into 15.2% moisture content wheat stored in two unheated experimental bins. The mites produced tridecane all year, although they overwintered at low numbers. Production of tridecane by *A. robustus* and *L. destructor* is being reported for the first time.

The present work aimed to study the effect of infection by *Caloglyphus berlesei* on chemical organic volatile compound of some stored products.

MATERIALS AND METHODS

Volatile compounds were evaluated for soybean, wheat, maize and fishmeal before and after infestation with mites by GC/MS (Gas Chromotography – Mass Spectrometry) (organic compounds) at Regional Center for Food and Feed (RCFF), Agriculture Research Center, Giza.

**Extraction:**

One grams of sample powder was extracted using anhydrous ethyl alcohol for three times (15 minutes each time) with the assistance of ultrasonic bath. The obtained turbid solution was filtrated and the solvent of filtrate was removed by rotary evaporation under reduced pressure. Then the extracted was filtered through a 0.45 mm membrane filter. 1 mL of sub sequent filtrate was injected to GC/MS for analysis.

**GC/MS analysis:**

The analysis was carried out using a GC (Agilent Technologies 7890A) interfaced with a mass-selective detector (MSD, Agilent 7000 Triple Quad) equipped with Agilent HP-5ms (5%-phenyl methyl poly siloxane) capillary column (30 m x 0.25 mm i. d. and 0.25 μm film thickness). The carrier gas was helium with the linear velocity of 1 ml/min. The injector and detector temperatures were 200º C and 250º C, respectively. Volume injected 1μl of the sample. The MS operating parameters were as follows: ionization potential 70 eV, interface temperature 250º C, and acquisition mass range 50–600. The identification of components was based on a comparison of their mass spectra and retention time with those of the authentic compounds and by computer matching with NIST and WILEY library as well as by comparison of the fragmentation pattern of the mass spectral data with those reported in the literature.

RESULTS AND DISCUSSION

**Volatile extracted from stored soybean:**

Data in (Table 1) showed that there were fifteen compounds evaluated from stored soybean by GC-MS. Butanoic acid, 2-amino-, (S) - was the first organic compound appeared after 4.86 mintues with 10.33% area sum. Hexadecanedioic acid was the final organic volatile compound appeared after 17.59 minutes with 1.6% area sum. 17-Octadecynoic acid was the highest area with 14.26 %, but 1-Octadeconamine, N-methyl- was the lowest area with 1.35%. Soybean infected by *Caloglyphus berlesei* after three months caused to presence of twenty one compounds, six of them were appeared. It seems that they refer to infection by *Caloglyphus berlesei*. These compounds (Dodecane, 1- methoxy- appeared at 11.98 min with area sum 5.42%; Undecane appeared at 12.37 min with area sum 3.45 %; 1-Aminododecane at 12.9 min with 1.55% area sum;2-Methylundecanal at 15.18 min with 3.11% area sum; Decanal at 15.4 min with 3.86 % area sum and 10- Undecenal at 16.98 min with 1.47 % area sum as shown in (Table 2), Dodecane, 1- methoxy- was the highest area of them, but 10- Undecenal was the lowest.
Table (1): Organic volatile compounds extracted from stored soybean after three months by GC-MS

| No | RT(min) | Name                                      | Area sum% |
|----|---------|-------------------------------------------|-----------|
| 1  | 4.86    | Butanoic acid, 2-amino-, (S)-             | 10.33     |
| 2  | 5.36    | (E)-3,4-Dimethoxycinnamic acid            | 5         |
| 3  | 7.8     | 1-Octadecanamine, N-methyl-               | 1.35      |
| 4  | 9.57    | Methylamine, N,N-bis(N.-hexadecyl)-       | 6.68      |
| 5  | 11.24   | Coumarin, 4,5,7-trimethoxy-3-(p- methoxyphenyl) | 4.2      |
| 6  | 12.63   | 2-OCTANAMINE                              | 8.49      |
| 7  | 13.15   | Piperazine, 2,5-dimethyl-                 | 9.18      |
| 8  | 13.4    | Isoleucine                                | 8.241     |
| 9  | 15.74   | dl-2-Aminopimelic acid                    | 5.94      |
| 10 | 15.95   | Edetic Acid                               | 7.85      |
| 11 | 16.25   | 5-Aminovaleric acid                       | 3.56      |
| 12 | 16.67   | 17-Octadecynoic acid                      | 14.26     |
| 13 | 17.2    | 9-Decenoic acid                           | 11.06     |
| 14 | 17.41   | Elaidic acid                              | 2.27      |
| 15 | 17.59   | Hexadecanedioic acid                      | 1.6       |

*RT: Retention time, GC-MS: Gas Chromatography – Mass Spectrometry*

Table (2): Organic volatile compounds extracted from stored soybean infected by *Caloglyphus berlesei* after three months by GC-MS

| No | RT(min) | Name                                      | Area Sum% |
|----|---------|-------------------------------------------|-----------|
| 1  | 4.86    | Butanoic acid, 2-amino-, (S)-             | 2.28      |
| 2  | 5.36    | (E)-3,4-Dimethoxycinnamic acid            | 2.08      |
| 3  | 7.8     | 1-Octadecanamine, N-methyl-               | 0.76      |
| 4  | 9.57    | Methylamine, N,N-bis(N.-hexadecyl)-       | 0.95      |
| 5  | 11.24   | Coumarin, 4,5,7-trimethoxy-3-(p- methoxyphenyl)- | 1.18     |
| 6  | 11.98   | Dodecane, 1-methoxy-                      | 5.42      |
| 7  | 12.37   | UNDECANE                                  | 3.45      |
| 8  | 12.63   | 2-OCTANAMINE                              | 2.03      |
| 9  | 12.9    | 1-AMINODODECANE                           | 1.55      |
| 10 | 13.15   | Piperazine, 2,5-dimethyl-                 | 0.95      |
| 11 | 13.4    | Isoleucine                                | 2.77      |
| 12 | 15.18   | 2-Methylundecanal                        | 3.11      |
| 13 | 15.4    | Decanal                                   | 3.86      |
| 14 | 15.74   | dl-2-Aminopimelic acid                    | 31.9      |
| 15 | 15.95   | Edetic Acid                               | 1.82      |
| 16 | 16.25   | 5-Aminovaleric acid                       | 1.43      |
| 17 | 16.67   | 17-Octadecynoic acid                      | 66.3      |
| 18 | 16.98   | 10-Undecenal                              | 1.47      |
| 19 | 17.2    | 9-Decenoic acid                           | 30.44     |
| 20 | 17.41   | Elaidic acid                              | 0.8       |
| 21 | 17.59   | Hexadecanedioic acid                      | 1.75      |
Organic volatile compounds extracted from stored wheat:

Data in Table (3) showed that there were forty three compounds evaluated from stored wheat by GC-MS. 8-Hydroxylinalool was the first matter appeared after 8.072 minutes with 2.29% area sum. Betulin was the final material appeared after 24.07 minutes with highest area sum 14.17%, but β Carotene was the lowest area with 0.37%. Wheat infected by *Caloglyphus berlesei* after three months caused to presence of twenty nine compounds, ten of them were appeared. It seems that they refer to infection by *Caloglyphus berlesei*. These compounds (2-Hexadecanol appeared at 4.514min with area sum 2.69%;

Table (3): Volatile extracted from stored wheat after three months by GC-MS

| No. | RT (mm) | Name                                                                 | Area sum % |
|-----|---------|----------------------------------------------------------------------|------------|
| 1   | 8.072   | 8-Hydroxylinalool                                                   | 2.29       |
| 2   | 8.512   | Benzaldehyde, p-isopropyl-                                          | 1.47       |
| 3   | 9.41    | Hexestrol                                                            | 0.88       |
| 4   | 9.789   | 2-(3,4-Dimethoxyphenyl)-6-methyl-3,4-chromanediol                     | 1.64       |
| 5   | 10.772  | 2,3-Dehydro-4-oxo-β-ionol                                            | 1.07       |
| 6   | 12.614  | 2-octanamine                                                        | 4.42       |
| 7   | 12.956  | Cymarin                                                             | 0.63       |
| 8   | 13.3    | Benzaldehyde, 6-hydroxy-4-methoxy-2,3-dimethyl-                       | 0.7        |
| 9   | 13.78   | Cedrol                                                              | 1.3        |
| 10  | 14.037  | Asarone                                                             | 0.62       |
| 11  | 14.101  | Methoprene                                                          | 0.82       |
| 12  | 14.232  | Dimethyl caffeic acid                                                | 0.96       |
| 13  | 14.523  | (-)-Isolongifolol                                                    | 1.65       |
| 14  | 14.587  | Resorcinol, 2-(3,7-dimethyl-2,6-octadienyl)-5-pentyl-                | 0.89       |
| 15  | 14.709  | Hexadecanoic acid, 2-methyl-                                         | 0.89       |
| 16  | 14.846  | Ethyl 2,4,6-trimethoxycinnamate                                      | 0.75       |
| 17  | 15.063  | Digitoxin                                                           | 0.95       |
| 18  | 15.182  | 6-Carbomethoxy-5,8-dimethoxy-1-tetralone                            | 0.76       |
| 19  | 15.317  | Cannabinol                                                          | 0.5        |
| 20  | 15.68   | Pentadecanoic acid                                                  | 0.53       |
| 21  | 15.72   | QUERCETIN 7,3',4'-TRIMETHOXY                                        | 2.96       |
| 22  | 15.943  | Retinol                                                             | 3.71       |
| 23  | 16.08   | Genistin                                                            | 0.91       |
| 24  | 16.157  | Retinoic acid                                                       | 0.69       |
| 25  | 16.248  | Dodecanedioic acid                                                  | 0.47       |
| 26  | 16.279  | cis-9, cis-12-Octadecadienoic acid                                  | 0.48       |
| 27  | 16.404  | Elaidic acid                                                        | 1.46       |
| 28  | 16.636  | α-Bisabolol                                                         | 2.09       |
| 29  | 16.694  | cis-Vaccenic acid                                                   | 1.82       |
| 30  | 16.807  | β Carotene                                                          | 0.37       |
| 31  | 16.969  | Ledane                                                              | 7.32       |
| 32  | 17.2    | β-Santalol                                                          | 7.05       |
| 33  | 17.4    | Bulnesol                                                            | 7.85       |
| 34  | 18.933  | Lupulon                                                             | 2.08       |
| 35  | 19.605  | Docosanoic acid                                                    | 2.33       |
| 36  | 20.293  | Palatinose                                                          | 0.69       |
| 37  | 20.772  | Enterodiol                                                          | 1.9        |
| 38  | 21.334  | Apigenin 8-C-glucoside                                              | 2.7        |
| 39  | 21.49   | Nabilone                                                            | 2.91       |
| 40  | 21.826  | D-(+)-Celllobiose                                                   | 10.05      |
| 41  | 22.2    | Agatholic acid                                                      | 1.46       |
| 42  | 22.3    | Methyl 3,3-dimethylhenicosanoate                                    | 1.42       |
| 43  | 24.07   | Betulin                                                             | 14.17      |
Tetradecane appeared at 11.7 min with area sum 0.82%; Nonadecane at 11.868 min with area sum 0.47%; Hexadecane at 12.436 min with area sum 0.54%; 2-Octanamine at 12.626 min with area sum 0.37%; 1-Aminododecane at 12.974 min with area sum 0.35%; 10-Undecenal at 16.902 min with area sum 1.99%; 4-Octadecenal at 17.641 min with area sum 3.06%; Phytol at 19.44 min with area sum 4.33%; and Tetratetracontane at 23.6 min with area sum 9.34% as shown in Table (4). Tetratetracontane was the highest area of them, but 1-Aminododecane was the lowest. There were twenty four compounds presence in stored wheat uninfected but not appeared in infected stored wheat, may be due to the presence of mites’ infestation.

Table (4): Volatile extracted from stored wheat infected by Caloglyphus berlesei after three months by GC-MS.

| No | RT (min) | Name                                      | Area sum% |
|----|----------|-------------------------------------------|-----------|
| 1  | 4.514    | 2-Hexadecanol                             | 2.69      |
| 2  | 8.09     | Benzaldehyde, p-isopropyl-                | 1.05      |
| 3  | 11.7     | Tetradecane                               | 0.82      |
| 4  | 11.868   | Nonadecane                                | 0.47      |
| 5  | 12.436   | Hexadecane                                | 0.54      |
| 6  | 12.626   | 2-octanamine                              | 0.37      |
| 7  | 12.974   | 1-AMINODODECANE                           | 0.35      |
| 8  | 13.301   | Benzaldehyde, 6-hydroxy-4-methoxy-2,3-dimethyl- | 0.23      |
| 9  | 13.6     | Cedrol                                    | 1.26      |
| 10 | 14.247   | Dimethyl caffeic acid                     | 0.54      |
| 11 | 14.538   | (-)-Isolongifolol                         | 2.16      |
| 12 | 14.858   | Ethyl 2,4,6-trimethoxycinnamate           | 2.18      |
| 13 | 15.124   | 6-Carbomethoxy-5,8-dimethoxy-1-tetralone  | 1.36      |
| 14 | 15.704   | QUERCETIN 7,3',4'-TRIMETHOXY              | 3.16      |
| 15 | 15.857   | Retinol                                   | 8.41      |
| 16 | 16.172   | Retinoic acid                             | 2.19      |
| 17 | 16.902   | 10-Undecenal                              | 1.99      |
| 18 | 17.198   | β-Santalol                                | 2.00      |
| 19 | 17.641   | 4-Octadecenal                             | 3.06      |
| 20 | 18.78    | Lupulon                                   | 2.43      |
| 21 | 19.44    | Phytol                                    | 4.33      |
| 22 | 20.3     | Palatinose                                | 3.45      |
| 23 | 20.961   | Enterodiol                                | 3.00      |
| 24 | 21.358   | Apigenin 8-C-glucoside                    | 6.17      |
| 25 | 21.89    | D-(+)-Cellobiase                          | 9.06      |
| 26 | 22.281   | Agathic acid                              | 4.05      |
| 27 | 22.3     | Methyl 3,3-dimethylhenicosanoate          | 6.73      |
| 28 | 23.6     | Tetratetracontane                         | 9.34      |
| 29 | 24.07    | Betulin                                   | 15.63     |

Organic volatile compounds extracted from stored maize:

Data in Table (5) showed that there were fifty compounds evaluated from stored maize by GC-MS. Isoshyobunone was the first matter appeared after 9.13 minutes with 7.4% area sum. Methyl 3,3-dimethylhenicosanoate was the final material appeared after 22.31 minutes with 0.2%, the lowest area sum, but Cymarin was the highest area with 9.83%. Maize infected by Caloglyphus berlesei after three months caused to presence of sixty six compounds, twenty of them were appeared for the first time. It seems that they refer to infection by Caloglyphus berlesei. These compounds were 2-Hexadecanol appeared at 4.804 min with area sum 2.64%; 2-Octadecane appeared at 11.774 min with area sum 0.37%; Nonadecane at 11.868 min with area sum 0.31%; n-Pentadecane at 11.92 min with area sum 0.25%; Dodecane, 1-methoxy-12.009 min with area sum 0.41%; Farnesane at 12.116 min with area sum 0.22%; Tridecane at 12.229 min with area sum 0.32%; Tetradecane, 3-methyl-12.323 min with area sum 0.66%; Undecane at 12.372 min with area sum 0.75%; Hexadecane at 12.427 min with area sum 0.39%; Heptadecane at 12.543 min with area sum 3.28%; 2-octanamine at 12.614 min with area sum 3%; 1-Aminododecane at 12.9 min with area sum 0.5%; Decanal at 15.427 min with area sum 0.56%; Dodecanedioic acid at 16.157 min with area sum 0.38%; 10-Undecenal at 16.972 min with area sum 1.27%; 4-Octadecenal at 17.58 min with area sum 9.34%.
1.43%; Docosanoic acid at 19.602 min with area sum 1.04%; Heptacosane at 19.886 min with area sum 1.53% and Tetratetracontane at 23.6 92 min with area sum 1.23% as shown in Table (6). Heptadecane was the highest area of them, but Farnesane was the lowest.

Table (5): Volatile extracted from stored maize after three months by GC-MS

| No | RT (min) | Name | Area sum% |
|----|----------|------|-----------|
| 1  | 9.13     | Isoshyobunone | 7.4       |
| 2  | 9.36     | Hexestrol | 6.28      |
| 3  | 9.9      | 2-(3,4-Dimethoxyphenyl)-6-methyl-3,4-chromanediol | 7.58      |
| 4  | 10.8     | 2,3-Dehydro-4-oxo-β-ionol | 3.7       |
| 5  | 12.73    | Santonin | 2.46      |
| 6  | 13.05    | Cymarin | 9.83      |
| 7  | 13.3     | Benzaldehyde, 6-hydroxy-4-methoxy-2,3-dimethyl- | 1.84      |
| 8  | 13.75    | Cedrol | 2.32      |
| 9  | 14.01    | Asarone | 1.26      |
| 10 | 14.12    | Methoprene | 3.33      |
| 11 | 14.28    | Dimethyl caffeic acid | 1.67      |
| 12 | 14.49    | (-)-Isolongifolol | 2.1       |
| 13 | 14.6     | Resorcinol, 2-(3,7-dimethyl-2,6-octadienyl)-5-pentyl- | 1.89      |
| 14 | 14.7     | Hexadecanoic acid, 2-methyl- | 2.5       |
| 15 | 14.8     | Ethyl 2,4,6-trimethoxycinnamate | 1.53      |
| 16 | 14.93    | Digitoxin | 1.63      |
| 17 | 15.05    | 6-Carbomethoxy-5,8-dimethoxy-1-tetralone | 2.92      |
| 18 | 15.2     | Sinapic acid | 3.46      |
| 19 | 15.34    | Cannabinol | 2.16      |
| 20 | 15.5     | Pentadecanoic acid | 0.81      |
| 21 | 15.71    | Retinol | 1.04      |
| 22 | 15.9     | Genistin | 1.05      |
| 23 | 16.07    | Retinoic acid | 1         |
| 24 | 16.2     | Oleic Acid | 1.09      |
| 25 | 16.24    | cis-9, cis-12-Octadecadienoic acid | 1.17      |
| 26 | 16.38    | Linoleic acid | 0.8       |
| 27 | 16.45    | Elaidic acid | 0.67      |
| 28 | 16.62    | α-Bisabolol | 1.39      |
| 29 | 16.7     | cis-Vaccenic acid | 3.52      |
| 30 | 16.8     | β-Carotene | 1.53      |
| 31 | 16.9     | Ledane | 0.81      |
| 32 | 17       | Tetradecanedioic acid | 0.63      |
| 33 | 17.3     | β-Santalol | 0.55      |
| 34 | 17.4     | Bulnesol | 0.86      |
| 35 | 17.6     | cis-10-Heptadecenoic Acid | 0.59      |
| 36 | 18.07    | 6-Aminocaproic acid | 0.48      |
| 37 | 18.18    | Flavone, 3,5,7-trimethoxy- | 5.72      |
| 38 | 18.3     | γ-Selinene | 0.65      |
| 39 | 18.507   | Palustrol | 0.44      |
| 40 | 18.7     | Arachidonic acid | 0.69      |
| 41 | 18.9     | Lupulon | 0.53      |
The presence of these compounds affected the area of other compounds, 2-Hexadecanol was appeared, but Tetratetracontane was the lowest. Tetradecanedioic acid was the highest area 11.58%. Hexestrol was the lowest area with 0.21%. All other compounds also changed in area. There were four compounds presence in stored maize uninfected but not appeared in infected stored maize, may be due to the presence of mites’ infestation. Methoprene at 14.12 min with area sum3.33 %; Resorcinol, 2-(3,7-dimethyl-1,6-octadienyl)-5-pentyl- at 14.6 min with area sum1.89 %; γ-Selinene at18.3 min w 0.5 β-Citronellol at 20.87 min with area sum 0.82%. Methoprene was the highest area of them and γ-Selinene was the lowest (Table 5). Several phytoseiid mites positively responded to mite-induced plant odors (e.g., de Boer et al. 2004; Maeda et al. 2006; Sabelis and van de Baan 1983; Sarmento et al. 2011; Shimoda et al. 2005).

Table (6): Volatile extracted from stored maize infected by Caloglyphus berlesei after three months by GC-MS

| No | RT(min) | Name                                      | Area sum% |
|----|---------|-------------------------------------------|-----------|
| 1  | 4.804   | 2-Hexadecanol                             | 2.64      |
| 2  | 8.512   | Isoshyobunone                              | 0.34      |
| 3  | 9.3     | Hexestrol                                 | 0.21      |
| 4  | 9.743   | 2-(3,4-Dimethoxyphenyl)-6-methyl-3,4-chromanediol | 0.31     |
| 5  | 10.769  | 2,3-Dehydro-4-oxo-β-ionol                  | 0.75      |
| 6  | 11.774  | Tetradecane                                | 0.37      |
| 7  | 11.868  | Nonadecane                                | 0.31      |
| 8  | 11.92   | n-Penta decane                            | 0.25      |
| 9  | 12.009  | Dodecane, 1-methoxy-                       | 0.41      |
| 10 | 12.116  | Farnesane                                 | 0.22      |
| 11 | 12.229  | Tridecane                                 | 0.32      |
| 12 | 12.323  | Tetradecane, 3-methyl-                     | 0.66      |
| 13 | 12.372  | UNDECANE                                  | 0.75      |
| 14 | 12.427  | Hexadecane                                | 0.39      |
| 15 | 12.543  | Heptadecane                               | 3.28      |
| 16 | 12.614  | 2-octanamine                              | 3.09      |
| 17 | 12.7    | Santonin                                  | 0.92      |
| 18 | 12.9    | 1-AMINODODECANE                           | 0.5       |
| 19 | 12.956  | Cymarin                                   | 0.75      |
| 20 | 13.282  | Benzaldehyde, 6-hydroxy-4-methoxy-2,3-dimethyl- | 1.27     |
| 21 | 13.783  | Cedrol                                    | 0.27      |
| 22 | 14.101  | Asarone                                   | 0.24      |
| 23 | 14.232  | Dimethyl caffeic acid                      | 0.58      |
| 24 | 14.526  | (-)-Isolongifolol                         | 2.21      |
| 25 | 14.712  | Hexadecanoic acid, 2-methyl-              | 0.83      |
| 26 | 14.849  | Ethyl 2,4,6-trimethoxycinnamate            | 0.32      |
| 27 | 15.017  | Digi toxin                                | 0.71      |
| 28 | 15.182  | 6-Carbomethoxy-5,8-dimethoxy-1-tetralone  | 0.4       |
| 29 | 15.252  | Sinapic acid                              | 1.1       |
| 30 | 15.368  | Cannabinol                                | 0.86      |

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Organic volatile compounds extracted from stored fishmeal:

Data in (Table 7) showed that there were twenty seven compounds evaluated from stored fishmeal by GC-MS. Benzoic acid, 2-Hydroxy-, Methyl ester was the first matter appeared after 10.16 mintues with 8.19% area sum. Betulin was the final material appeared after 24.016 minutes with the highest area 13.81%, 2,3-Dehydro-4-oxo-β-ionol was the lowest area sum 0.84%. Fishmeal infected by *Caloglyphus berlesei* after three months caused to presence of forty six compounds, twenty five of them were appeared. It seems that they refer to infection by *Caloglyphus berlesi*.

Table (6): Volatile extracted from stored maize infected by *Caloglyphus berlesei* after three months by GC-MS (Continue)

| No | RT(min) | Name                          | Area sum% |
|----|---------|-------------------------------|-----------|
| 31 | 15.427  | Decanal                       | 0.56      |
| 32 | 15.494  | Pentadecanoic acid            | 1.61      |
| 33 | 15.72   | Retinol                       | 0.74      |
| 34 | 15.943  | Genistin                      | 3.29      |
| 35 | 16.086  | Retinoic acid                 | 0.86      |
| 36 | 16.157  | Dodecanedioic acid            | 0.38      |
| 37 | 16.202  | Oleic Acid                    | 0.42      |
| 38 | 16.245  | cis-9, cis-12-Octadecadienoic acid | 0.36    |
| 39 | 16.367  | Linoleic acid                 | 0.56      |
| 40 | 16.407  | Elaidic acid                  | 0.58      |
| 41 | 16.636  | α-Bisabolol                   | 1.16      |
| 42 | 16.7    | cis-Vaccenic acid             | 1.34      |
| 43 | 16.807  | β Carotene                    | 1.36      |
| 44 | 16.966  | Ledane                        | 1.58      |
| 45 | 16.972  | 10-Undecenal                  | 1.27      |
| 46 | 17.109  | Tetradecanedioic acid         | 11.58     |
| 47 | 17.27   | β-Santol                      | 7.45      |
| 48 | 17.452  | Bulnesol                      | 2.36      |
| 49 | 17.58   | 4-Octadecenal                 | 1.43      |
| 50 | 17.745  | cis-10-Heptadecenoic Acid     | 2.4       |
| 51 | 18.075  | 6-Aminocaproic acid           | 3.88      |
| 52 | 18.206  | Flavone, 3,5,7-trimethoxy-    | 0.89      |
| 53 | 18.54   | Palustrol                     | 1.28      |
| 54 | 18.65   | Arachidonic acid              | 2.54      |
| 55 | 18.841  | Lupulon                       | 1.66      |
| 56 | 19.3    | Phytol                        | 3.14      |
| 57 | 19.602  | Docosanoic acid               | 1.04      |
| 58 | 19.886  | Heptacosane                   | 1.53      |
| 59 | 20.29   | Palatinose                    | 1.66      |
| 60 | 20.766  | Enterodiol                    | 2.24      |
| 61 | 21.337  | Apigenin 8-C-glucoside        | 1.69      |
| 62 | 21.478  | Nabilone                      | 1.09      |
| 63 | 21.754  | D(+) Cellobiose               | 2.77      |
| 64 | 21.817  | Agatholic acid                | 1.41      |
| 65 | 22.3    | Methyl 3,3-dimethylhenicosanoate | 0.66  |
| 66 | 23.692  | Tetratetracontane             | 1.23      |
at 15.246 min with area sum 0.57%; Decanal at 15.423 min with area sum 0.99%; Oleic Acid at 16.208 min with area sum 0.55%; Linoleic acid at 16.37 min with area sum 0.81%; 4-Octodecenal at 17.58 min with area sum 0.85%; Arachidonic acid at 18.642 min with area sum 0.63%; Heptacosane at 19.886 min with area sum 0.96%; Palatinose at 20.2 min with area sum 0.89%; Enterodiol at 20.408 min with area sum 1.29%; Apigenin 8-C-glucoside at 21.33 min with area sum 1.08% and Tetratettrac octane at 23.685 min with area sum 1.91% as shown in (Table 8).

There were six compounds presence in stored fishmeal uninfected but not appeared in infected stored fishmeal, may be due to the presence of mites prevent them. Pentadecanoic acid at 15.683 min with area sum 4.2%; Ledane at 16.911 min with area sum 3.88%; cis-10-Heptadecenoic Acid at 17.595 min with area sum 3%; β-Citronellol at 20.955 min with area sum 7.19% ; Agathatic acid at 21.926 min with area sum 4.69% and Betulin at 24.016 min with area sum 13.81%.Betulin was the highest area of them and cis-10-Heptadecenoic Acid the lowest (Table 7).

Table (7) : Volatile extracted from stored fishmeal after three months by GC-MS

| No | RT (min) | Name                                           | Area sum% |
|----|----------|------------------------------------------------|-----------|
| 1  | 10.16    | BENZOIC ACID, 2-HYDROXY-, METHYL ESTER         | 8.19      |
| 2  | 10.8     | 2,3-Dehydro-4-oxo-β-ionol                      | 0.84      |
| 3  | 12.696   | Santonin                                       | 1.86      |
| 4  | 12.956   | Cymarin                                        | 1.56      |
| 5  | 13.6     | Cedrol                                         | 4.31      |
| 6  | 14.147   | Asarone                                        | 3.46      |
| 7  | 14.232   | Dimethyl caffeic acid                          | 2.9       |
| 8  | 14.522   | (-)-Isolongifolol                              | 2.44      |
| 9  | 14.849   | Ethyl 2,4,6-trimethoxycinnamate                | 1.14      |
| 10 | 15.179   | 6-Carboxyhex-5,8,10,14-tetralone               | 2.34      |
| 11 | 15.362   | Cannabinol                                     | 1.86      |
| 12 | 15.683   | Pentadecanoic acid                             | 4.2       |
| 13 | 15.943   | Genistin                                       | 2.4       |
| 14 | 16.153   | Dodecanedioic acid                             | 2.36      |
| 15 | 16.627   | α-Bisabolol                                     | 3.1       |
| 16 | 16.911   | Ledane                                         | 3.88      |
| 17 | 17.106   | Tetradecanedioic acid                          | 1.11      |
| 18 | 17.244   | β-Santalol                                     | 2.01      |
| 19 | 17.467   | Bulnesol                                       | 1.54      |
| 20 | 17.595   | cis-10-Heptadecenoic Acid                      | 3         |
| 21 | 19.382   | Phytol                                         | 2.72      |
| 22 | 19.62    | Docosanoic acid                                | 6.16      |
| 23 | 20.955   | β-Citronellol                                  | 7.19      |
| 24 | 21.492   | Nabilone                                       | 3.19      |
| 25 | 21.926   | Agathatic acid                                 | 4.69      |
| 26 | 22.3     | Methyl 3,3-dimethylhenicosanoate               | 7.71      |
| 27 | 24.016   | Betulin                                        | 13.81     |

From the previous data about volatile compounds in stored products (soybean, wheat, maize and fishmeal) it appeared that 1-aminododecane and 10-decenal were related with Caloglyphus berlesi in all stored products. These might be due to potential biomarkers. Mites, which are often included with insects in discussions of stored grains and cereal products, have been investigated for odors and volatiles in infested grains (Tuma, et al. 1990). Tridecane is a major compound produced by mites.

These results agree with Kuwahara, et al. (1980) whom which found that several volatiles produced by mites. Curtis, et al. (1981) they found that headspace above two strains of Acarus siro maintained on wheat germ/bran has been shown to contain the hydrocarbons, decane, undecane and tridecane together with the furanoid terpene, perillen. When the bodies of the separated mites were extracted with diethylether a new compound was found in addition to those in the headspace, also observed tridecane for Acarus siro. 1-Pentadecene and hexadecane were found in both adults of T. confusum and wheat flour infested with T. confusum beetles. 1-Pentadecene is associated with insect odour (Seitz and Sauer 1996). Also, (Geiselhardt,
et al., 2008) gave out 1 tridecene and they labeled it as a male sex pheromone produced from sperm of *Parastizopus* (Coleoptera;Tenebrionidae) and extracted the secretion and analyzed it using GC/MS. This confirmed the presence of tridecane. Abuelnnor, *et al.* (2010) found that the *T. confusum* larvae specifically emitted the volatiles decaanal.

### Table (8): Volatile extracted from stored fishmeal infected by *Caloglyphus berlesei* after three months by GC-MS

| No | RT (min) | Name | Area sum% |
|----|----------|------|-----------|
| 1  | 4.56     | 2-Hexadecanol | 2.06 |
| 2  | 9.819    | 2-(3,4-Dimethoxyphenyl)-6-methyl-3,4-chromanediol | 0.64 |
| 3  | 10.161   | BENZOIC ACID, 2-HYDROXY-4-METHYL ESTER | 4.65 |
| 4  | 10.802   | 2,3-Dehydro-4-oxo-β-ionol | 53.97 |
| 5  | 11.774   | Tetradecane | 0.75 |
| 6  | 12.012   | Dodecane, 1-methoxy- | 0.57 |
| 7  | 12.122   | Farnesane | 0.62 |
| 8  | 12.326   | Tetradecane, 3-methyl- | 0.81 |
| 9  | 12.372   | UNDECANE | 0.73 |
| 10 | 12.424   | Hexadecane | 0.57 |
| 11 | 12.558   | Heptadecane | 0.65 |
| 12 | 12.613   | 2-octanamine | 1.95 |
| 13 | 12.684   | Santonin | 0.91 |
| 14 | 12.9     | 1-AMINODODECANE | 0.55 |
| 15 | 12.955   | Cymarin | 0.63 |
| 16 | 13.371   | Benzaldehyde, 6-hydroxy-4-methoxy-2,3-dimethyl- | 0.98 |
| 17 | 13.786   | Cedrol | 0.56 |
| 18 | 14.147   | Asarone | 1.1 |
| 19 | 14.232   | Dimethyl caffeic acid | 1.53 |
| 20 | 14.522   | (-)-Isolongifolol | 0.79 |
| 21 | 14.846   | Ethyl 2,4,6-trimethoxycinnamate | 0.59 |
| 22 | 15.081   | Digitoxin | 1.04 |
| 23 | 15.182   | 6-Carbomethoxy-5,8-dimethoxy-1-tetralone | 1.08 |
| 24 | 15.246   | Sinapic acid | 0.57 |
| 25 | 15.39    | Cannabinol | 0.66 |
| 26 | 15.423   | Decanal | 0.99 |
| 27 | 15.942   | Genistin | 1 |
| 28 | 16.15    | Dodecanedioic acid | 0.72 |
| 29 | 16.208   | Oleic Acid | 0.55 |
| 30 | 16.37    | Linoleic acid | 0.81 |
| 31 | 16.63    | α-Bisabolol | 0.93 |
| 32 | 16.95    | 10-Undecenal | 1.61 |
| 33 | 17.109   | Tetradecanedioic acid | 0.77 |
| 34 | 17.244   | β-Santalol | 0.55 |
| 35 | 17.466   | Bulnesol | 0.75 |
| 36 | 17.58    | 4-Octadecenal | 0.85 |
| 37 | 18.642   | Arachidonic acid | 0.63 |
| 38 | 19.4     | Phytol | 0.84 |
| 39 | 19.632   | Docosanoic acid | 0.59 |
| 40 | 19.886   | Heptacosane | 0.96 |
| 41 | 20.2     | Palatinose | 0.89 |
| 42 | 20.408   | Enterodiol | 1.29 |
| 43 | 21.33    | Apigenin 8-C-glucoside | 1.08 |
| 44 | 21.492   | Nabilone | 0.93 |
| 45 | 22.641   | Methyl 3,3-dimethylhenicosanoate | 1.55 |
| 46 | 23.685   | Tetratetracontane | 1.91 |
CONCLUSION

This work showed that the infection of stored products (Soybean, Wheat, Maize and Fishmeal) by Caloglyphus berlesei has the potential to unfavorably influence. Thus, a greater understanding of mites. Stored products interactions and chemical ecology is needed for the success of management practices. The manipulation of volatile emission in mites has enormous potential for agricultural applications.

These specific volatiles may act as semi chemicals for these mites and could aid in semi chemical monitoring for the early detection of infestation by these mites.

Further research is needed to assess the organic volatile compound production capabilities of other species of stored – grain mites reared under laboratory conditions.

REFERENCES

Abuelnnor, N.A.; Jones, P.R.H.; Ratcliffe, N.M.; Lacy Costello, B.D. and Spencer-Phillips, P.T.N. (2010). Investigation of the semiochemicals of confused flour beetle Tribolium confusum Jaquelinduval and grain weevil Sitophilus granarius (L.) in stored wheat grain and flour.10th International Working Conference on Stored Product Protection 72 Julius-Kühn-Archiv. 72 - 76.

Bruin, J., Sabelis, M.W. and Dicke, M. (1995). Do plants tap SOS signals from their infested neighbours? Trends Ecol. Evol. 4: 167–170.

Curtis, R.F.; Frohock,H.; Fenwick, G.R. and Berreen, J.M. (1981). Volatile compounds from the mite Acarus siro L. in food. J. of Stored Products Research. 17(4): 197–203.

de Boer J.G., Posthumus M.A., Dicke M. (2004). Identification of volatiles that are used in discrimination between plants infested with prey or non-prey herbivores by a predatory mite. J Chem Ecol 30:2215–2230

Dicke, M. (1988). Prey preference of the phytoseiid mite Typhlodromus pyri. I. Response to volatile kairomones. Exp. & Appl. Acarology. 3: 1–13.

Dicke, M., Sabelis, M.W., Takabayashi, J., Bruin, J. and Posthumus, M.A. (1990). Plant strategies of manipulating predator-prey interactions through allelochemicals: prospects for application in pest control. J. Chem. Ecol. 11: 3091–3118.

Geiselhardt, S.; Ockenfels, P. and Peschke, K. (2008). 1-Tridecene male produced sex pheromone of the tenebrionid beetle Parastizopus transgariepinus. Natur wissens chaften. 95: 247 251.

Gulati, R. and Mathur, S. (1995). Effect of Eucalyptus and Mentha leaves and Curcuma rhizomes on Tyrophagus putrescentiae (Schrank) (Acari: Acaridae) on wheat. Exp. & Appl. Acarology. 19: 511-518.

Hubert, J. (2011).The pest importance of stored product mites (Acari:Acaridae).Animal Science, Issues and Professions,Nova Science Publishers,Inc.New York,46pp.

Koveos, D.S., Kouloussis, N.A. and Broufas, G.D. (1995). Olfactory responses of the predatory mite Amblyseius andersoni Chant (Acari, Phytoseiidae) to bean plants infested by the spider mite Tetranychus urticae Koch (Acari, Tetranychidae). J. Appl. Entomol. 9: 615–619.

Kuwahara, Y.;Matsumoto, K. and Wada, Y. (1980). Pheromone study on acarid mites IV. Citral: Composition and function as an alarm pheromone and its secretory glands in four species of acarid mites. Ipn.I. Sanit. ZooL, 31: 73-80.

Langenheim, J.H. (1994). Higher plant terpenoids: A phytocentric overview of their ecological roles. J. Chem. Ecol. 6: 1223–1280.
Maeda T., Liu Y., Ishiwarri H., Shimoda T. (2006.) Conditioned olfactory responses of a predatory mite, Neoseiulus womersleyi, to volatiles from prey-infested plants. Entomol Exp Appl 121:167–175

Musken, H.; Franz, J.T.; Wahl, R.; Paap, A.; Cromwell, O.; Masuch, G. and Bergmann, K.C. (2003). Sensitization to different mite species in German farmers: in vitro analyses. J. of Investigational Allergology and Clinical Immunology. 13(1): 26-35.

Pallini, A., Janssen, A. and Sabelis, M.W. (1997). Odour-mediated responses of phytophagous mites to conspecific and heterospecific competitors. Oecologia 110: 179–185.

Peñuelas, J., Llusia, J. and Estiarte, M. (1995). Terpenoids: A plant language. Trends Ecol. Evol.7, 289.

Ridgway, C.; Chambers, J.; Portero-Larragueta, E. and Prosser, O. (1999). Detection of mite infestation in wheat by electronic nose with transient flow sampling. J. of the Science of Food and Agriculture. 79(15): 2067-2074.

Sabelis M.W. and H. van de Baan (1983). Location of distant spider mite colonies by phytoseid predators: demonstration of specific kairomones emitted by Tetranychus urticae and Panonychus ulmi. Entomol. Exp. Appl., 33:303–314.

Sarmento R.A., F. Lemos, P.M. Bleeker, R.C. Schuurink, A. Pallini, M.G.A. Oliveira, E.R. Lima, M.W. Sabelis, A. Janssen (2011). A herbivore that manipulates plant defence. Ecol Lett 14:229–236.

Seitz, L. M. and D. B. Sauer (1996). Volatile Compounds and Odors in Grain Sorghum Infested with Common Storage Insects. Cereal Chem. 73(6):744-750.

Turlings, T.C.J., J.H. Tumlinson and W.J. Lewis (1990). Exploitation of herbivore-induced plant odors by host-seeking parasitic wasps. Science 250: 1251–1253.

Toma, D.; R.N. Sinha; W.E. Muir and D. Abramson (1990). Odour volatiles associated with mite-infested bin-stored wheat. J. of chemical ecology. 16(3): 713-724.

Tuma, D.; R.N. Sinha; W.E. Muir and D. Abramson (1990). Odour volatiles associated with mite-infested bin-stored wheat. J. of chemical ecology. 16(3): 713-724.

Turlings, T.C.J., J.H. Tumlinson and W.J. Lewis (1990). Exploitation of herbivore-induced plant odors by host-seeking parasitic wasps. Science 250: 1251–1253.

Turlings, T.C.J., J.H. Tumlinson and W.J. Lewis (1990). Exploitation of herbivore-induced plant odors by host-seeking parasitic wasps. Science 250: 1251–1253.