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Phytochemicals from *Ficus sycomorus* L. leaves act as insecticides and acaricides

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Phytophagus insects cause damage and economic loss in crops. The indiscriminate application of synthetic products has led to various problems. Among all, bioactive natural compounds considered to have an activity because of the presence of several chemicals that can exert their activities both as fumigants and by direct contact. The bioactive phytochemicals from *Ficus sycomorus* leaves were analyzed by gas chromatography - mass spectrometry (GC-MS). Insecticidal and acaricidal of the bioactive components were also determined. Results show that the chemical analysis of vaporous from *F. sycomorus* leaves allowed identification of 1, 2 benzenedicarboxilic acid, diisooctyl ester (45.06%), n-Hexadecanoic acid (7.67), 1H-pyrazole,4-Nitro (5.13), 3-Hexen-1- ol, benzoate,Z (4.57), oleic acid (4.30), hexanedioic acid, bis (2-ethyl hexyl) ester (4.15), methyl oleate (2.41), 3- buten- 2-one, 4-(2, 6, 6-trimethyl-1-cyclohexen-1-yl) (2.08), 9- octadecenoic acid (Z)- 2-hydroxy-1-(hydroxymethyl) ethyl ester (1.79), benzene methanol (1.59), Cycloheptasiloxane,tetradecamethyl (1.38), Z, Z- 3, 13- octadecadien-1-ol (1.31), 2- pentadecanone (1.27), 1-methylbicyclo [4.1.0] heptanes 1-methylnorcarane (1.06), L- linalool (1.04), cyclohexene (1.03) and methyl jasmonate (0.94) as main parts. The bioactive phytochemicals from *F. sycomorus* leaves were reported to be more toxic as the fumigant toxicity tests than the contact phase to the tested insects. The concentrations at 0.1, 0.01 and 0.001% were found to be repellent to adult females of *Tetranychus utricae,* followed by *Aphis craccivora* and *Sitophilus oryzae,* respectively. Here we demonstrate that bioactive phytochemicals from *F. sycomorus* leaves have a wide range of insecticidal and acaricidal and could become an alternative to synthetic pesticides for controlling certain important insects and mites.

Key words: *Ficus sycomorus* leaves, gas chromatography - mass spectrometry (GC-MS), *Sitophilus oryzae*, *Aphis craccivora, Tetranychus urticae.*

INTRODUCTION

In recent years, scientists have focused on the increase of food production needed for the fast expansion of the world population. Unfortunately, large yield losses occur because of insects and plant diseases caused by fungi, bacteria and viruses (Fletcher et al., 2006). Crop loss because of insect pests varies between 10 and 30% for major crops (Kordali et al., 2008). *Sitophilus* species are serious cosmopolitan pests of stored grain (Liu and Ho, 1999). Rice weevil (*Sitophilus oryzae* L.) is the main

representatives of this genus, which principally attack rice (*Oryza sativa* L.), maize (*Zea mays* L.), wheat (*Triticum sativum* Lam.), and sorghum (*Sorghum bicolor* (L.) Moench) among others, through direct feeding on grain kernels causing unfavorable effects on food quality, safety, and preservation (Rajendran and Sriranjini, 2008). The two-spotted spider mite, *Tetranychus urticae* Koch, is one of the most important pests of fruits, vegetable and ornamental plants and worldwide (Aslan et al., 2004).

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The mite has been reported to attack about 1200 species of plants of which more than 150 are economically important (Zhang, 2003). Spider mites have evolved resistance to more than 80 acaricides so far, and resistance has been reported from more than 60 countries (Pontes et al., 2007). *Aphis craccivora* Koch, (Aphididae), commonly known as black cowpea aphid, is one of the most important polyphagous insect-pests that colonizes, with a marked preference for legumes, plant hosts belonging to families Cruciferae, Cucurbitaceae, Asteraceae and Fabaceae throughout the world (Palumbo and Tickes, 2001). Synthetic insecticides and fumigants are widely used to control pests. However, the indiscriminate application of synthetic products has led to various problems including toxic residues in treating products, environmental pollution, and resistance against pesticides (Ye et al., 2010). Therefore, because of increasing drawbacks of the continued use of conventional fumigants, an effort is needed to develop new alternative pesticides to replace those being currently used.

Biofumigants have received much attention as pest control agents due to the presence of several modes of action, including insecticides, repellent or antifeedant properties (Viuda-Martos et al., 2010). Also, these products do not leave harmful residue to the environment, low toxicity in warm-blooded animals, high volatility and have medicinal properties for human, with lower toxicity to mammals (Cosimi et al., 2009). The search for new plant species with insecticide properties has been increasing in the past few years because of the indiscriminate use of synthetic pesticides for crop protection. Among bioactive natural compounds, several plant essential oils and plant extracts considered to have an activity because of the presence of several chemicals that can exert their activities both as fumigants and by direct contact. These active insecticide, repellent, antifeedant, and insect growth regulatory properties (Santos et al., 2011; Safia and Aoumeur, 2011).

Plants synthesize a broad range of secondary metabolites, including alkaloids and terpenoids, which are toxic to herbivores and pathogens, and so are believed to act as defense compounds (Wittstock and Gershenzon, 2002). These compounds can reduce insect development, interfere with digestion, and finally kill them (Navia-Giné´ et al., 2009).

Ficus sycomorus L., a medicinal plant belonging to the family Moraceae comprises about 755 fig tree species worldwide (Van Noort et al., 2007). *F. sycomorus* L. is widely distributed in tropical West Africa and grown in the Mediterranean basin of Egypt since antiquity and is known for their medicinal and aromatic properties (George and Lawrences, 1961). Phytochemical investigations on a few *Ficus* spp. were undertaken and led to identification of over 100 compounds. Most of these compounds are phenanthroindolizidine alkaloids from the leaves and stems of *Ficus hispida* and *Ficus septica* (Gao et al., 2004). Several coumarins were isolated from several different *Ficus* spp. (Chang et al., 2005) and multiple flavonoids have been identified from *Ficus* spp. stems, leaves, and roots (Li et al., 2007). Also, prominent were triterpenoids from the roots, leaves (Teixeira et al., 2006). Also, 54 different triacylgylcerols were identified in *Ficus carica* seed oil using mass spectrometry (Holcapek et al., 2005).

Yet, no reports were published about the volatile parts of *F. sycomorus* L. leaves as potential pesticides. Therefore, the aim of this study is to identify the chemical composition of *F. sycomorus* leaves by gas chromatography - mass spectrometry (GC-MS), and evaluate the fumigant, contact toxicity and repellency as an alternative to chemical insecticide against adult *S. oryzae, A. cracccivora*, and adult females of *T. urticae* under laboratory conditions.

MATERIALS AND METHODS

Plant materials

Leaves of *F. sycomorus* were collected in the middle of May 2012 from Aboutouala, Mania El-kamh province, Sharkia governorate, Egypt. The leaves were randomly collected from plant parts and shade dried. The plant material was dried naturally on laboratory benches at room temperature (28-30°C) for 5 days until crisp.

Isolation of volatiles from *F. sycomorus* leaves

A hundred grams of Sycamore leaves were placed in a flask and 400 ml of distilled water was added. Sycamore leaves were extracted by hydrodistillation using a Clevenger-type apparatus for 4 h. Water was heated to produce steam that carried the most volatile fractions of the aromatic material with it. The watery phase was extracted with dichloromethane (3×50 ml) and dried with anhydrous sodium sulphate. The dichloromethane solution of the volatiles was concentrated to 5 ml by evaporation under vacuum in a rotary evaporator at 30°C under reduced pressure. The volatile phase from *F. sycomorus* leaves was stored at 4°C prior to further analyses. The volatile phase in dichloromethane was performed for gas chromatography and mass spectrometry analysis. The volatiles yield calculated about the dry matter.

Gas chromatography-mass spectrometry analysis (GC/MS)

Volatile compound analysis was performed with a gas chromatography system (Aglient 6890 GC) with an Agilent mass spectrometric detector, with a direct capillary interface and fused silica capillary column HP-5 MS (30 m × 0.32 mm × 0.25 μm film thickness).

Helium was used as the carrier gas at a flow about 1.0 ml/min pulsed splitless. The solvent delay was 3 min. and the injection size was 1.0 μl. The mass spectrometric detector was operated in an electron impact ionization mode with an ionizing energy of 70 eV. Scanning from m/z 50 to 500 and the ion source temperature was 230ºC. The electron multiplier voltage (EM voltage) was maintained 1250 v above auto tune. The instrument was manually turned using perfluorotributyl amine (PFTBA). Oven temperature program at 45ºC (2 min), 150°C (5 min) at a rate of 2°C min⁻¹, then at 150ºC (2 min), 280°C (5 min) at a rate of 8°C min⁻¹; split 30:1 during 1.50

min, carrier gas He: 1 ml min^{-1}, constant flow; sample volume 1 µl. To identify the parts was based on comparison of their mass spectra with those of Wiley and Nist Tutore Libraries (Adams, 2001).

Insect cultures

Rice Weevil, *S. oryzae* were obtained from laboratory cultures kept in the dark in incubators at 28 ± 2°C and 70 to 80% relative humidity. The insects were reared on whole wheat grains at first 13 to 14% moisture content in a plastic container under the laboratory conditions at 28 ± 2°C, 75 ± 5% R.H. and a photoperiod of L12:D12 . The subcultures and the tests were carried out under the same conditions. Adult of *S. oryzae,* 7 to 14 days old were used for the experiments. For showing colonies of *T. urticae* (Koch) in the laboratory, individuals of adult females of the mite were collected from castor been, leaves (*R. communis* L.) while first adults *A. craccivora* (Koch.) from squash leaves (*Cucurbita pepo* L.) at Zagazig region, Egypt. Adult females of the mite and adult aphid were reared in the laboratory on kidney bean (*Phaseolus vulgaris* L.) plants grown in pots (12 × 10 cm) and maintained at 28 ± 2°C, 75 ± 5% R.H and a photoperiod of 16:8 (L:D). *A. craccivora* (Koch) and *T. urticae* (Koch), one-day-old adults were used.

Fumigant toxicity bioassay

S. oryzae

A 2.0 cm diameter filter paper (Whatman, No.2) was placed on the underside of the screw cap of a glass vial measuring 2.5 cm diameter, 5.5 cm height, and 24 ml volume. Ten micro liters of the volatiles (0.00, 0.30, 0.15, 0.075, 0.037 and 0.0187%) were added to the filter paper. The solvent was allowed to evaporate for 15 s before the cap was placed tightly on the glass vial, containing 20 insects, to form a sealed chamber. Dichloromethane was used as a control. Six replicates were used in all treatments and control and they were incubated at 28 ± 2°C and 70 to 80% relative humidity for 24 h. Mortality was determined 24 h after treatment. Results from all replicates were subjected to probit analysis using the EPA probit analysis program.1.1.3 to determine LC$_{50}$.

A. craccivora and T. urticae

Glass Petri plates (12 cm in diameter) were used as a mite-chamber for to determine volatile phase effect of *F.sycomorus* leaves. Ten adult of the same age from the stock colonies of *A. craccivora* and *T. urticae* (female) was transferred individually onto excised kidney bean (*Phaseolus vulgaris* L.) leaves (3 cm diameter) placed with its dorsal side on wet filter paper in a Petri dish using a soft paintbrush and allowed to settle for half an hour before exposure to the volatile phase of *F. sycomorus* leaves. The filter paper was saturated with sterile distilled water. The top of the mite-chamber was covered by using the other half of the Petri plate. Different concentrations of the volatile parts of *F. sycomorus* leaves were prepared at the concentrations of 0.0, 0.60, 0.30, 0.15, 0.075 and 0.0375%. The method of application of the volatile parts of *F. sycomorus* leaves, which aimed to prevent a direct contact between the mites or aphid and the volatile phase, has been described by Soylu et al. (2006). The volatile parts of *F. sycomorus* leaves were applied by micropipette on filter paper disks. The disks were previously placed on the inner surface of the inverted lid of the Petri dish, which were placed 20 mm from the center of the plate in order to prevent direct contact with the volatile phase. Plates were sealed immediately with paraffin to prevent loss of volatile phase from the plates. Three replications were made for each concentration. As an untreated control, three Petri dishes containing only 10 µl of dichloromethane were used. The treated mite-chambers were returned to the incubator set at 28±2°C, 65±2% RH and a photoperiod of 16:8 (L:D) h. Mortality was determined under a binocular light microscope 24 h after treatment. Probit analysis was used to determine lethal concentrations (LC$_{50}$ and LC$_{90}$), by using the program, version 1.1.3. Abbott's formula was used to correct mortality in controls.

Contact toxicity bioassay

S. oryzae

The contact effect of volatile parts from *F. sycomorus* leaves against pest was evaluated on filter paper discs (9 cm in diameter) which were treated with fumigants diluted dichloromethane. The filter papers were placed in glass Petri dishes (9 cm in diameter). The aliquot of 10 µl of volatile parts was applied to the filter paper discs, and the same aliquot of dichloromethane was applied to control evaluation. Volatile parts were carried out at the concentrations of 0.0, 0.30, 0.15, 0.075, 0.037 and 0.0187% for 24 h with six replicates of each control and treatment. The dichloromethane was allowed to evaporate for 10 min before the introduction of 20 adults in a Petri dish and these were kept in controlled temperature and humidity chamber at 28 ± 2°C, 65 ± 5% RH. The death insects was counted 24 h after the application. Results from all replicates were subjected to probit analysis using the EPA probit analysis program.1.1.3 to determine LC$_{50}$.

A. craccivora and T. urticae

A leaf-dipping method (Park et al., 2002) was used to evaluate the activity of the test samples. For adulticidal of the samples against *A. craccivora* and *T. urticae*, disks (5.4 cm diameter) of kidney bean (*Phaseolus vulgaris* L.) leaves (two weeks after germination) were used. Test samples were carried out at the concentrations of 0.0, 0.60, 0.30, 0.15, 0.075 and 0.0375%. Three leaf disks per concentration were separately dipped in each test solution for 30 s. Solvents were evaporated under a fume hood for 10 min. Ten adults of *A. craccivora* and ten adult female of *T. urticae* was transferred individually on treated and controled (disks treated with only dichloromethane) leaf disks placed in Petri plates. All treated samples were maintained at 28±2°C, 65±2% RH and a photoperiod of 16:8 (L: D). Mortality was determined 24 h after the application. Results from all replicates were subjected to probit analysis using the Environmental Protection Agency (EPA) probit analysis program.1.1.3 to determine LC$_{50}$.

Repellent activity bioassay

S. oryzae

The area preference test described by McDonald et al. (1970) was used to evaluate repellent action of *S. oryzae.* Test areas consisted of 9 cm filter paper No. 1 that was cut two parts. In the first part a concentration of 0.1, 0.01, and 0.001% of volatile parts from *F. sycomorus* leaves was applied as possible as uniformly with a micropipette. The other part (control) was only treated with 10 µl of dichloromethane. Both the treated part and the control part were air dried to evaporate the solvent. A full disc was carefully remade by attaching the treated part to the control part with adhesive paper tape. Each filter paper was placed in a Petri dish and 20 adults of *S. oryzae* were released in the center of each filter paper disc and covered. Each treatment was replicated five times. Number of insects on both treated (NT) and untreated halve (NC) were

Table 1. Compositions and Percentages of Volatiles from *F. sycomorus* leaves.

Peak	Retention time (min)	Compounds	Area (%)
1	5.74	N-ethyl-1, 3 dithioisoindoline	0.89
2	6.81	3-hexen-1-ol, (Z)	0.48
3	15.84	Benzene methanol	1.59
4	20.06	L- linalool	1.04
5	20.37	1-methylbicyclo [4.1.0] heptanes 1-methylnorcarane)	1.06
6	26.07	salicylic acid methyl ester	0.73
7	26.42	1, 3- cyclohexadiene-1-carboxaldehyde, 2, 6, 6-trimethyl	0.82
8	27.80	1-cyclohexene- 1- carboxaldehyde	0.71
9	41.14	Alpha- ionone	0.59
10	44.66	3- buten- 2-one, 4-(2, 6, 6-trimethyl-1-cyclohexen-1-yl)	2.08
11	45.70	Pentadecane	0.50
12	46.17	Cycloheptasiloxane,tetradecamethyl	1.38
13	46.88	2(4H) – benzofuranone, 5, 6, 7, 7a-tetrahydro-4, 4,7a-trimethyl	0.46
14	49.61	3-hexen-1- ol, benzoate, (Z)	4.57
15	50.00	Megastigmatrienone	0.46
16	51.03	3- hexadecene, (Z)	0.40
17	53.99	Methyl jasmonate	0.94
18	55.81	Silane	0.89
19	63.94	4-(3, 4-dimethoxybenzylidene) - 1- (4- nitro phenyl)- 3-phenyl-2-pyrzolin 5-one - 3- phenyl -2-pyrazoline- 5-one	0.68
20	65.49	2- pentadecanone	1.27
21	66.05	Hexadecanoic acid, methyl ester	0.49
22	66.85	n-Hexadecanoic acid	7.67
23	68.69	Z, Z-3, 13- octadecadien-1-ol	1.31
24	69.02	9- octadecenoic acid (Z), methyl ester (methyl oleate)	2.41
25	69.71	oleic acid	4.30
26	69.90	1H -pyrazole, 4-nitro	5.13
27	70.93	palmetoyl chloride	0.44
28	71.18	cyclohexene	1.03
29	71.42	5- heptadecenal	0.56
30	72.11	piperidine	0.78
31	72.69	Hexanedioic acid	4.15
32	72.87	9- octadecenoic acid (Z)- 2-hydroxy-1-(hydroxymethyl) ethyl ester	1.79
33	74.30	1, 2-benzenedicarboxilic acid, diisooctyl ester	45.06

recorded after 24 h. Percent repellency (PR) values for the test was computed as PR = [(NC-NT)/ (NC+NT)] ×100 (Obeng-Ofori and Reichmuth, 1997).

A. craccivora and T. urticae

The repellency of volatile parts from *F. sycomorus* leaves at 0.1, 0.01 and 0.001% against *A. craccivora* and *T. urticae*, were assessed. Leaf discs of kidney bean were placed in Petri-dishes lined with moist cotton wool. Half of each disc was painted with the proper concentration, while the other left untreated. Twenty females of *A. craccivora* and *T. urticae* was placed separately of the midrib. Orientation of *A. craccivora* or *T. urticae* on treated or control half was recorded after 24 h from the beginning of the experiment, also the number of eggs laid on the control versus the treated half was counted after 24 h. The percentage of repellency value was calculated.

Statistical analyses

The significance of the data was evaluated by the ANOVA one way test.

RESULTS AND DISCUSSION

Chemical composition of *F. sycomorus* leaves

The bioactive phytochemicals of *F. sycomorus* leaves were analyzed by using hydrodistillation and GC–MS. The results revealed 33 compounds representing 96.66% of the constituents (Table 1 and Figure 1). According to the analysis results, seventeen compounds represented 86.78% of the total mass of the bioactive parts were
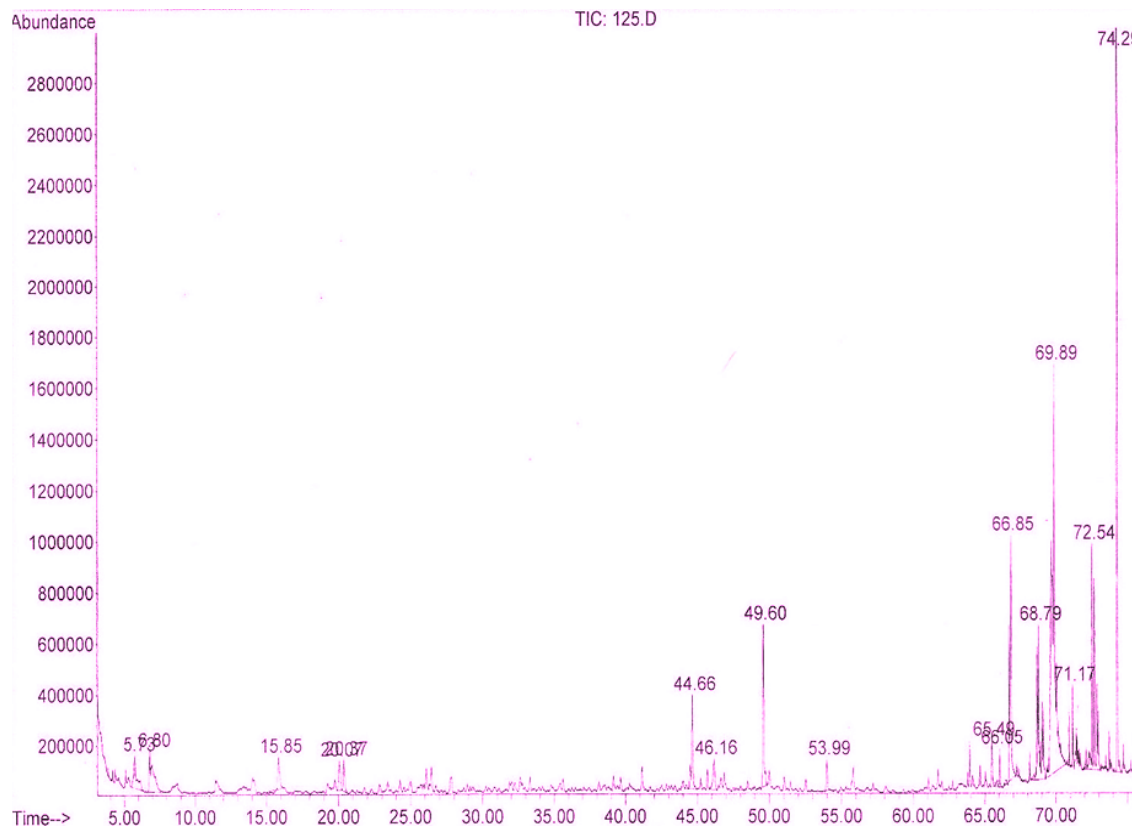
Figure 1. GC–MS volatiles chromatogram of *F. sycomorus* leaves.

identified as main parts. 1, 2-benzenedicarboxilic acid, diisooctyl ester (45.06%) was the most plentiful part of the volatiles in *F. sycomorus* leaves. Other main parts of the volatiles were found to be n-Hexadecanoic acid (7.67), 1H-pyrazole,4-nitro (5.13), 3-hexen-1- ol, benzoate,Z (4.57), oleic acid (4.30), hexanedioic acid bis (2-ethyl hexyl) ester (4.15), methyl oleate (2.41), 3-buten- 2-one, 4-(2, 6, 6-trimethyl-1-cyclohexen-1-yl) (2.08), 9- octadecenoic acid (Z)- 2-hydroxy-1-(hydroxymethyl) ethyl ester (1.79), benzene methanol (1.59), Cycloheptasiloxane, tetradecamethyl (1.38), Z, Z-3, 13- octadecadien-1-ol (1.31), 2- pentadecanone (1.27), 1-methylbicyclo [4.1.0] heptanes 1-methylnorcarane (1.06), L- linalool (1.04), cyclohexene (1.03) and Methyl jasmonate (0.94). The identified compounds have many biological properties. For instance, 1, 2-benzenedicarboxilic acid, diisooctyl ester (45.06%) was the most plentiful part of the volatiles in *F. sycomorus* leaves possesses antimicrobial activity (Ushadevi, 2008; Senthilkumar et al., 2011; Shafaghat et al., 2012).

1,2-Benzenedicarboxylic acid, di-isooctyl ester was present in *Caesalpinia sappan* ethanol extract (Sarumathy et al., 2011), in the ethanolic extract of the stem bark of *Schleichera oleosa* (51.15%), in *Dipteracanthus patulus* (9.89) (Gopalakrishnan et al., 2011) in the marine isolates of *P. lividum* and *T. lignorum*

(Ushadevi, 2008). These compounds (BTG 504 and BTG 505), identified as napthoquinones, are effective against a range of commercially important pests including the tobacco whitefly, *Bemisia tabaci*, aphids and the two-spotted spider mite, *T. urticae* (Khambay et al., 1999). N-hexadecanoic acid- palmitic acid (7.67) can be an antioxidant, hypocholesterolemic, nematicide, pesticide, lubricant (Praveen kumar et al., 2010). The major parts identified from *Euphorbia hirta* include hexadecanal, n-hexadecanoic acid was observed to have repellent against Anopheles species and thus useful for malaria control (Modupe et al., 2009). Hexadecanoic acid and 9-octadecenoic acid (Z), methyl ester - Oleic acid ester are known to have potent antibacterial and antifungal (Seidel and Taylor, 2004). Oleic acid in concentrations as low as 0.7% (v/v) has been found to be fungistatic against a wide of moulds and yeasts (Sheba et al., 1999). 1H-pyrazole, 4-Nitro (5.13%) is one among the seventeen compounds of the present study. Pyrazoles belong to one of the most important classes of heterocyclic compounds, which are significant for medicinal chemistry (Hoo et al., 2007).

Molecules of many modern drugs, e.g., antiphlogistic, antifungal, antidiabetic, and analgesic, as well on insectacaricide used in practice, contain a pyrazole ring as a fragment (Bildirici et al., 2007). The pyrazole ring

Table 2. Toxicity of volatiles from *F. sycamores* leaves against *S. oryza, T. urtica* and A. *craccivora.*

Insect	Life stage	LC$_{50}$ (%)	95% confidence limits	LC$_{90}$ (%)	95% confidence limits	Slope±SE
Contact toxicity						
S. oryzae	Adults	0.054[c]	0.01-0.12	0.668[c]	0.22-837.8	1.17±0.45
A. craccivora	Adults	0.213[b]	0.13-0.42	1.035[b]	0.49-9.37	1.87±0.51
T. urtica	Adults	0.350[a***]	0.01-0.12	3.424[a***]	0.89-9216.9	1.29±0.48
Fumigant toxicity						
S. oryzae	Adults	0.027[c]	0.001-0.05	0.313[b]	0.13-94.02	1.21±0.48
A. craccivora	Adults	0.084[b]	0.05-0.12	0.233[c]	0.15-0.60	2.90±0.72
T. urtica	Adults	0.129[a***]	0.09-0.2	0.367[a***]	0.24-0.96	2.81±0.65

***High significance.

has been shown to be the basic for a few antibacterial substances (Adnan et al., 2009). Hexadenoic acid has earlier been reported as a part in alcohol extract of the leaves of *Kigelia pinnata* (Grace et al., 2002) and *Melissa officinalis* (Sharafzadeh et al., 2011). Parasuraman et al. (2009) identified 17 compounds with n-Hexadecanoic acid and Octadecanoic acid as the major compounds in the leaves of *Cleistanthus collinus.* GC-MS analysis of ethyl acetate extract of *Goniothalamus umbrosus* revealed the presence of n-Hexadecanoic acid (Siddig et al., 2009). N-hexadecanoic acid, Hexadecanoic acid and 9, 12 - Octadecadienoic acid was identified in the ethanol leaf extract of *Aloe vera* (Arunkumar and Muthuselvam, 2009) and *Vitex negundo* (Praveen kumar et al., 2010). GC-MS of *Pyrenacantha staudtii* leaves showed the presence of tetradecanoic acid (22%) and hexanoic acid (13 %) as insecticidal against *Rhyzopertha dominica and Tribolium castaneum*, respectively (Abiodun et al., 2009). Devi et al. (2009) reported that *Euphorbia longan* leaves mainly contained n-hexadecanoic acid and 9, 12-Octadecadienoic acid. Hexadecanoic acid (51.55), 1,2 - Benzenedicarboxylic acid, bis (2-ethylhexyl) ester (20.19) and 1,2-Benzenedicarboxylic acid, dibutyl ester (2.32) from *Senna italica* subsp. were found to be more effective against adults of *Hyalomma marginatum rufipes* as anti-tick (Magano et al., 2008). Several plant essential oils and plant extracts contain several different bioactive compounds that may act individually, additively or in synergy to pest control (Viuda-Martos et al., 2010). It has been recognized that some plant-derived insect-control agents could be developed into products suitable for integrated pest management because they are high effectiveness, selective to pests, have no or little harmful affect against nontarget organisms or the environment (Jiang et al., 2007). These reports are by the result of this study.

Insecticidal and acaricidal

The bioactive phytochemicals effects of different concentrations of *F. sycomorus* leaves on the mortality of adults *S. oryzae, A. craccivora* and adult females of *T. urticae* are shown in Table 2. Data showed a highly toxic effect to all the tested insects. Volatile parts were found to cause a gradual increase in mortality with an increase in the concentration between 0.018 and 0.3% H. However, 100% adult mortality of *S. oryzae, A. craccivora* and *T. urticae* were recorded at 0.4% and above levels at 24 h of treatment. From the Table 2 it can be seen that, volatiles from *F. sycomorus* leaves was significantly more toxic to *S. oryzae* than *A. craccivora* and *T. urticae* as fumigant and contact toxicity tests. In fumigant toxicity assays, volatiles of *F. sycomorus* leaves were the most toxic one tested for *S. oryzae* where the LC$_{50}$ and LC$_{90}$ values were 0.027 and 0.313% followed by 0.084 and 0.233% for *A. craccivora*, then 0.129 and 0.376% for *T. urticae*. In contact toxicity assays, the corresponding LC$_{50}$ and LC$_{90}$ values were recorded 0.054 and 0.668% for S. *oryzae*, 0.213 and 1.035% for *A. craccivora*, 0.350 and 3.424% for *T. urticae*, respectively. At LC$_{50}$, the bioactive phytochemicals from *F. sycomorus* leaves was 4.47fold more toxic to *S. oryzae* and 1.53 fold more toxic to *A. craccivora* than that of *T. urticae* as a fumigant activity, respectively. The corresponding LC$_{50}$ were 6.48 fold more toxic to *S. oryzae* and 1.64 fold more toxic to *A. craccivora* than that of *T. urticae* as a contact, respectively. Volatiles from the *F. sycomorus* leaves were reported to be more toxic to all three insects as the fumigant toxicities than the contact toxicities. Probit analysis showed that at LC$_{50}$, the bioactive components were 2.0 fold more toxic to *S. oryzae,* 2.54 fold more toxic to *A. craccivora* and 2.71 more toxic to *T. urticae* as a fumigant test than that of contact test. The matching LC$_{90}$ were 2.13 fold more toxic to *S. oryzae,* 4.44 fold more toxic to *A. craccivora* and 9.33 more toxic to *T. urticae* as fumigant toxicities than that of contact toxicities. The contact and fumigant toxicities of various monoterpenes and how activity varies with structure was investigated. Our results clearly confirm the volatile parts from *F. sycomorus* leaves or their interaction possess insecticidal effectiveness against *S. oryzae, A. cracccivora* and acaricidal against *T. urticae*. Although many studies have

Table 3. Mean percent repellency (PR) values of volatile phase from *F.sycomorus* leaves against *S. ory zae, T. urtica* and *A. craccivora.*

Insects	0.10%	0.01%	0.001%
	Percent repellency (Mean % ± S.D) 24 h	Percent repellency (Mean % ± S.D) 24 h	Percent repellency (Mean % ± S.D) 24 h
Sitophilus oryzae	35.3±7.64	19.0±3.61	5.3±1.53[b]
Tetranychus urtica	80.0±4.51	43.0±2.51	23.0±2.08[a]
Aphis craccivora	42.0± 2.52	20.0± 1.73	0.00[c]
Significantly	N.S.	N.S.	***

N.S. NO significance, ***High significance.

shown the contact or fumigant efficacy gained from several plants against different insects and mite species (Santos et al., 2011), until, no published data were available on the efficacy of volatiles from *F. sycomorus* leaves as insecticidal and acaricidal against *S. oryzae, A. craccivora* and *T. urticae*. The insecticidal and acaricidal of volatiles from *F. sycomorus* leaves is attributed mainly to its major compounds. Each of the volatile parts has its own contribution on biological activity against the tested insect. The bioactive phytochemicals from the *F. sycomorus* leaves were reported to be more toxic to all the three insects as the fumigant toxicities than the contact toxicities. Volatile compounds of many plant extracts and essential oils are composed of alkanes, alcohols, aldehydes and terpenoids, monoterpenoids (Visser, 1986). Many of them show the fumigant (Ahn et al., 2006). Focus on the vapor or fumigant toxicity of essential oils of plants and their constituents has sharpened since the 1980s. Plant essential oils and their constituents almost have higher boiling points and such plant products that show insect toxicity in the vapor state have been recently reviewed by Rajendran and Sriranjini (2008). The volatile phases of the essential oils were reported to be more toxic than the contact phase to the microorganisms (Soylu et al., 2006). The mode of action of the tested botanical extracts may be largely attributable to its fumigant action (Park et al., 2003). *T. urticae* was more susceptible to the Piper oil by fumigation than by contact (Ma´rio et al., 2012). Toxicity in the fumigation bioassays most likely was attributed to penetrate the oil vapors in the respiratory (Ma´rio et al., 2012). The insecticidal varied with insect species, oil concentrations, exposure time and chemical composition of the oil, which in turn depends on the source, season and ecological conditions, method of extraction, time of extraction and plant part used (Lee et al., 2001). Akhtar and Isman (2004) showed that *Zanthoxylum alatum* was the most potent as a feeding deterrent and repellent, and also caused the highest mortality of Pieris brassiceae larvae.

Repellency

The bioactive parts from *F. sycomorus* leave showed

repellent against adults *S. oryzae, A. craccivora* and adult females of *T. urticae* at the concentrate 0.1, 0.01 and 0.001% (Table 3). The test concentrations were found repellent to adult females of *T. utricae*, followed by *A. craccivora* and *S. oryzae*, respectively. Repellency increased when the concentrations were increased. Obviously *T. urticae* females preferred nontreated areas over treated areas. Volatile constituent's evidence was significant at 0.001% for three insects while the repellency not statistically different at 0.1, 0.01% (Table 3). Some monoterpenes such as a-pinene, cineole, eugenol, limonene, terpinolene, citronellol, citronellal, camphor and thymol are common constituents of a few essential oils described in the literature, as presenting insect repellent (Sammour et al., 2011; Tayoub et al., 2012). Our results clearly confirm the volatile parts from *F. sycomorus* leaves or their interaction possess insecticidal effectiveness against *S. oryzae, A. cracccivora* and acaricidal against *T. urticae*.

Conclusion

In conclusion, The volatiles from *F. sycomorus* leaves have a wide range of insecticidal and acaricidal and could become an alternative to synthetic pesticides for using in agro-industries and to screen and develop such novel types of selectors and natural pesticides in the biocontrol of many agricultural plant insects and mites causing drastic losses to crops.

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