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Abstract: The *Diaphorina citri* Kuwayama (*D. citri*) is one of the major pests in the citrus industry, which spreads Citrus Huanglongbing disease. It has developed resistance to chemical insecticides. Therefore, searching for greener solutions for pest management is critically important. The main aim of this study was to evaluate the repellent and insecticidal efficacy of essential oils (EOs) from four species of *Myrtaceae* plants: *Psidium guajava* (PG), *Eucalyptus robusta* (ER), *Eucalyptus tereticornis* (ET), and *Baeckea frutescens* (BF) against *D. citri* and to analyze their chemical composition. GC-MS analysis was performed, and the results indicated that the EOs of PG, ER, ET, and BF were rich in terpenoids, ketones, esters, and alcohol compounds. The repellent rate of all four EOs showed that it decreased with exposure time but increased with concentrations of EOs. Repellent rates ranged from 80.50% to 100.00% after treating *D. citri* for 6 hours with four EOs at 100% concentration and decreased to 67.71% to 85.49% after 24 hours of exposure. Among the compounds from the EOs tested, eucalyptol had the strongest repellent activity, with a 24-hour repellency rate of 100%. The contact toxicity bioassay results showed that all EOs have insecticidal toxicity to *D. citri*; the LC50 for nymphs was 36.47-93.15 mL/L, and for adults, it was 60.72-111.00 mL/L. These results show that when PG is used as the reference material, both ER, ET, and BF EOs have strong biological activity against *D. citri*, which provides a scientific basis for the further development of plant-derived agrochemicals.

Keywords: essential oil; *Diaphorina citri*; insecticidal activity; repellent activity

1. Introduction

The Asian citrus psyllid, *Diaphorina citri* Kuwayama (Hemiptera: Psyllidae), is a globally important citrus pest. It feeds on leaf sap, causing leaf wilting and excreting on the leaves, leading to sooty mold pollution. Above all, it is a natural vector of Citrus Huanglongbing (HLB)[1, 2]. HLB is a bacterial disease caused by the bacterium *Candidatus* Liberibacter asiaticus (CLas), which occurs in the phloem tissue of citrus plants [3, 4]. Plants infected with CLas will gradually die within 2-4 years. Therefore, HLB has caused serious damage to the global citrus industry. Due to the current inability to cure HLB, controlling the citrus psyllid has become the primary measure for managing this disease[5].

At present, the use of chemical pesticide is the main measures for controlling *D. citri* in the field[6]. However, the extensive use of agrochemicals has led to serious environmental problems and the development of insecticide resistance[7, 8]. Three *D. citri* field populations in Florida, USA had developed high levels of resistance to the neonicotinoid agent thiamethoxam[9]. The resistance multiples of *D. citri* adults and 4th instar nymphs in three field populations in Mexico to malathion were 345-432 times, and to chlorpyrifos were 1424-2435 times, indicating extremely high levels of resistance[10]. Such a high resistance factor makes field control of citrus psyllids even more difficult. Therefore, it is of utmost urgency to develop environmentally friendly and non-agrochemical measures to control *D. citri*.

The olfactory receptor of insects is an important system that regulates their behaviors such as foraging, searching for mates, mating, laying eggs, and avoiding natural enemies [11, 12]. Odorant binding proteins (OBPs) are one of the olfactory proteins in insects, and among various olfactory proteins in insects, they play a major role in the perception of odor factors [13, 14]. OBPs are water-soluble macromolecular proteins. When insects sense and recognize odors in the environment, OBPs are responsible for binding and transporting these odorants [12]. Because of their role in insect signal transduction, OBPs are considered significant research targets for pest control [15]. OBPs had been identified from various Hemipteran insects, such as *Acyrthosiphon pisum* (15 OBPs)[16], *Sogatella furcifera* (12 OBPs)[17], *Bemisia tabaci* (8 OBPs)[18] and *D. citri* (9 OBPs)[19].

Plant-based natural products are a new research focus in agricultural pest control[20-22], including *D. citri*. Plant-derived bioactive compounds have advantages such as renewability, affordability, biodegradability, strong specificity, environmental friendliness, and no resistance to pests. They can be used as effective alternatives of chemical pesticides against pests of significant medical and veterinary importance, as well as in agriculture[23-26]. Moreover, some plants can emit highly volatile and irritating odor substances, which have a significant repellent effect on pests. Previous studies had shown that the odor substances of PG could have a strong repellent effect on *D. citri*[27-29]. Additionally, some natural products of plants also have a certain killing effect on pests, and they do not cause resistance or pollution to the environment, offering a new environmentally friendly way of pest control[30].

Psidium guajava (PG) is considered an important tropical fruit widely distributed in tropical and subtropical regions. PG fruit is very rich in nutritional elements and has been introduced into citrus production areas in southern China[31]. *Eucalyptus robusta* (ER) and *Eucalyptus tereticornis* (ET) are dense shade trees, both native to Australia and widely distributed in citrus producing areas in southern China[32, 33]. They are important timber plants, and their leaves can be used for medicine and have fumigation properties. *Baeckea frutescens* (BF) is a small shrub mainly distributed in subtropical regions and also found in southern China. Its leaves have a volatile odor and can be used as medicine[34]. The leaves of these four plants all contain volatile substances, thus having the potential to be used as plant-based pesticides for pest control.

The prevention and control of citrus psyllid in orchards requires a combination of multiple measures to achieve optimal results[35]. Therefore, using plant-based natural product as a repellent to control *D. citri* is a novel strategy. In this study, we screened four plants PG, ER, ET and BF, extracted their essential oils (EOs) through distillation, and tested their repellent and insecticidal efficiency against *D. citri* through bioassay experiments. Then, we identified the compound components of these four EOs, selected the major small molecule compounds with the highest content in each EOs, and conducted molecular docking experiments using DcitOBP7 from *D. citri* as the macromolecular target protein. Finally, behavioral experiments were conducted again using small molecule compounds with lower binding energies to support the results of molecular docking. We hope to develop new plant-based natural product repellents through this study, providing new strategies for non-pesticide control of *D. citri*.

2. Result

2.1. Repellent Bioassay of Eos

The comparison of the distribution quantity of *D. citri* on EO and control check (CK) of all EOs over time and concentration was shown in Figure 1, while the significant differences in their repellent rate was shown in Figure 2. When the concentration was 100% or 50%, there was a significant difference in the number of CK and EO selected by *D. citri* among all four EO treatments. When the concentration was 25%, there was no significant difference in the number of CK and EO selected by *D. citri* in the ER and ET EO treatments, ER only showed significant differences at 8, 10, 12, and 24 hours after treatment, and PG only showed no significant difference at 2 hours after treatment. When the concentration was 12.5%, there was no significant difference in the number of CK and EO selected by *D. citri* among all four EO treatments. Only at a concentration of 100% and after 4 hours of treatment, there was a significant difference in the repellent rates of PG and BF towards *D. citri*, while there was no significant difference in the repellent rates of the other four EOs at the same concentration and time period.
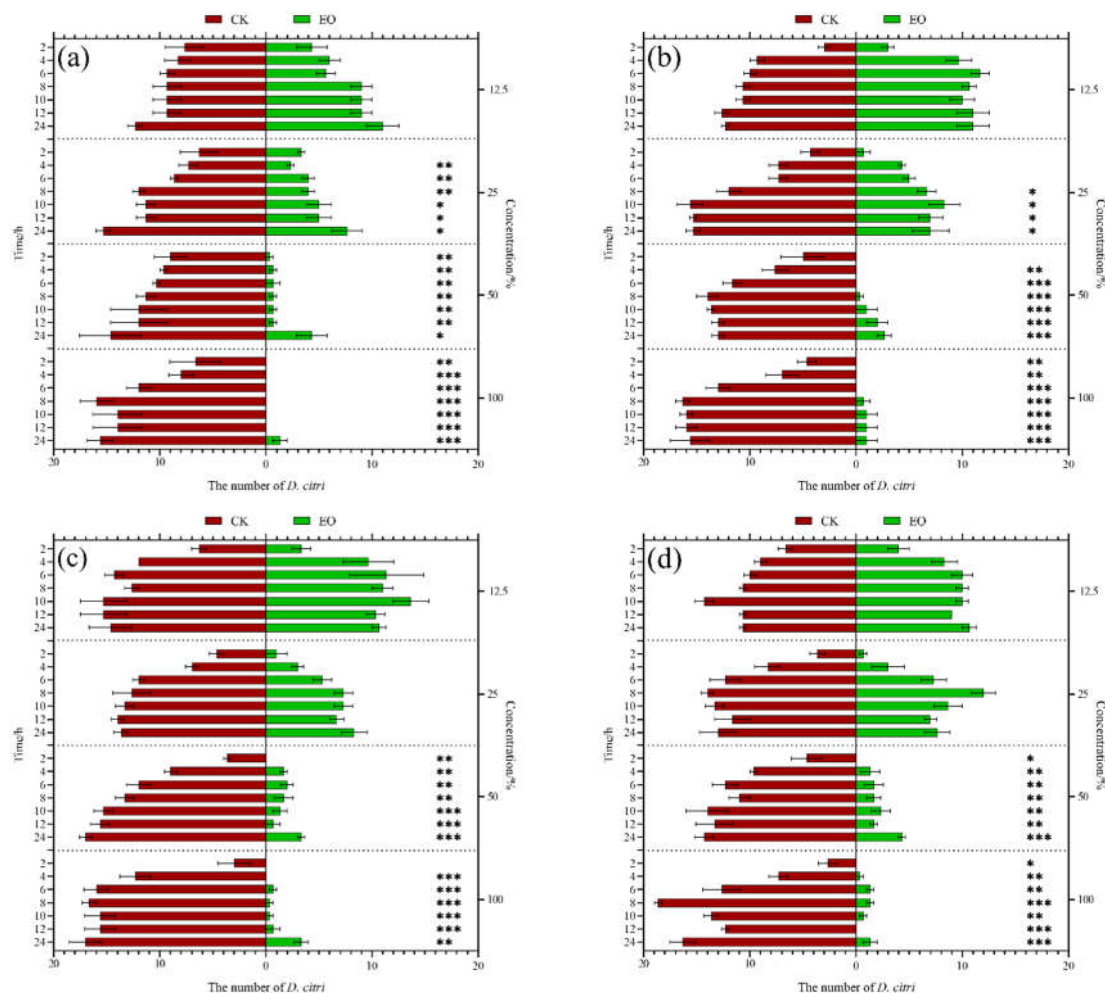

Figure 1. Comparison of the quantity of *Diaphorina citri* Kuwayama on tender shoots of *Murraya paniculata* with and without essential oil (EO) application. The red bar represents CK and the green bar represents EO. (a) PG. (b) ER. (c) ET. (d) BF. *$P < 0.05$, ** $P < 0.01$ and *** $P < 0.001$.
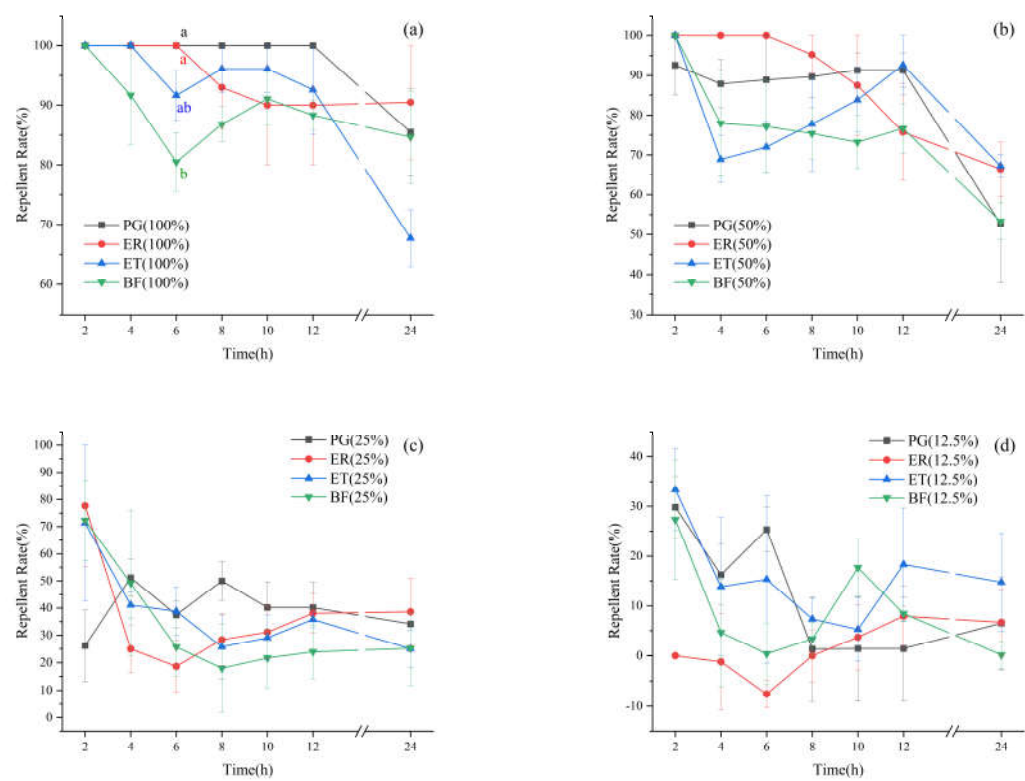
Figure 2. Analysis of significant differences in the repellent rate of *Diaphorina citri* by different concentrations of essential oils. **Note:** Different letters represent a statistically significant difference using one-way analysis of variance (HSD, *P* < 0.05).

2.2. Toxicity Bioassay

The toxicity data of citrus psyllids treated with all EOs for 24 hours were given in Table 1. The toxicity bioassay results showed that the LC_{50} of BF EOs on nymphs and adults were 36.47 mL/L and 60.72 mL/L, respectively, with the best effect. For PG, the LC50 values were 93.15 mL/L for nymphs and 111.00 mL/L for adults. ER exhibited LC50 values of 53.85 mL/L for nymphs and 90.44 mL/L for adults, while ET showed LC50 values of 56.50 mL/L for nymphs and 77.19 mL/L for adults. The mortality rate of all EOs on nymphs was generally higher than that on adults.

Table 1. Toxicity of plant essential oils (EOs) against workers of *Diaphorina citri* at 24 hours post-treatment.

Stage	E.oil	n	Slope ± SEM	LC$_{50}$ (95% CI)	LC$_{90}$ (95% CI)	χ^2	df
Nymph	PG	90	1.80±0.20	93.15（77.13-115.63）	480.04（326.26-861.10）	1.90	3
	ER	90	1.40±0.18	53.85（42.39-73.36）	441.24（249.63-826.42）	1.31	3
	ET	90	1.11±0.18	56.50（41.76-86.80）	484.80（357.29-602.69）	0.84	3
	BF	90	1.63±0.17	36.47（30.27-44.79）	222.12（151.00-391.32）	3.06	3
Adult	PG	90	1.53±0.16	111.00（91.73-137.59）	766.79（506.96-1008.07）	1.83	3
	ER	90	1.11±0.15	90.44（70.31-119.15）	777.67（457.71-1084.19）	0.79	3
	ET	90	1.36±0.15	77.19（62.36-95.72）	680.89（438.03-926.14）	0.23	3
	BF	90	1.63±0.16	60.72（50.22-72.60）	370.69（270.22-578.70）	0.12	3

Note: LC_{50} and LC_{90} values were represented in mL/L and CIs were confidence intervals.

2.3. Chemical Analysis of the EOs

Chemical compositions of the EOs from the four plants were given in Table 2. A total of 121 compounds were identified from the four EOs. Terpenoids were the main components in all four

plant EOs, accounting for 65.31%, 44.00%, 46.91%, and 46.15%, respectively. Additionally, the compound with the highest content in PG is β-cubebene (9.42%), in ER was α-phellandrene (12.20%), in ET was α-pinene (15.59%), and in BF was o-cymine (13.62%). β-caryophyllene, which was considered the main repellent component of PG, was present in PG (6.15%), ER (1.04%), ET (0.72%), and BF (2.89%), with the highest content in PG.

Table 2. Chemical composition of four plants essential oils (EOs).

No.	Compounds	NIST RI	Relative abundance (%)			
			PG	ER	ET	BF
1	4-Hexen-3-one	855	-	0.51	-	-
2	Dimethyl sulfone	922	-	0.60	3.02	-
3	Benzene, (1-methylethyl)-	926.57	-	-	1.68	-
4	Cyclobutanespiro-2'-bicyclo[1.1.0]butane-4'-spirocyClobutane	930	-	1.10	5.85	0.68
5	α-Pinene	936.35	-	3.40	15.59	3.21
6	Cyclopentene, 1-butyl-	938	-	-	0.93	-
7	Bicyclo (3.3.1)non-2-ene	964	-	-	0.68	-
8	4-methyl-1- (1-methylethyl)-Bicyclo[3.1.0]hex-2-ene	966	-	-	1.21	-
9	Bicyclo[3.1.0]hexane, 4-methylene-1- (1-methylethyl)-	972	-	0.45	1.62	-
10	2,6-Octadiene, 2,6-dimethyl-	978	-	0.43	1.44	-
11	β-Pinene	979.71	-	1.81	6.25	0.70
12	1,7-Octadiene, 2-methyl-6-methylene-	984	-	0.38	1.31	-
13	DiSulfur compounds, ethyl 1-methylethyl	985	-	-	0.58	-
14	Pyridine, 3-propyl-	986	-	-	0.73	-
15	Benzene, (1-methylpropyl)-	1001	-	0.37	-	-
16	α-Phellandrene	1006	-	12.20	0.77	0.55
17	Terpilene	1018.03	-	-	-	-
18	4-Hexen-1-ol, acetate	1020	-	0.67	0.67	0.63
19	o-Cymene	1022	-	10.70	4.13	13.62
20	4,6-Octadiyn-3-one, 2-methyl-	1023	0.69	-	-	-
21	2-Azabicyclo[3.2.1]octan-3-one	1025	-	1.86	2.05	1.65
22	p-Cymene	1025.98	-	3.77	1.55	5.34
23	Limonene	1026	3.66	3.08	2.32	0.76
24	2-Methyl-1,3-dithiacyclopentane	1026	-	1.15	-	1.51
25	1,7-Nonadiene, 4,8-dimethyl-	1026	-	0.85	0.46	-
26	Thiazole, 5-ethenyl-4-methyl-	1027	-	0.76	0.93	0.63
27	Pyridine, 2,3,4,5-tetrahydro-6-propyl-	1028	-	0.56	0.65	-
28	Indane	1029	-	1.69	0.63	2.19
29	Cyclohexanol, 3,5-dimethyl-	1030	-	3.26	2.83	2.94
30	β-Phellandrene	1031	2.68	1.77	4.36	2.82
31	D-Limonene	1031.27	3.15	4.13	3.19	2.35
32	Eucalyptol	1034.33	-	5.91	6.87	4.31
33	3-Octen-2-one, (E)-	1035	-	3.23	3.92	2.89
34	Ocimene	1037	0.53	5.72	1.21	-
35	2-Acetyl-5-methylfuran	1037.22	-	0.50	0.57	-
36	(S)-2,5-Dimethyl-3-vinylhex-4-en-2-ol	1039	0.96	4.32	4.32	2.93
37	3-Octen-2-one	1040	-	1.89	1.40	1.55
38	BenzeneacetAldehyde	1045.59	-	0.48	-	-
39	(E)-β-Ocimene	1049	-	0.96	-	-
40	γ-Terpinene	1060.24	-	0.57	-	3.02
41	Benzenemethanol, α-methyl-	1061.21	-	-	-	0.94
42	trans-4-Thujanol	1070	-	-	-	1.82
43	BenzAldehyde, 3-methyl-	1070.12	-	0.48	-	2.37
44	(Z)-Pent-2-enyl butyrate	1091	-	-	-	1.77
45	Linalool	1100.58	-	-	-	0.98
46	6-Nonenal, (Z)-	1103.52	-	-	-	1.03
47	Pinocarveol	1138	-	-	0.91	-
48	Myrcenone	1145	-	-	0.52	-
49	p-Mentha-1 (7),2-dien-8-ol	1163	-	-	0.70	-
50	Pinocarvone	1164	-	-	1.41	-
51	Phenol, 4-ethyl-	1165.40	-	-	0.63	-
52	(E)-2,6-Dimethylocta-5,7-dien-2-ol	1169	-	-	-	1.66
53	Lavandulol	1170	-	-	-	1.36
54	Borneol	1170.41	-	-	0.68	-

No.	Compound	RI				
55	Terpinen-4-ol	1181.45	-	0.53	-	2.38
56	2-Butenoic acid, hexyl ester	1191	-	-	-	0.73
57	(-)-Dihydrocarveol	1192	-	-	-	0.56
58	α-Terpineol	1195.55	-	-	-	0.59
59	Benzamide	1344	-	0.46	-	-
60	2,3,5,9-tetramethyltricyclo[6.3.0.01,5]undec-3-ene	1348	-	4.20	-	-
61	Terpinyl acetate	1350	-	2.89	-	-
62	(1α,3β,4β)-p-menthane-3,8-diol	1355	-	0.61	-	-
63	Neryl acetate	1365.22	-	3.09	-	-
64	Methyl 4-aminobenzoate	1372	1.30	-	-	-
65	6,8-Nonadien-2-one, 8-methyl-5- (1-methylethyl)-, (E)-	1373	0.59	-	-	-
66	(-)-α-Copaene	1376	3.54	-	-	-
67	Di-epi-α-cedrene- (I)	1382	5.52	-	-	-
68	(-)-β-Bourbonene	1384	1.12	-	-	-
69	(-)-Modhephene	1385	0.63	-	-	-
70	Damascenone	1386	0.71	-	-	-
71	Acetic Acid, phenoxy-	1389	0.79	-	-	-
72	β-Cubebene	1390	9.42	-	-	-
73	Niacinamide	1419	0.80	-	-	-
74	Ethyl mandelate	1421	-	0.69	0.51	1.98
75	Benzoic Acid, 4-methoxy-	1424.27	1.49	-	-	-
76	Benzenemethanol, 4-hydroxy-	1426	1.42	-	-	0.61
77	3-Hexanone, 1-phenyl-	1427	5.14	0.87	0.60	2.34
78	2-Propenoic Acid, 3-phenyl-	1427.53	-	-	-	1.04
79	Quinoxaline, 2,3-dimethyl-	1428	0.50	-	-	-
80	(E,E)-2,4-Undecadienal	1430	2.16	0.40	-	1.08
81	(+)-Calarene	1432	5.08	0.36	-	1.43
82	β-Caryophyllene	1432.49	6.15	1.04	0.72	2.89
83	γ-Elemene	1433	2.27	-	-	0.92
84	Ethyl β-safranate	1434	3.22	0.47	-	1.31
85	trans-α-Bergamotene	1435	3.27	0.43	-	1.22
86	2-Hydroxymethylbenzimidazole	1437	3.14	-	-	0.96
87	Ethanone, 1- (3-hydroxyphenyl)-	1439	0.76	-	-	-
88	Azulene, 1,2,3,3a,6,8a-hexahydro-1,4-dimethyl-7- (1-methylethyl)-, (1R,3aS,8aS)-	1440	3.09	-	-	-
89	Naphthalene, 1,2,4a,5,8,8a-hexahydro-4,7-dimethyl-1- (1-methylethyl)-, (1α,4aβ,8aα)- (.+/-.)-	1440	3.34	-	-	0.99
90	Aromandendrene	1440	0.85	-	-	-
91	(+)-α-Muurolene	1440	-	0.62	-	1.67
92	Benzyl angelate	1446	1.25	-	-	-
93	-6-Methyl-2-methylene-6- bicyclo[3.1.1]heptane	1446	1.02	-	-	-
94	(-)-Aristolene	1447	2.08	-	-	-
95	Benzene, 1- (1,5-dimethylhexyl)-4-methyl-	1449	1.17	-	-	-
96	(-)-α-Himachalene	1449	1.11	-	-	-
97	Acetophenone, 4'-hydroxy-	1455	0.76	-	-	2.21
98	(E)-β-Famesene	1457	-	-	-	1.02
99	5,9-Undecadien-2-ol, 6,10-dimethyl-	1459	-	-	-	0.74
100	1,1'- (1,4-phenylene)bis-Ethanone	1461	0.67	-	-	-
101	Benzene, [1-[[1- (1-methylethyl)-3-butenyl]oxy]ethyl]-, [S- (R*,R*)]-	1463	-	-	-	0.91
102	2-Pinen-10-yl isobutyrate	1466	-	-	-	1.46
103	(1R,9R,E)-4,11,11-Trimethyl-8-methylenebicyclo[7.2.0]undec-4-ene	1466	-	-	-	0.90
104	Acoradiene	1471	-	-	-	0.64
105	(4R,4aS,6S)-4,4a-Dimethyl-6- (prop-1-en-2-yl)-1,2,3,4,4a,5,6,7-octahydronaphthalene	1476	0.57	-	-	-
106	Eudesma-2,4,11-triene	1479	0.54	-	-	-
107	(-)-Germacrene D	1481	1.52	-	-	-
108	3- (4-Hydroxyphenyl)propanal	1490	-	0.37	1.01	-
109	(1S,2E,6E,10R)-3,7,11,11-Tetramethylbicyclo[8.1.0]undeca-2,6-diene	1495	-	0.81	2.07	-
110	Benzyl tiglate	1498	-	-	0.68	-
111	α-Muurolene	1499	0.66	-	0.63	-
112	Epizonarene	1501	-	-	0.57	-
113	α-Cuprenene	1509	-	-	0.61	-
114	(E)-α-Bisabolene	1512	0.65	0.47	1.27	-
115	(-)-γ-Cadinene	1513	-	-	0.74	-
116	cis-Calamenene	1523	3.01	-	-	-
117	(+)-δ-Cadinene	1524	0.64	-	-	-

118	Cadinadiene,cadina-1,4-diene		1532	0.92	-	-	-
119	(+)-α-Cadinene		1538	0.89	-	-	-
120	β-Vetivenene		1540	0.50	-	-	-
121	3,7 (11)-Eudesmadiene		1542	1.29	-	-	-
	Total			97.21	97.89	97.97	96.79
	Terpineoids			65.31	44.00	46.91	46.15
	Ketone			12.24	10.00	8.16	9.61
	Ester			6.12	8.00	8.16	9.61
	Alcohol			4.08	6.00	6.12	13.46
	Acid			4.08	5.00	-	1.92
	Hydrocarbons			-	6.00	12.24	1.92
	Heterocyclic compound			4.08	8.00	8.16	5.77
	Aromatics			-	6.00	6.12	5.77
	Amine			2.04	2.00	-	-
	Aldehyde			2.04	8.00	2.04	5.77

Note："-" means not detected.

2.4. Repellent Bioassay of Compounds

Compounds with higher concentrations of various EOs were used to test their repellent activity, Dimethyl disulfide was included as a positive control drug, and the results were shown in Table 3. Within 6 hours of treatment, β-caryophyllene maintained a 100% repellent rate, while it decreased to 83.23-94.07% from 8 to 24 hours. α-pinene showed 100% effectiveness within 4 hours and decreases to 76.92-93.33% from the 6th to the 24th hour. Eucalyptol maintained a high repellent effect for 24 hours, which was 100% except for the 6th hour (94.86%) and the 8th hour (92.22%). o-cymine exhibited 100% efficacy within 4 hours and decreases to 54.77-91.91% within 6 to 24 hours. The repellent rates of limonene and (R)-(+)-limonene within 24 hours were -17.32-12.87% and -11.27-11.44%, respectively, with more attractive effects.

Table 3. Analysis of differences in repellent rates of different compounds on *Diaphorina citri* at the same time.

Time（h）		2	4	6	8	10	12	24
Compounds	CAS	Mean±SEM%						
β-Caryophyllene	87-44-5	100±0.00	100±0.00	100±0.00	94.07±2.97a	83.23±5.2abc	83.23±5.2ab	85.00±4.28ab
α-Terpinene	99-86-5	100±0.00	100±0.00	85.05±7.87abcd	67.72±2.69abcd	70.61±10.32abcd	68.15±10.76bcd	80.37±1.61abc
β-Pinene	127-91-3	80.61±11.56abc	50.27±8.93bc	42.06±4.83def	58.36±12.59cde	60.69±5.52bcd	73.45±4.92abc	72.01±5.01abc
Linalool	78-70-6	55.19±2.89bc	69.11±1.38ab	62.29±4.85bcd	54.94±1.6def	52.31±1.31cd	52.98±0.79cd	53.7±0.85c
Eucalyptol	470-82-6	100±0.00	100±0.00	94.86±2.57ab	92.22±4.01ab	100±0.00	100±0.00	100±0.00
α-Pinene	80-56-8	100±0.00	100±0.00	80.43±5.66abc	86.98±3.49abc	93.33±6.67a	87.13±3.57ab	76.92±4.47abc
Phellandrene	99-83-2	100±0.00	77.78±11.11ab	83.07±5.82abc	93.65±6.35a	84.13±11.45ab	72.26±5.56abcd	52.84±2.47c
Ocimene	13877-91-3	4.58±17.56de	-14.31±9.51e	-9.39±4.13h	-4.32±2.27hi	-4.32±2.27e	-4.53±2.43f	7.34±6.82d
D-Limonen	5989-27-5	-9.16±2.38e	-11.27±4.36e	-8.43±1.56h	-8.43±3.24i	-8.97±2.19e	11.44±3.23f	2.7±1.96d
γ-Terpinene	99-85-4	48.89±14.57bcd	26.83±8.04cd	28.92±1.31defg	25.41±4.81fgh	43.39±10.62d	53.33±4.63cd	20.08±6.76d
o-Cymene	527-84-4	100±0.00	100±0.00	91.91±4.05ab	71.42±5.72abcd	70.98±5.49abcd	63.14±1.57bcd	54.77±5.29c
Cineole	406-67-7	100±0.00	65.02±3.37abc	45.95±2.57def	63.24±7.25bcde	48.03±4.16d	79.35±10.62abc	65.72±9.22bc
1,4-Diethylbenzene	105-05-5	-1.06±7.35e	4.15±9.54de	-9.09±4.29h	-1.45±1.45hi	1.15±1.15e	1.76±7.54f	9.70±5.78d
Limonene	138-86-3	-10.82±10.64e	-17.32±8.52e	-9.09±4.29h	-9.09±4.29i	-9.09±4.29e	12.87±3.25f	3.20±1.62d
3-Carene	13466-78-9	8.91±13.76cde	1.14±5.46de	1.42±3.34gh	-10.07±4.36i	-9.97±4.43e	11.42±3.1f	3.70±6.42d

1-Phenylhexan-3-one	29898-25-7	8.38±16.9de	1.63±8.13de	17.21±12.83fgh	5.41±1.84hi	5.41±1.84e	16.76±3.32ef	21.56±1.52d
Myrtol	8002-55-9	65.02±3.37ab	39.09±5.09bcd	23.74±4.27efgh	12.27±2.58ghi	12.27±2.58de	12.27±2.58f	21.43±4.59d
df		16	16	16	16	16	16	16
F-Value		24.532	32.647	38.263	47.305	41.381	37.517	35.776
P-Value		0.001	<0.001	<0.001	0.005	0.001	0.001	0.001

Note: Different letters represent a statistically significant difference using one-way analysis of variance (HSD, $P < 0.05$).

2.5. Molecular Docking

In order to explore the mechanism of action of active compounds, six compounds with significant repellent activity were further tested for their binding abilities with DcitOBP7. And the results of molecular docking were shown in Figure 3, which illustrated the compounds' strong binding affinity to the protein pocket with a noteworthy docking score ranging between -5.9 and -7.3 kcal/mol. For all the docking analyses, a lower score indicated a better binding affinity. The molecular docking analysis revealed that α-Pinene had the best binding affinity of -7.3 kcal/mol with DcitOBP7. α-Pinene, β-Caryophyllene, α-Terpinene and β-Pinenewere docked at the same position as DcitOBP7, while Limonene and Eucalyptol were docked in different positions.

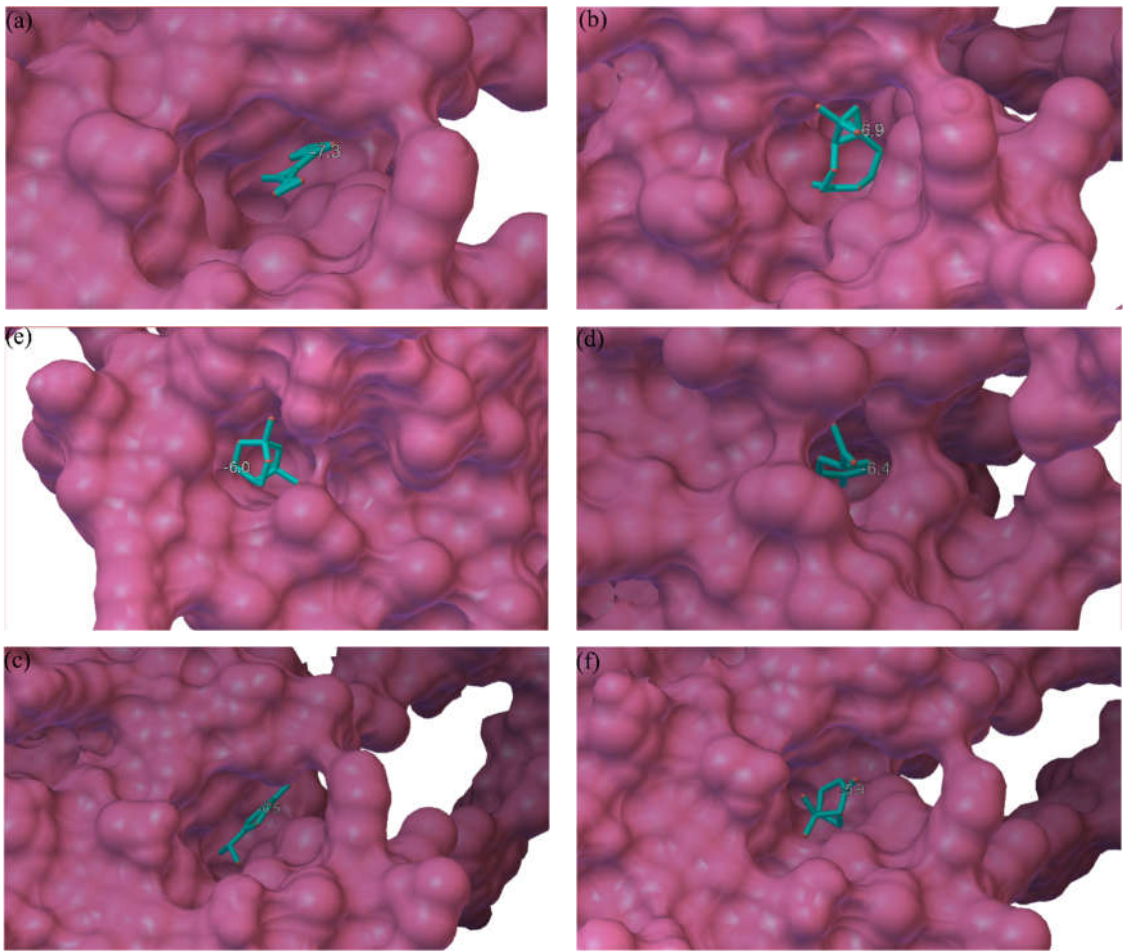**Figure 3.** Molecular docking results of six ligands with DcitOBP7. (a) α-Pinene. (b) β-Caryophyllene. (c) α-Terpinene. (d) Limonene. (e) Eucalyptol. (f) β-Pinene.

3. Discussion

In order to simulate the living habits of citrus psyllids and the actual situation in orchards, this study used arranged tender shoots in cages to test the repellent activity of EOs. So far, several studies had been conducted on the repellent efficiency of PG against citrus psyllids. *Gottwald et al.*[36] indicated that intercropping PG with citrus could reduce the infestation rate of citrus psyllids by 50-100% compared to planting citrus alone. *Zaka et al.*[37] indicated that when PG leaves were around citrus leaves, the feeding quantity of citrus psyllids decreased by 36.62% to 52.70%. Therefore, guava volatile oil could be regarded as a positive control with good effects. Indoor repellent tests showed that the repellent rate of PG against citrus psyllids within 24 hours was 85.49%-100%, which was sufficient to confirm the significant repellent effect of PG on citrus psyllids. Additionally, EOs from ER, ET and BF exhibited similar repellent effects on citrus psyllids within 24 hours as guava oil. Significance test results indicated that, except for the 100% concentration at 6 hours and 12.5%, there was no significant difference in the repellent efficiency under the same concentration and time conditions. Among all EOs, EO from ERcould maintain a high repellent rate even at low concentrations and exhibited long-term effects. ER, ET, and BF were all distributed in southern China, overlapping with the main citrus producing areas. This suggested that ER, ET and BF all had the potential to be used as plant-based pesticides for *D. citri* repellent.

Through GC-MS analysis, a total of 121 compounds were identified. Subsequently, high abundant and commercially available compounds were used for the test of repellent activity individually. Table 5 showed that, some compounds could significantly repel D. citri, while others had no significant repellent effect on psyllids, and even had a certain attractive effect. β-caryophyllene is one of the main chemical constituents of PG EO, and its effectiveness in repelling *D. citri* has been confirmed[38]. In this study, β-caryophyllene also exhibited good repellent activity, with a repellent rate of 85.00 ± 4.28% after 24 hours. The composition of the other three plant EOs was significantly different from that of PG, but they all had a repellent effect similar to that of PG EO. This might be closely related to *α*-pinene and eucalyptol. It was worth noting that eucalyptol showed significant repellent activity during testing, with a 24-hour repellent rate of 100%. Eucalyptol was not detected in PG EO, while the relative content in EOs of ER, ET, and BF was 5.91%, 6.87%, and 4.31%, respectively. This indicated that eucalyptol might be the major active constituent of the EOs extracted from these three plants.

In toxicity bioassay, the mortality rate of nymphs was higher than that of adults. This was because after soaking the leaves, the EOs formed an oil film on the surface of the plant leaves, hindering the feeding of insects. At the same time, EOs could clog the pores of insects and caused them to suffocate. Their preventive and control effectswere similar to those of mineral oil pesticides[39]. Compared to adults, nymphs had softer mouthparts and were more difficult to penetrate plant leaves covered with oil film for feeding. In addition, nymphs' tolerance to food shortage and respiratory restrictionwas significantly lower than that of adults. Therefore, using the EOs from the four natural plant in this study could repel adult insects with flight ability and eliminate nymphs with weaker activity levels, providing a theoretical basis for the development of new *D. citri* repellents and insecticides[40].

The docking results of the DcitOBP7 molecule showed that both the central and edge regions of DcitOBP7 had hydrophobic pocket-like cavities, which provided a possibility for the binding of various ligands to DcitOBP7. The results of molecular docking experiments revealed that the top three ligands with the lowest binding energies: *α*-pinene, β-caryophyllene, and limonene could all be embedded into the central hydrophobic pocket-like cavity of DcitOBP7[41]. In addition, eucalyptol, which had a stronger tendency to repellent *D. citri*, could be placed in the hydrophobic pockets at the center and edge of DcitOBP7, respectively, and there were few other ligands embedded at the docking sites located at the edge, which created conditions for eucalyptol to achieve diversified docking on DcitOBP7.

In comparison with the experimental results of molecular docking and compound repellent rate determinations, although the lowest binding energy of eucalyptol and DcitOBP7 docking was higher than that of the other four compounds, it had a separate docking site at the edge of DcitOBP7.

Additional, in behavioral experiments, eucalyptol showed stronger persistence compared to other compounds, and still had a significant effect on *D. citri* after 24 hours. Moreover, the compound had lower corrosiveness to plant leaves, and after 24 hours of use, the leaves of *Murraya paniculata* could still maintain a fresh green state. Eucalyptol itself had insecticidal activity and was used to kill insects. Research had shown that eucalyptol had an impact on *M Domestica* and *C Megacephala* has toxicity[42-44]. Therefore, eucalyptol had high potential as both a plant-based pesticide and a repellent. However, due to the toxicity of eucalyptol to mammals, attention should be paid to its safety issues in practical applications[45, 46]. Compared with eucalyptol,α- pinene had the lowest binding energy, and experimental results also indicated that within 12 hours α- pinene has a strong repellent effect. However, its repellent rate would significantly decrease after 24 hours. Consequently, this compound was not suitable for use as a repellent alone. If the sustained effectiveness can be enhanced by adding additives and slow-release agents, there is great potential in avoiding the application of *D. citri*.

In the previous study reported by *María et al.*[47], limonene showed a significant attractive effect on *D. citri*. In this study, we found that limonene exhibited a maximum attractive effect of 17.32% on *D. citri*, which was consistent with *María et al*.'s report. D-limonen also had an attraction effect of 11.27% on citrus psyllids. Therefore, limonene has the potential to be developed as a new *D. citri* attractive agents. In subsequent experiments, we will continue to conduct research on the effectiveness of attractive agents.

This study once again confirmed the repellent effect of PG OE on *D. citri.* PG is rich in various vitamins, such as vitamin C, and mineral elements, which can effectively promote the synthesis of nitric oxide in the human body, and has the effects of dilating blood vessels and lowering blood pressure[48, 49]. The high content of β-caryophyllene in PG leaves can also be used as a pest repellent and attractant in agricultural pest control.

As a widely planted and vigorous plant, ER has advantages such as rapid growth, abundant yield, and outstanding carbon sequestration capacity. In the southern China, especially in citrus-producing areas, ER has become an important forestry resource[50]. Therefore, using ER as raw material to developing natural plant repellent targeting *D. citri* will greatly enhance the economic value of ER, alleviate the pressure of *D. citri* prevention and control, and slow down the growth of *D. citri* resistance. In addition to providing plant EOs, planting ER around citrus orchards can establish repellent isolation zones, thereby blocking the flight of *D. citri*. This will also provide a basis for green prevention and control of *D. citri* in the field.

4. Materials and Methods

4.1. Plant and Insect Materials

4.1.1. Plant Materials

Twigs and leaves from four kinds of plants, PG, ER, ET and BF, were brought from local market. Plant samples were kept in the laboratory of Guangxi Academy of Speciality Crops.

4.1.2. Insect Materials

D. citri were raised in the Insect Laboratory of Guangxi Academy of Specialty Crops (110°18′51" E, 25°5′18" N), and 300 healthy *M. paniculata* plants were planted in a netted area within the greenhouse (25 ± 2 ℃, 70±10 % relative humidity, photoperiod of 16 hours light: 8 hours dark) as food source for them.

4.2. Extraction of the EOs

Plant EOs were extracted using the steam distillation method. Plant samples were ground into powder and subjected to steam distillation using a Clevenger-type apparatus for 2 hours. The collected oil samples were dried over anhydrous Na_2SO_4 and stored at 4 ℃ for further analysis.

4.3. Repellent Bioassay of EOs

25% acetone-aqueous solution was used to dilute Eos., The EOs were set with concentration gradients of 100%, 50%, 25% and 12.5%. Thirty *D. citri* adults were placed in plastic pipes (h=10 cm, d=1.5 cm) and a hunger treatment for 6 hours. Two healthy young shoots of *M. exotica* were cut and inserted into two plastic tubes that filled with water, one of the shoots was evenly applied EOs and the other was applied water. The two tubes with shoots were placed at opposite corners in a net cage (60 cm × 60 cm × 60 cm). The *D. citri* that have completed hunger treatment were released in the center of the cage, where there is an equal distance to the two tubes with shoots, each EOs and all its dilution gradients were tested. There were three independent replicates for each treatment. All the treated *D. citri* were maintained in an incubator (25±2 ℃, 70±10% RH with a 14:10 h L:D photoperiod) and the number of *D. citri* on different treatment shoots was checked after being treated for 2, 4, 6, 8, 10, 12 and 24 hours. The repellent efficiency was calculated using the following formula:

$$\text{Repellent Rate\%} = \frac{Nc - Ne}{Nc + Ne} \times 100\%$$

Note: Nc：the number of *Diaphorina citri* that chose contril check; Ne: the number of *D. citri* that chose essential oils.

4.4. Toxicity Bioassay

Toxicity bioassay of *D. citri* was performed using a leaf dip bioassay method. The EOs were diluted with a 25% acetone-water solution to different concentrations. For each EOs, *M. paniculata* leaves were immersed for 10 seconds in essential oils and their dilute solutions, and in 25% acetone-water solution (controls). The leaves were air-dried for 30 minutes before being placed individually in a plastic cup (h = 20 cm, d = 5 cm) . After the leaves had dried, thirty *D. citri* adults and nymphs were place on them. There were three independent replicates in each treatment. All the treated *D. citri* were maintained in the incubator (25 ± 2 ℃, 70 ± 10% RH with a 14 : 10 h L : D photoperiod) and the number of deaths in *D. citri* on different treatment leaves was checked after 24 hours of treatment.

4.5. Composition analysis of the EOs by GC-MS

The composition of EOs was analyzed by gas chromatography coupled to mass spectrometry (GC-MS) Agilent Model 8890 GC and a 7000D mass spectrometer (Agilent), equipped with a 30 m × 0.25 mm × 0.25 μm DB-5MS capillary column. Helium was used as the carrier gas with a linear velocity of 1.2 mL/min. The injector temperature was maintained at 250 °C. The oven temperature was programmed from 40 °C 3.5 min, increasing at 10 °C/min to 100°C, then at 7 °C/min to 180 °C, and finally at 25 °C/min to 280 °C, hold for 5 min. Mass spectra were recorded in electron impact (EI) ionisation mode at 70 eV. The quadrupole mass detector, ion source and transfer line temperatures were set, respectively, at 150, 230 and 280°C. The MS was operated in selected ion monitoring (SIM) mode for the identification of analyses. The chemical constituents were identified by comparing their mass spectra alongside the linear retention indices using those from the NIST20 database and the consulted/existing literature. Relative abundance percentages of individual compounds were quantified as the average peak area percentages, without using correction factors.

4.6. Repellent Bioassay of Compounds

Based on the results of section 4.5, the main compounds in EOs were selected and subjected to repellent bioassay. The bioassay method was the same as in section 4.3, but it did not involve a designed concentration gradient. β-caryophyllene, terpinene, β-pinene, linalool, eucalyptol, α-pinene, phellandrene, ocimene, D-limonene, γ-terpinene, o-cymene, cineole, 1,4-diethylbenzene, limonene, 3-carene, 1-phenylhexan-3-one and myrtol were purchased from Macklin Chemical Reagent Co., Ltd (Shanghai, China).

4.7. Molecular Modeling and Docking

The tertiary structure of DcitOBP7 was modeled using the AlphaFold2 software. The 3D structure of ligands was downloaded in the PubChem database (http://pubchem.ncbi.nlm.nih.gov/). Molecular docking was performed using AutoDock Vina 1.2.0 and visual analysis of molecular docking results was conducted using AutoDockTools-1.5.7.

4.8. Statistical Analysis

The t-test was used to analyze differences in the number of *D. citri* on shoots processed differently. One way ANOVA was used to analyze differences in repellent rates between different plant EOs and different compounds. Data are shown as mean values ± standard error of mean (SEM). p values < 0.05 were considered statistically significant. All statistical analysis was performed using SPSS version 22.0 software.

5. Conclusions

In conclusion, this study reports the repellent activity of EOs from four plants PG, ER, ET, and BF. In the EOs repellent experiment, the feeding selectivity of citrus psyllids treated with starvation was used as a criterion to evaluate the repellent efficiency. Based on this evaluation standard, all four tested plant EOs have significant repellent efficiency against citrus psyllid. Through molecular docking and compound repellent experiments, we have identified several compounds that are sensitive to citrus psyllids and have high repellent efficiency, just like β-caryophyllene, α-pinene and eucalyptol, which can provide a basis for the prevention and control of citrus psyllids.

Author Contributions: Conceptualization, Yi-jie Li, Tian-ao Liu, Bing-hai Lou and Hong-bo Jiang; Data curation, Yang Han; Formal analysis, Yi-jie Li and Tian-ao Liu; Funding acquisition, Yi-jie Li, Bing-hai Lou and Hong-bo Jiang; Investigation, Yi-jie Li and Tian-ao Liu; Methodology, Yi-jie Li, Tian-ao Liu and Hang Zhao; Project administration, Bing-hai Lou; Resources, Bing-hai Lou and Hong-bo Jiang; Software, Yi-jie Li, Tian-ao Liu and Yang Han; Supervision, Bing-hai Lou; Validation, Cui-yun Lei and Ya-qin Song; Visualization, Bing-hai Lou; Writing – original draft, Yi-jie Li, Tian-ao Liu and Bing-hai Lou; Writing – review & editing, Yi-jie Li, Tian-ao Liu, Hang Zhao and Bing-hai Lou. All authors have read and agreed to the published version of the manuscript.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data presented in this study are available in the article.

Conflicts of Interest: The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

Sample Availability: Not applicable.

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