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Supporting Information

Gas chromatography-mass spectrometry feature-based molecular networking and biological activities against *Solenopsis invicta* of *Citrus* peel essential oils from Vietnam

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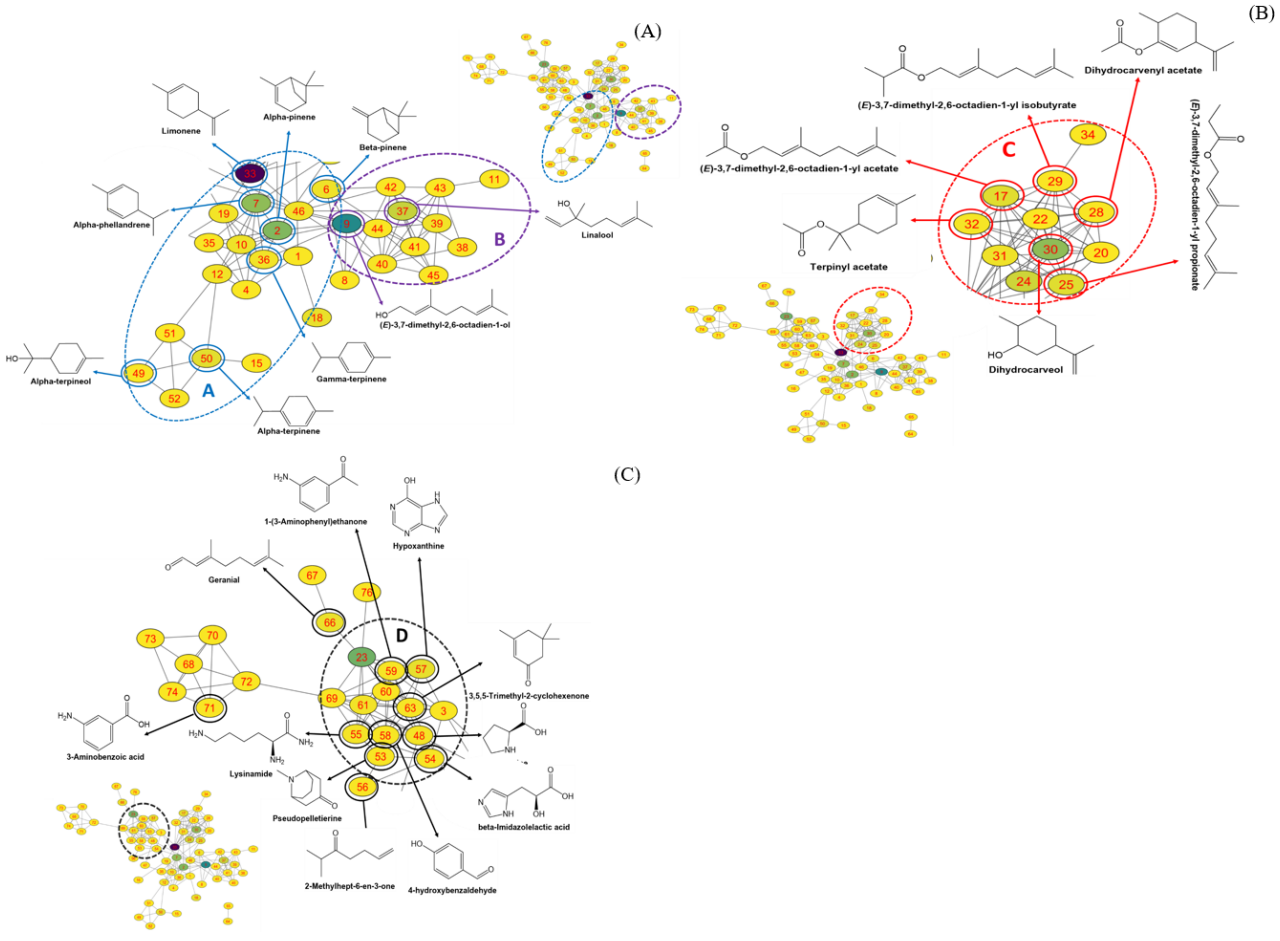

Fig. S1 Molecular networking of the essential oil from *C. reticulata* peel (A-C) generated using the GNPS web platform (https://gnps.ucsd.edu)

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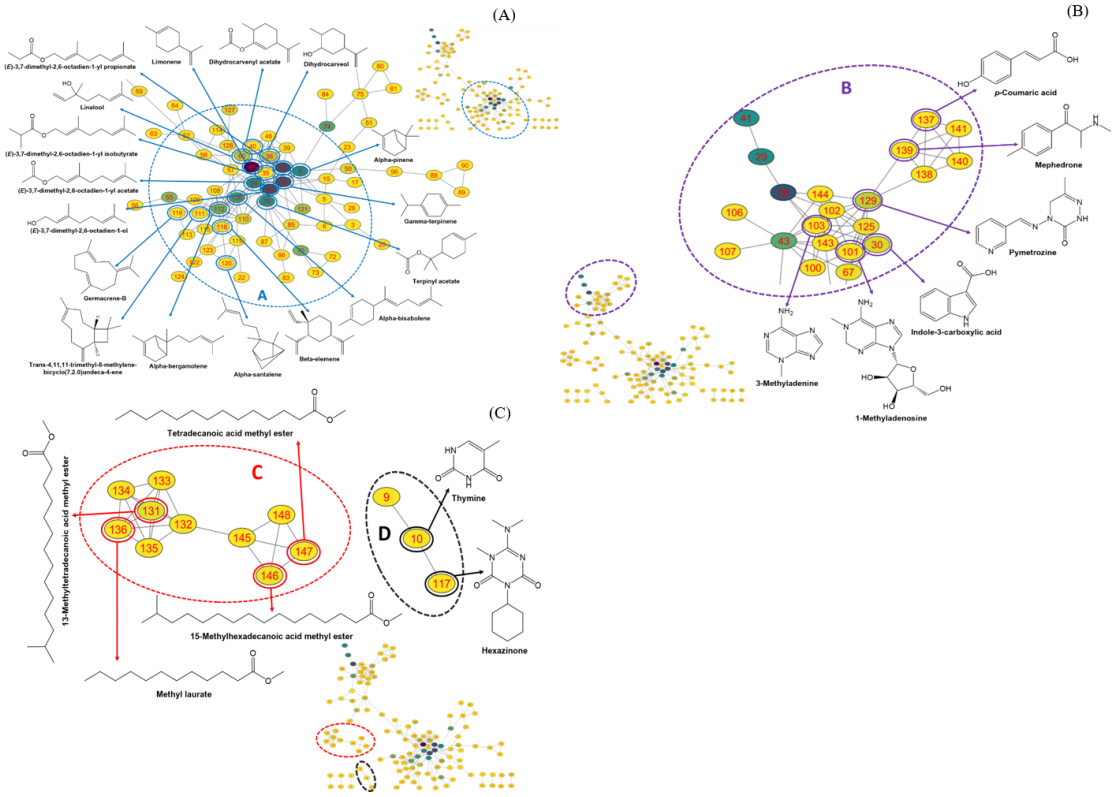

Fig. S2 Molecular networking of the essential oil from *C. limonia* peel (A–C) generated using the GNPS web platform (https://gnps.ucsd.edu)

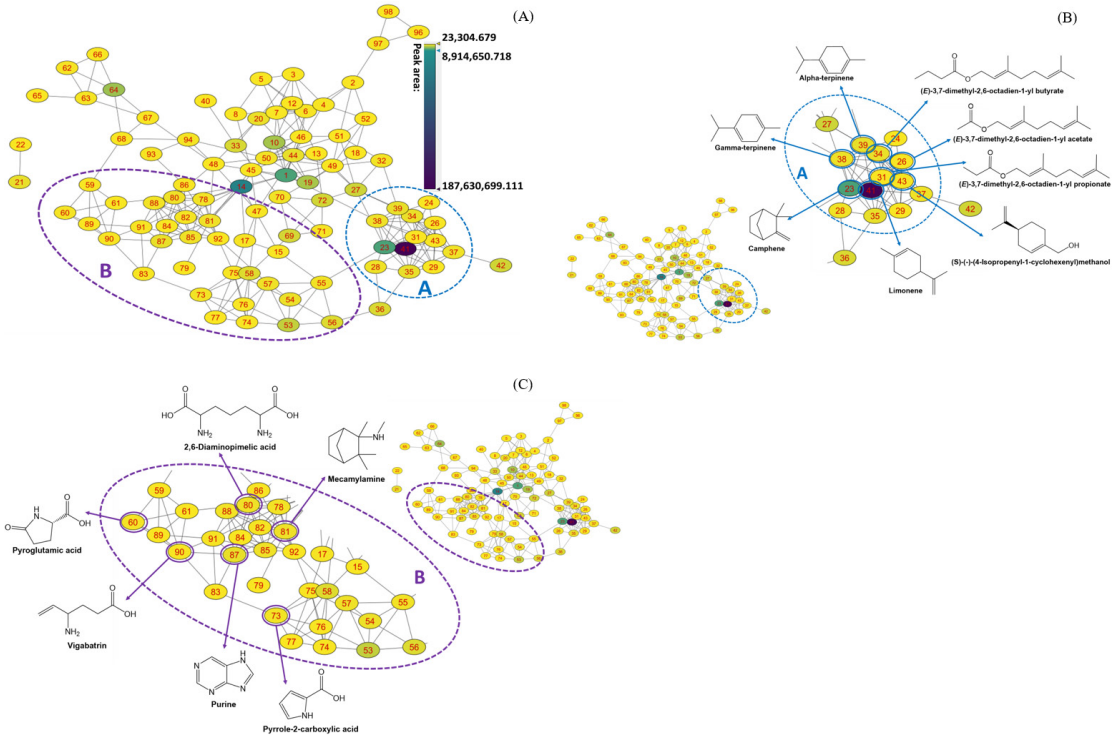

Fig. S3 Molecular networking of the essential oil from *C. sinensis* peel (A-C) generated using the GNPS web platform (https://gnps.ucsd.edu).

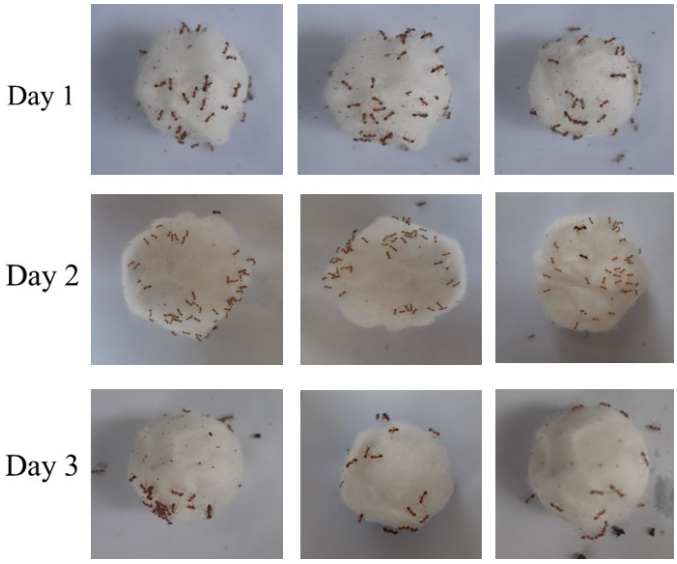

Fig. S4 The repellency against the *S. invicta* of cotton balls with only sugar water after 1–3 days of outdoor environmental exposure

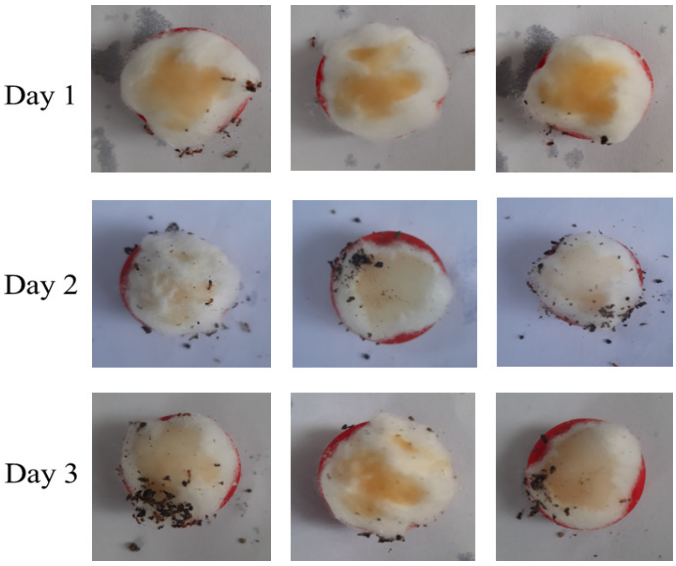

Fig. S5 The repellency against the *S. invicta* of cotton balls containing sugar water and an industrial ant repellent product after 1–3 days of outdoor environmental exposure

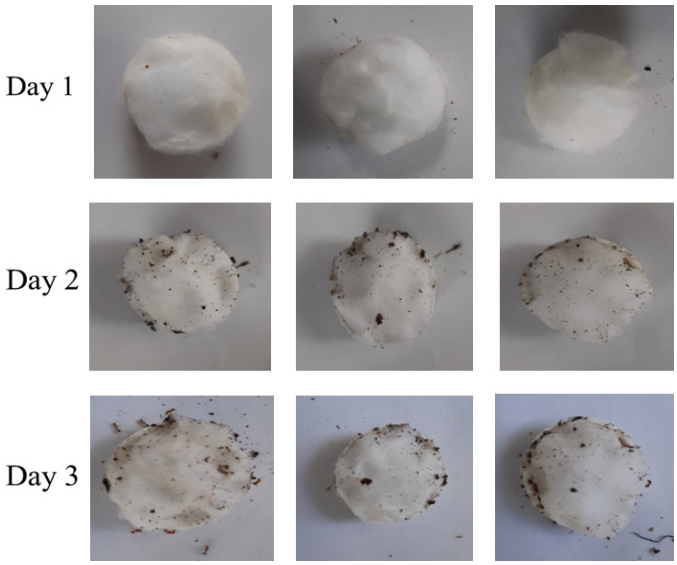

Fig. S6 The repellency against the *S. invicta* of cotton balls containing sugar water and the *C. reticulata* essential oil after 1–3 days of outdoor environmental exposure

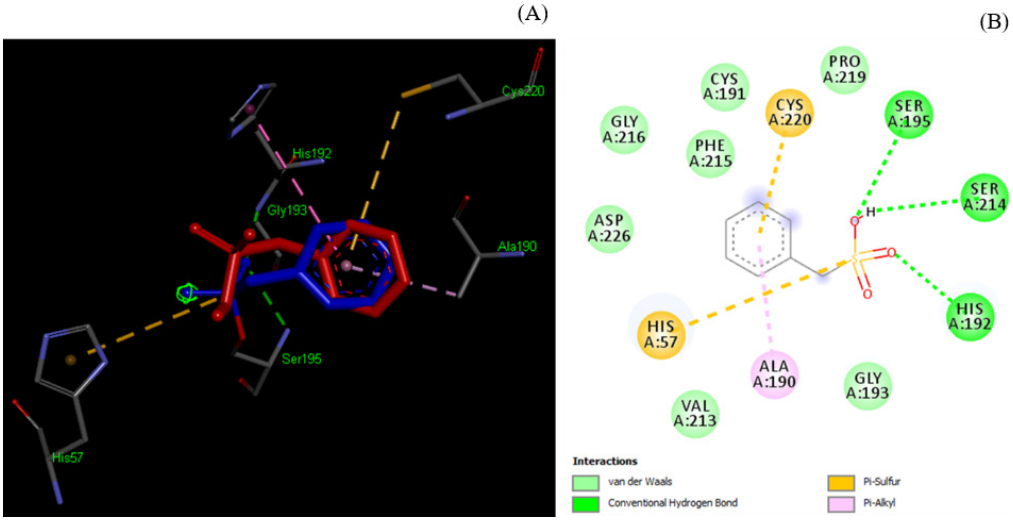

Fig. S7 (A) 3D molecular docking simulation of co-crystallized ligand (PMSF) with the fire ant chymotrypsin protein (PDB: 1EQ9), (Different colors (red and blue) in the figure signify different application methods including AutoDock tool (red) and previous reports from the Protein Data Bank (blue)); (B) 2D interaction diagram of 1EQ9 and PMSF using AutoDock too

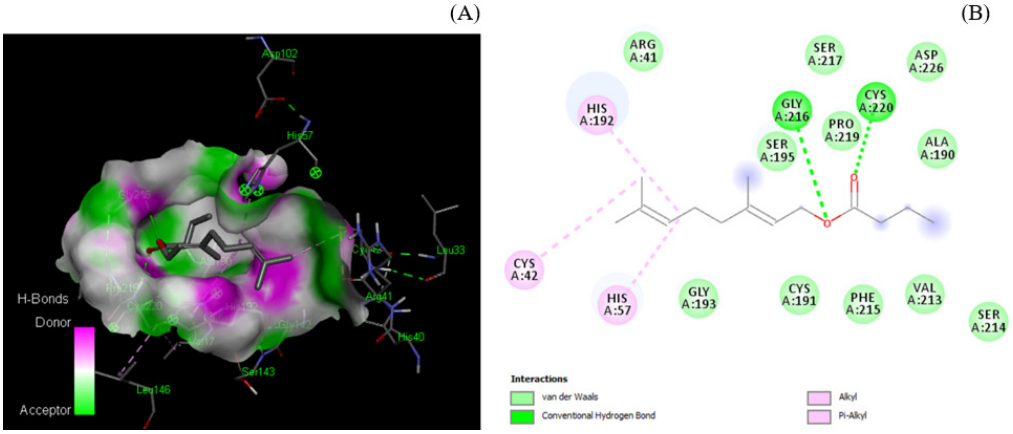

Fig. S8 (A) 3D molecular docking simulation (A) and 2D interaction diagram; (B) of (*E*)-3,7-dimethyl-2,6-octadien-1-yl butyrate with the fire ant chymotrypsin protein (PDB: 1EQ9) using AutoDock tool

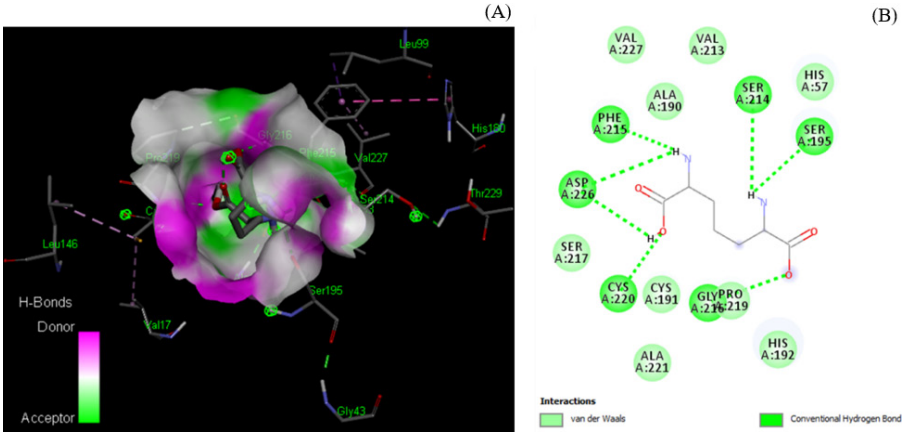

Fig. S9 (A) 3D molecular docking simulation (A) and 2D interaction diagram; (B) of 2,6-diaminopimelic acid with the fire ant chymotrypsin protein (PDB: 1EQ9) using AutoDock tool

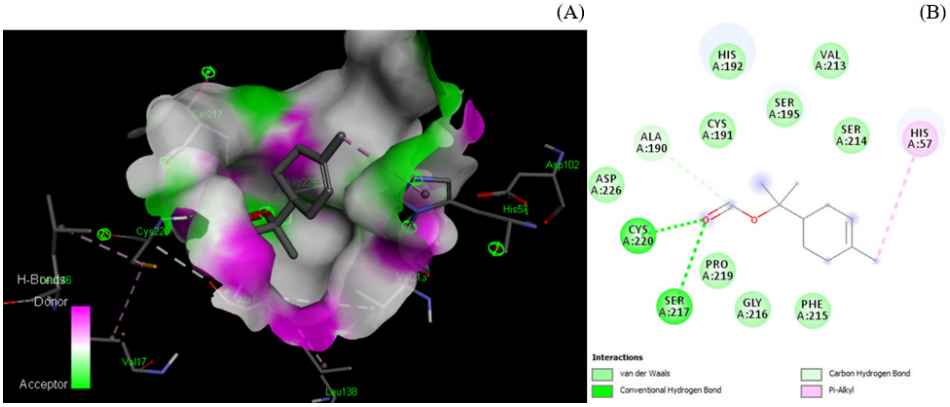

Fig. S10 (A) 3D molecular docking simulation (A) and 2D interaction diagram; (B) of *α*-terpinyl formate with the fire ant chymotrypsin protein (PDB: 1EQ9) using AutoDock tool

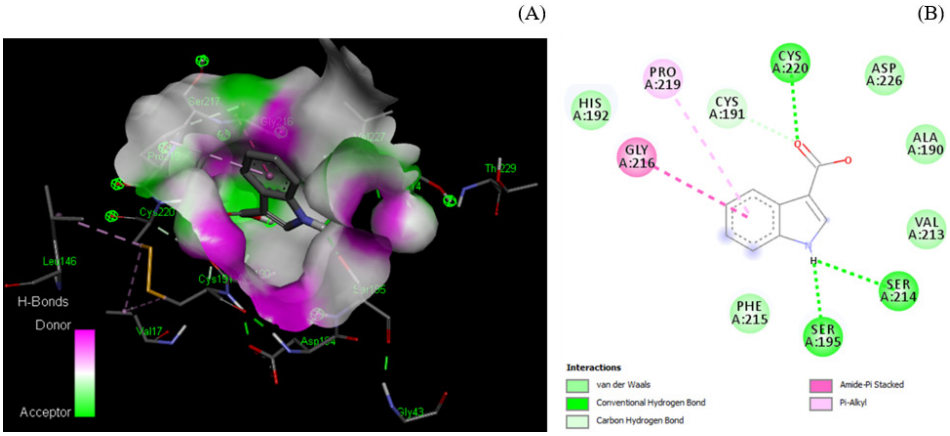

Fig. S11 (A) 3D molecular docking simulation (A) and 2D interaction diagram; (B) of indole-3-carboxylic acid with the fire ant chymotrypsin protein (PDB: 1EQ9) using AutoDock tool

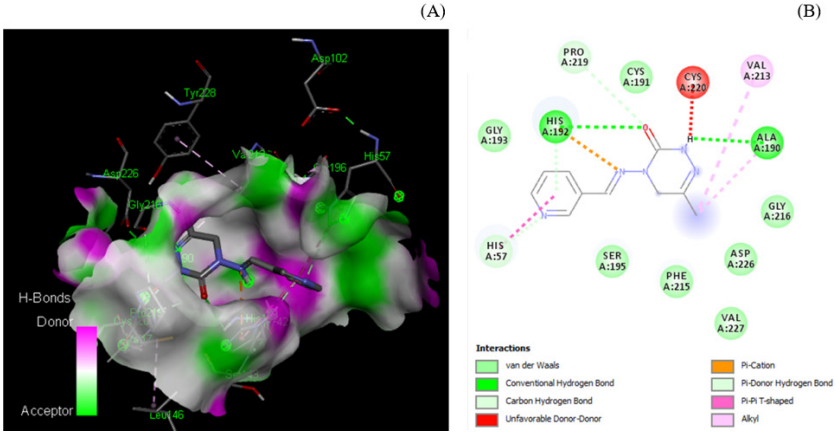

Fig S12 (A) 3D molecular docking simulation (A) and 2D interaction diagram; (B) of pymetrozine with the fire ant chymotrypsin protein (PDB: 1EQ9) using AutoDock tool

Table S1 Operating conditions for capillary GC-MS

	Citrus reticulata	Citrus limonia	Citrus sinensis
Parameter		GC	
Column	TB-SQC column (15 m × 0.25 mm × 0.25 µm)	TB-SQC column (15 m × 0.25 mm × 0.25 µm)	TB-SQC column (15 m × 0.25 mm × 0.25 µm)
Temp. program	• 50 °C kept 3 min • 50–240 °C at 7 °C/min and kept 5 min	• 50 °C kept 3 min • 50–110 °C at 6 °C/min and kept 2 min • 110–200 °C at 10 °C/min and kept 5 min • 200–280 °C at 5 °C/min and kept 9 min	• 50 °C kept 3 min • 50–200 °C at 4 °C/min and kept 1 min • 200–260 °C at 10 °C/min and kept 5 min
Carrier gas	Helium	Helium	Helium
Flow rate	1.2 mL/min	1.2 mL/min	1.2 mL/min
Injection volume	1.0 µL (Split mode 1:20)	1.0 µL (Split mode 1:20)	1.0 µL (Split mode 1:20)
Injector temp.	220 °C	220 °C	220 °C
Parameter		MS	
MS transfer line temp.	250 °C	250 °C	250 °C
Ionization mode	Electron ionization (positive)	Electron ionization (positive)	Electron ionization (positive)
Source temp.	220 °C	280 °C	220 °C

Table S2 Putative compounds of the essential oil from *C. reticulata* peel based on molecular networking and fragmentation analysis with the GC-MS spectra

Row retention time	Cluster index	Compound name	Data collector	MassDiff	MQScore	Fragmention mass mass	Peak area	Peak area (%)	SpectrumID	Kovat index
4.43	2	α-Pinene	SODA AROMATIC CO., LTD.	43.0914	0.902375	93.0336	6078960	2.05	CCMSLIB00005452833	1215.79
5.31	6	β-Pinene	TAKEUCHI T, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	15.075	0.809712	121.05	601421	0.2	CCMSLIB00005453263	1323.22
5.31	7	α-Phellandrene	YAMAMOTO M, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	43.1277	0.903476	92.9973	5383202	1.82	CCMSLIB00005453103	1324.05
5.6	9	(*E*)-3,7-dimethyl-2,6-octadien-1-ol	FUJISE Y, HAMAMATSU UNIV. SCHOOL OF MEDICINE	61.0711	0.916295	93.0649	22489117	7.6	CCMSLIB00005451556	1359.03
6.36	17	(E)-3,7-dimethyl-2,6-octadien-1-yl acetate	FUJISE Y, HAMAMATSU UNIV. SCHOOL OF MEDICINE	145.993	0.864183	50.1527	1327223	0.45	CCMSLIB00005451557	1456.80
6.39	25	(E)-3,7-dimethyl-2,6-octadien-1-yl propionate	FUJISE Y, HAMAMATSU UNIV. SCHOOL OF MEDICINE	153.108	0.869357	57.0536	1342437	0.45	CCMSLIB00005451558	1458.92
6.4	30	Dihydrocarveol (trans-equatorial)	YAMAMOTO M, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	48.1311	0.769338	106.0049	5376094	1.82	CCMSLIB00005453096	1458.92
6.41	33	Limonene	MASS SPECTROSCOPY SOC. OF JAPAN (MSSJ)	69.0795	0.931883	67.0455	2.27E+08	76.84	CCMSLIB00005448278	1459.78
9.02	37	Linalool	TAKEUCHI T, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	61.1405	0.895439	92.9955	2626593	0.89	CCMSLIB00005453257	1802.91
9.22	48	Proline	Ryffel F, Eric He	46.0671	0.836167	70.0039	382451.9	0.13	CCMSLIB00000081786	1831.77
10.82	49	α-terpineol	YAMAMOTO M, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	101.146	0.728596	52.9898	141431.1	0.05	CCMSLIB00005451143	2078.74
11.89	63	3,5,5-Trimethyl-2-cyclohexenone	HASHIMOTO K, KYOTO COLLEGE OF PHARMACY	55.9504	0.76283	82.1536	290432.2	0.1	CCMSLIB00005451815	2257.07
12.24	64	Perillaldehyde	TAKEUCHI T, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	83.1617	0.82213	66.9423	148169.8	0.05	CCMSLIB00005453262	2318.84
12.49	66	Geranial	TAKEUCHI T, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	83.0397	0.799464	69.0803	818098.7	0.28	CCMSLIB00005453241	2364.60
5.6	8	Myrtenal	SODA AROMATIC CO., LTD.	44.0426	0.706447	106.0614	74476.62	0.03	CCMSLIB00005454344	N/D
6.39	28	Dihydrocarvenyl acetate (equatorial)	YAMAMOTO M, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	67.0781	0.802669	129.0679	129828.3	0.04	CCMSLIB00005451771	N/D

Table S2 Continued

Row retention time	Cluster index	Compound name	Data collector	MassDiff	MQScore	Fragmention mass mass	Peak area	Peak area (%)	SpectrumID	Kovat index
6.4	29	(*E*)-3,7-dimethyl-2,6-octadien-1-yl isobutyrate	FUJISE Y, HAMAMATSU UNIV. SCHOOL OF MEDICINE	114.176	0.844364	110.0019	91464.36	0.03	CCMSLIB00005451559	N/D
6.4	32	Terpinyl acetate	TAKEUCHI T, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	78.0569	0.7804	118.0891	320896.7	0.11	CCMSLIB00005453270	N/D
7.61	36	γ-terpinene	YAMAMOTO M, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	43.1103	0.804822	93.0147	491868.6	0.17	CCMSLIB00005453102	N/D
10.84	50	α-terpinene	YAMAMOTO M, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	15.1135	0.861155	121.0115	1214597	0.41	CCMSLIB00005453104	N/D
11.05	53	Pseudopelletierine	Massbank	57.8282	0.740086	96.1718	158887.9	0.05	CCMSLIB00000220046	N/D
11.06	54	β-imidazolelactic acid	Massbank	74.9022	0.767175	82.0978	223514.1	0.08	CCMSLIB00000219000	N/D
11.07	55	Lysinamide	Massbank	61.9012	0.891341	84.0988	391072	0.13	CCMSLIB00000219136	N/D
11.08	56	2-Methylhept-6-en-3-one	MASS SPECTROSCOPY SOC. OF JAPAN (MSSJ)	55.0879	0.784073	71.0161	193427.5	0.07	CCMSLIB00005447927	N/D
11.82	57	Hypoxanthine	Massbank	67.9209	0.871261	69.0791	1008839	0.34	CCMSLIB00000218666	N/D
11.88	58	4-Hydroxybenzaldehyde	JGI:196764	27.8374	0.838881	95.2067	163254.2	0.06	CCMSLIB00006690081	N/D
11.88	59	1-(3-Aminophenyl) ethanone	Massbank	41.9021	0.83581	94.1739	227217.2	0.08	CCMSLIB00000841498	N/D
14.24	71	3-Aminobenzoic acid	Data deposited by negarg	27.9807	0.714604	92.0643	107095.1	0.04	CCMSLIB00003135628	N/D
4.43	1	N/I				119.0099	149495.92	0.05		
4.43	3	N/I				106.0614	108329.76	0.04		
4.44	4	N/I				137.1541	74619.12	0.03		
5.61	10	N/I				68.0157	654281.73	0.22		
5.61	11	N/I				61.9497	31884.54	0.01		
5.92	12	N/I				92.9978	474019.22	0.16		
6.33	15	N/I				113.9999	61563.97	0.02		
6.35	16	N/I				88.0814	84401.88	0.03		
6.36	18	N/I				119.9975	897433.78	0.30		
6.36	19	N/I				89.9392	409031.73	0.14		
6.36	20	N/I				126.0652	35648.48	0.01		
6.37	22	N/I				139.2552	31836.29	0.01		
6.37	23	N/I				65.9346	6912577.95	2.34		
6.38	24	N/I				134.9553	2242007.04	0.76		

Table S2 Continued

Row retention time	Cluster index	Compound name	Data collector	MassDiff	MQScore	Fragmention mass mass	Peak area	Peak area (%)	SpectrumID	Kovat index
6.40	31	N/I				75.8816	620136.46	0.21		
6.41	34	N/I				72.9341	53517.94	0.02		
7.60	35	N/I				136.0264	162249.23	0.05		
9.04	38	N/I				71.9734	125966.21	0.04		
9.04	39	N/I				68.0259	537436.86	0.18		
9.05	40	N/I				95.1966	256972.15	0.09		
9.06	41	N/I				139.2333	85733.25	0.03		
9.09	42	N/I				108.2479	142730.14	0.05		
9.10	43	N/I				50.9747	64514.37	0.02		
9.16	44	N/I				98.0783	100474.46	0.03		
9.18	45	N/I				97.2225	95757.57	0.03		
9.21	46	N/I				90.9516	504636.19	0.17		
9.21	47	N/I				134.0033	73701.70	0.02		
10.92	51	N/I				59.694	28053.40	0.01		
10.92	52	N/I				80.142	227787.72	0.08		
11.88	60	N/I				53.0267	139147.38	0.05		
11.89	61	N/I				93.0027	493680.07	0.17		
12.25	65	N/I				105.1282	45568.15	0.02		
12.55	67	N/I				137.1887	93382.70	0.03		
14.23	68	N/I				119.0527	129228.64	0.04		
14.24	69	N/I				68.1289	123240.32	0.04		
14.24	70	N/I				78.971	155411.10	0.05		
14.25	72	N/I				80.0881	85586.19	0.03		
14.26	73	N/I				175.4013	38640.10	0.01		
14.28	74	N/I				106.0614	60325.44	0.02		
16.06	76	N/I				161.0662	136575.63	0.05		

N/I: Not identified; N/D: Not determined

Table S3 Putative compounds of the essential oil from *C. limonia* peel based on molecular networking and fragmentation analysis with the GC-MS spectra

Row retention time	Cluster index	Compound name	Data collector	MassDiff	MQScore	Fragmention mass mass	Peak area	Peak area (%)	SpectrumID	Kovat index
4.38	4	α-Pinene	SODA AROMATIC CO., LTD.	43.1089	0.91651	93.0161	16261826	3.07	CCMSLIB00005452833	1210.39
4.56	7	Ethylcyclohexane	IIDA Y, DAISHIMA S, FAC. OF ENGINEERING, SEIKEI UNIV.	30.0778	0.732694	82.0472	282830.3	0.05	CCMSLIB00005457637	1232.04
4.57	8	Isopropylcyclohexane	IIDA Y, DAISHIMA S, FAC. OF ENGINEERING, SEIKEI UNIV.	1.1732	0.812753	124.9678	61280.39	0.01	CCMSLIB00005457643	1232.46
5.29	16	(E)-3,7-dimethyl-2,6-octadien-1-ol	FUJISE Y, HAMAMATSU UNIV. SCHOOL OF MEDICINE	61.0793	0.92287	93.0567	66335905	12.52	CCMSLIB00005451556	1321.58
5.72	24	(E)-3,7-dimethyl-2,6-octadien-1-yl acetate	FUJISE Y, HAMAMATSU UNIV. SCHOOL OF MEDICINE	103.078	0.889168	93.0681	14821142	2.8	CCMSLIB00005451557	1321.58
6.45	35	(E)-3,7-dimethyl-2,6-octadien-1-yl isobutyrate	FUJISE Y, HAMAMATSU UNIV. SCHOOL OF MEDICINE	151.262	0.824722	72.9161	63929.34	0.01	CCMSLIB00005451559	1374.04
6.49	38	(E)-3,7-dimethyl-2,6-octadien-1-yl propionate	FUJISE Y, HAMAMATSU UNIV. SCHOOL OF MEDICINE	119.129	0.843797	91.0328	67065203	12.66	CCMSLIB00005451558	1374.04
6.53	45	Limonene	MASS SPECTROSCOPY SOC. OF JAPAN (MSSJ)	69.0784	0.927077	67.0466	1.52E+08	28.61	CCMSLIB00005448278	1464.9
7.25	52	γ-Terpinene	YAMAMOTO M, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	43.0288	0.925687	93.0962	64115506	12.1	CCMSLIB00005453102	1470.00
9.18	60	Linalool	TAKEUCHI T, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	83.1727	0.863799	70.9633	2219475	0.42	CCMSLIB00005453257	1826.32
10.17	68	Citronellal	TAKEUCHI T, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	56.0221	0.76345	98.1139	223595.5	0.04	CCMSLIB00005453243	1975.32
10.61	70	Terpinen-4-ol	"SDBSWeb : https://sdbs.db.aist.go.jp (National Institute of Advanced Industrial Science and Technology, 2019)"	42.9984	0.780448	111.0016	3665172	0.69	CCMSLIB00005689698	2045.67
11.1	74	Anethole	SODA AROMATIC CO., LTD.	0.001999	0.849636	148.091	4723001	0.89	CCMSLIB00005454817	2124.53
11.1	79	α-Terpinyl formate	HISAHIRO HAGIWARA, CHEMICAL RESEARCH INSTITUTE OF NON-AQUEOUSSOLUTIONS TOHOKU UNIV.	61.1146	0.868877	121.0164	8283367	1.56	CCMSLIB00005452213	2125.08
11.1	80	Propyleneglycol	Tsujimoto Y, Tsugawa H, Bamba T, Fukusaki E, engineering department, Osaka Univ.	41.7849	0.789277	117.8373	145644.1	0.03	CCMSLIB00005458725	2125.08

Table S3 Continued

Row retention time	Cluster index	Compound name	Data collector	MassDiff	MQScore	Fragmention mass mass	Peak area	Peak area (%)	SpectrumID	Kovat index
11.16	85	Adamantane	"SDBSWeb : https://sdbs.db.aist.go.jp (National Institute of Advanced Industrial Science and Technology, 2019)"	55.9636	0.735785	80.0364	760680.3	0.14	CCMSLIB00005681810	2133.36
11.16	86	α-Terpineol	YAMAMOTO M, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	100.115	0.770705	54.021	213485.7	0.04	CCMSLIB00005451143	2134.46
12.47	91	Citronellol	TAKEUCHI T, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	87.1278	0.796596	69.0232	1876886	0.35	CCMSLIB00005453244	2360.92
12.53	95	3,5,5-Trimethyl-2-cyclohexen-1-one	KOGA M, UNIV. OF OCCUPATIONAL AND ENVIRONMENTAL HEALTH	56.0763	0.804056	82.0277	1309622	0.25	CCMSLIB00005447013	2372.66
4.39	5	(E,Z)-2,6-dimethyl-2,4,6-Octatriene	"SDBSWeb : https://sdbs.db.aist.go.jp (National Institute of Advanced Industrial Science and Technology, 2019)"	31.9285	0.777699	104.0715	71650.75	0.01	CCMSLIB00005676814	N/D
5.18	10	Thymine	Massbank	54.1079	0.75782	72.8921	83111.14	0.02	CCMSLIB00000220713	N/D
5.31	21	α-Methoxy-Benzeneacetic acid	"SDBSWeb : https://sdbs.db.aist.go.jp (National Institute of Advanced Industrial Science and Technology, 2019)"	45.9178	0.719067	120.0822	88183.36	0.02	CCMSLIB00005682876	N/D
6.4	30	Indole-3-carboxylic acid	Massbank	44.2174	0.799582	117.8376	975153.7	0.18	CCMSLIB00000205716	N/D
6.49	36	Dihydrocarveol (trans-equatorial)	YAMAMOTO M, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	58.0702	0.862666	96.0658	2093844	0.4	CCMSLIB00005453096	N/D
6.52	40	Dihydrocarvenyl acetate (axial)	YAMAMOTO M, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	48.055	0.815868	148.091	19719.98	0	CCMSLIB00005451769	N/D
6.53	44	2,5-Dimethylphenol	KOGA M, UNIV. OF OCCUPATIONAL AND ENVIRONMENTAL HEALTH	22.9953	0.797539	145.0683	22515.92	0	CCMSLIB00005446160	N/D
8.29	55	α-Terpinene	YAMAMOTO M, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	0.070602	0.907028	136.0544	3372085	0.64	CCMSLIB00005453104	N/D
9.17	58	3-Methyl-6-(1-methylethyl)-2-cyclogexen-1-ol	FUJISE Y, HAMAMATSU UNIV. SCHOOL OF MEDICINE	70.0986	0.737017	84.0374	282777.7	0.05	CCMSLIB00005451561	N/D
9.18	59	Ectoine	Massbank	74.9579	0.754656	68.0421	479481.8	0.09	CCMSLIB00000218175	N/D

Table S3 Continued

Row retention time	Cluster index	Compound name	Data collector	MassDiff	MQScore	Fragmention mass mass	Peak area	Peak area (%)	SpectrumID	Kovat index
9.19	61	L-Valine	Massbank	45.9651	0.725158	72.0349	143116.7	0.03	CCMSLIB00000221029	N/D
11.1	75	Bisoprolol	Ed Esquenazi	211.224	0.73479	116.0128	375959	0.07	CCMSLIB00000004929	N/D
11.1	76	Hippuric acid	ReSpect	76.1085	0.8093	104.0715	92316.15	0.02	CCMSLIB00000219041	N/D
11.1	77	Aminolevulinic acid	HMDB	80.0671	0.718519	51.9989	131706.6	0.02	CCMSLIB00000426207	N/D
13.58	101	1-Methyladenosine	HMDB	131.063	0.925233	150.2053	153996.9	0.03	CCMSLIB00005445367	N/D
13.59	103	3-Methyladenine	JGI:192883	0.433197	0.980069	149.6438	114317.2	0.02	CCMSLIB00006686200	N/D
15.42	110	β-caryophyllene	SASAKI S, TOYOHASHI UNIV. OF TECH.	71.1905	0.782636	132.9975	1284215	0.24	CCMSLIB00005448787	N/D
15.42	111	Trans-4,11,11-trimethyl-8-methylene-bicyclo(7.2.0) undeca-4-ene	SASAKI S, TOYOHASHI UNIV. OF TECH.	123.157	0.760475	81.031	332856.4	0.06	CCMSLIB00005448793	N/D
15.86	112	α-bergamotene	SASAKI S, TOYOHASHI UNIV. OF TECH.	85.1803	0.845406	119.0077	4099579	0.77	CCMSLIB00005448760	N/D
15.88	114	Humulene	SASAKI S, TOYOHASHI UNIV. OF TECH.	95.0612	0.714365	109.1268	214830.6	0.04	CCMSLIB00005448778	N/D
15.9	116	Germacrene-B	EGUCHI S, FAC. OF SCIENCE, HIROSHIMA UNIV.	81.0752	0.722742	123.1128	77508.92	0.01	CCMSLIB00005456224	N/D
17.28	117	Hexazinone	Massbank	168	0.814408	85.0004	50561.29	0.01	CCMSLIB00000221043	N/D
17.3	118	β-Elemene	EGUCHI S, FAC. OF SCIENCE, HIROSHIMA UNIV.	96.0473	0.744749	108.1407	694274.8	0.13	CCMSLIB00005456232	N/D
17.32	120	α-Santalene	SASAKI S, TOYOHASHI UNIV. OF TECH.	108.089	0.709781	96.0987	82140.23	0.02	CCMSLIB00005448782	N/D
17.32	121	d-Cadinene	SASAKI S, TOYOHASHI UNIV. OF TECH.	43.1356	0.813878	161.0524	2567103	0.48	CCMSLIB00005448763	N/D
17.4	126	α-Bisabolene	SASAKI S, TOYOHASHI UNIV. OF TECH.	111.192	0.830081	92.9964	5352300	1.01	CCMSLIB00005448790	N/D
17.46	129	Pymetrozine	Massbank	113.097	0.853644	105.0072	1780677	0.34	CCMSLIB00000212718	N/D
18.08	131	13-Methyltetradecanoic acid methyl ester	KOGA M, UNIV. OF OCCUPATIONAL AND ENVIRONMENTAL HEALTH	169.292	0.777992	86.9479	593117.1	0.11	CCMSLIB00005448129	N/D
18.16	136	Methyl laurate	YAMAMOTO M, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	0.112	0.815648	214.081	39879.39	0.01	CCMSLIB00005456322	N/D
18.77	137	p-Coumaric acid	JGI:209277	0.038742	0.831826	147.0053	72127.01	0.01	CCMSLIB00006702594	N/D
18.81	139	Mephedrone	Massbank	33.0547	0.816776	145.0683	66520.56	0.01	CCMSLIB00000211688	N/D
21.59	146	15-Methylhexadecanoic acid methyl ester	KOGA M, UNIV. OF OCCUPATIONAL AND ENVIRONMENTAL HEALTH	210.335	0.740565	73.9372	318518.7	0.06	CCMSLIB00005448141	N/D

Table S3 Continued

Row retention time	Cluster index	Compound name	Data collector	MassDiff	MQScore	Fragmention mass mass	Peak area	Peak area (%)	SpectrumID	Kovat index
3.69	1	N/I				97.1087	32002.84	0.01		
3.73	2	N/I				81.0308	168000.86	0.03		
4.37	3	N/I				62.0101	26343.87	0.00		
4.40	6	N/I				65.9834	102700.76	0.02		
5.18	9	N/I				85.05	92463.53	0.02		
5.25	12	N/I				74.9731	65570.15	0.01		
5.29	15	N/I				106.021	317156.58	0.06		
5.30	17	N/I				61.9392	90763.34	0.02		
5.31	20	N/I				63.9774	104680.91	0.02		
5.31	22	N/I				109.1215	100851.61	0.02		
5.34	23	N/I				90.0189	77070.39	0.01		
5.74	26	N/I				106.0573	67842.05	0.01		
6.36	27	N/I				114.0281	100670.25	0.02		
6.39	28	N/I				119.062	68385461.98	12.90		
6.40	29	N/I				115.0103	7796732.90	1.47		
6.41	32	N/I				113.1058	64265.25	0.01		
6.45	34	N/I				90.0069	384080.18	0.07		
6.51	39	N/I				138.1388	364182.60	0.07		
6.52	41	N/I				102.9833	6276391.17	1.18		
6.53	43	N/I				65.9564	4413779.48	0.83		
6.53	46	N/I				147.0053	46234.33	0.01		
6.91	50	N/I				92.9968	954805.42	0.18		
7.25	51	N/I				104.0057	528649.68	0.10		
8.29	56	N/I				87.0478	77601.27	0.01		
9.17	57	N/I				109.0691	216471.44	0.04		
9.25	62	N/I				82.13	286767.50	0.05		
9.27	63	N/I				54.021	66504.41	0.01		
9.30	64	N/I				98.1139	86126.12	0.02		
9.72	65	N/I				118.9212	146933.27	0.03		
9.73	66	N/I				137.1923	243812.92	0.05		
9.75	67	N/I				108.0945	475915.68	0.09		
10.18	69	N/I				109.0989	528711.16	0.10		
10.64	72	N/I				86.9991	83956.92	0.02		
10.66	73	N/I				155.1383	122686.67	0.02		
11.10	81	N/I				73.931	39853.70	0.01		
11.12	82	N/I				76.073	55255.28	0.01		
11.12	83	N/I				61.9939	45749.37	0.01		

Table S3 Continued

Row retention time	Cluster index	Compound name	Data collector	MassDiff	MQScore	Fragmention mass mass	Peak area	Peak area (%)	SpectrumID	Kovat index
11.15	84	N/I				134.0519	189118.47	0.04		
11.23	87	N/I				140.2321	87666.61	0.02		
11.73	88	N/I				84.0529	126341.07	0.02		
11.73	89	N/I				124.9832	50557.68	0.01		
11.74	90	N/I				112.0736	65866.78	0.01		
12.47	92	N/I				109.0562	621753.08	0.12		
12.47	93	N/I				156.1311	108125.90	0.02		
12.53	96	N/I				108.0506	192193.91	0.04		
12.98	97	N/I				78.9823	178651.09	0.03		
13.57	100	N/I				134.1441	209511.34	0.04		
13.58	102	N/I				135.1096	230183.37	0.04		
14.92	106	N/I				161.0022	316279.17	0.06		
14.93	107	N/I				123.1818	66277.03	0.01		
15.40	108	N/I				134.0166	448774.42	0.08		
15.41	109	N/I				176.0202	66904.17	0.01		
15.87	113	N/I				117.8373	53832.51	0.01		
15.89	115	N/I				102.9792	47598.39	0.01		
17.31	119	N/I				77.9994	182189.48	0.03		
17.32	122	N/I				163.2441	57546.52	0.01		
17.37	123	N/I				65.9834	60024.65	0.01		
17.37	124	N/I				189.9409	56473.11	0.01		
17.40	125	N/I				68.0818	577364.30	0.11		
17.41	127	N/I				123.0746	1182528.00	0.22		
17.41	128	N/I				122.0794	615706.05	0.12		
18.09	132	N/I				100.9753	73174.34	0.01		
18.09	133	N/I				115.0167	82890.75	0.02		
18.10	134	N/I				93.0208	125504.15	0.02		
18.11	135	N/I				121.0156	66479.42	0.01		
18.79	138	N/I				79.0358	157696.02	0.03		
18.82	140	N/I				131.0866	62495.07	0.01		
18.84	141	N/I				159.0854	81963.33	0.02		
20.85	143	N/I				108.4346	109654.37	0.02		
20.86	144	N/I				69.0755	112767.31	0.02		
21.57	145	N/I				129.0449	40505.39	0.01		
21.61	148	N/I				242.0416	12157.38	0.00		

N/I: Not identified; N/D: Not determined

Table S4 Putative compounds of the essential oil from *C. sinensis* peel based on molecular networking and fragmentation analysis with the GC-MS spectra

Row retention time	Cluster index	Compound name	Data collector	MassDiff	MQScore	Fragmention mass mass	Peak area	Peak area (%)	SpectrumID	Kovat index
4.54	1	α-Pinene	SODA AROMATIC CO., LTD.	43.1317	0.902236	92.9933	7174597	2.63	CCMSLIB00005452833	1229.53
5.56	10	α-phellandrene	YAMAMOTO M, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	43.0589	0.937496	93.0661	3495650	1.28	CCMSLIB00005453103	1354.03
5.58	13	2-Ethyl-5-methylphenol	MASS SPECTROSCOPY SOC. OF JAPAN (MSSJ)	14.1479	0.802369	121.9411	23304.68	0.01	CCMSLIB00005449922	1356.53
5.96	14	(E)-3,7-dimethyl-2,6-octadien-1-ol	FUJISE Y, HAMAMATSU UNIV. SCHOOL OF MEDICINE	61.1368	0.930942	92.9992	27892845	10.23	CCMSLIB00005451556	1403.24
6.39	20	Isopropylbenzene	IIDA Y, DAISHIMA S, FAC. OF ENGINEERING, SEIKEI UNIV.	41.1046	0.778615	78.9894	690987.9	0.25	CCMSLIB00005457713	1456.80
6.98	26	(E)-3,7-dimethyl-2,6-octadien-1-yl acetate	FUJISE Y, HAMAMATSU UNIV. SCHOOL OF MEDICINE	49.1677	0.84868	146.9783	119241.7	0.04	CCMSLIB00005451557	1530.99
6.94	23	Camphene	TAKEUCHI T, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	30.2059	0.792476	105.9191	7200198	2.64	CCMSLIB00005453236	1531.42
6.98	27	3,4-Dimethylbenzyl alcohol	"SDBSWeb : https://sdbs.db.aist.go.jp (National Institute of Advanced Industrial Science and Technology, 2019)"	1.1232	0.801634	134.8768	1866558	0.68	CCMSLIB00005678817	1531.42
6.99	32	(+)-3-Carene	"SDBSWeb : https://sdbs.db.aist.go.jp (National Institute of Advanced Industrial Science and Technology, 2019)"	50.0014	0.736402	85.9986	305903.9	0.11	CCMSLIB00005673771	1533.16
7.02	38	γ-terpinene	YAMAMOTO M, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	17.9784	0.807068	118.0216	520045.2	0.19	CCMSLIB00005453102	1537.06
7.03	39	α-terpinene	"SDBSWeb : https://sdbs.db.aist.go.jp (National Institute of Advanced Industrial Science and Technology, 2019)"	65.9478	0.791946	70.0522	756822.8	0.28	CCMSLIB00005674959	1538.37
7.03	41	Limonene	MASS SPECTROSCOPY SOC. OF JAPAN (MSSJ)	42.0599	0.943206	94.0651	1.88E+08	68.83	CCMSLIB00005448278	1538.37
8.57	47	Benzene	IIDA Y, DAISHIMA S, FAC. OF ENGINEERING, SEIKEI UNIV.	0.978806	0.86336	79.0258	167171.1	0.06	CCMSLIB00005457699	1741.03
10.59	57	Linalool	TAKEUCHI T, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	44.9102	0.757235	109.2258	518052.5	0.19	CCMSLIB00005453257	2012.56

Table S4 Continued

Row retention time	Cluster index	Compound name	Data collector	MassDiff	MQScore	Fragmention mass mass	Peak area	Peak area (%)	SpectrumID	Kovat index
6.45	22	5-Aminovaleric acid	JGI:192817	0.9814	0.794116	99.0946	55268.8	0.02	CCMSLIB00006686134	N/D
6.99	31	(E)-3,7-dimethyl-2,6-octadien-1-yl propionate	FUJISE Y, HAMAMATSU UNIV. SCHOOL OF MEDICINE	96.1354	0.810135	114.0266	75782.28	0.03	CCMSLIB00005451558	N/D
7.01	34	(E)-3,7-dimethyl-2,6-octadien-1-yl butyrate	FUJISE Y, HAMAMATSU UNIV. SCHOOL OF MEDICINE	90.1421	0.742284	134.0359	641734.5	0.24	CCMSLIB00005451560	N/D
7.03	43	(S)-(-)-(4-isopropenyl-1-cyclohexenyl)methanol	"SDBSWeb : https://sdbs.db.aist.go.jp (National Institute of Advanced Industrial Science and Technology, 2019)"	51.0874	0.744503	100.9126	143446.1	0.05	CCMSLIB00005674072	N/D
12.42	60	Pyroglutamic acid	Massbank	60.9159	0.766331	69.0841	235004.7	0.09	CCMSLIB00000220168	N/D
13.37	64	Anethole	SODA AROMATIC CO., LTD.	0.125198	0.852416	147.9638	3902746	1.43	CCMSLIB00005454817	N/D
14.14	73	Pyrrole-2-carboxylic acid	JGI:215417	17.9302	0.750911	94.1091	293040.3	0.11	CCMSLIB00006708734	N/D
15.36	80	2,6-Diaminopimelic acid	DB	108.021	0.777489	82.1757	530827.3	0.19	CCMSLIB00005442102	N/D
15.36	81	Mecamylamine	Massbank	86.8401	0.824582	81.1599	513576.7	0.19	CCMSLIB00000219325	N/D
15.42	87	Purine	Massbank	26.8935	0.765704	94.1065	750494.4	0.28	CCMSLIB00000220025	N/D
15.44	90	Vigabatrin	MoNA	59.201	0.859266	70.885	136470.8	0.05	CCMSLIB00000568855	N/D
15.93	93	Perillaldehyde	TAKEUCHI T, DEP. CHEMISTRY, FAC. SCIENCE, NARA WOMEN'S UNIV.	15.1332	0.736806	134.9708	196957.3	0.07	CCMSLIB00005453262	N/D
4.54	2	N/I				106.0362	114416.34	0.04		
4.54	3	N/I				63.0781	45330.06	0.02		
4.55	4	N/I				51.0913	142136.55	0.05		
4.56	5	N/I				121.9411	87706.29	0.03		
4.56	6	N/I				77.9684	193443.41	0.07		
4.61	7	N/I				137.1418	106815.60	0.04		
4.68	8	N/I				95.1722	63543.34	0.02		
5.56	12	N/I				95.0972	36993.39	0.01		
5.97	15	N/I				106.0362	101773.26	0.04		
5.98	17	N/I				51.9858	185189.27	0.07		
6.35	18	N/I				134.2273	32310.93	0.01		
6.38	19	N/I				92.9933	3357838.69	1.23		
6.95	24	N/I				125.9947	59377.10	0.02		
6.98	28	N/I				83.0316	474761.47	0.17		
6.98	29	N/I				147.5778	90320.93	0.03		

Table S4 Continued

Row retention time	Cluster index		Compound name	Data collector	MassDiff	MQScore	Fragmention mass mass	Peak area	Peak area (%)	SpectrumID	Kovat index
7.00	33	N/I					56.9914	1450102.23	0.53		
7.02	35	N/I					75.8543	777455.37	0.29		
7.02	36	N/I					63.9811	1353289.71	0.50		
7.02	37	N/I					89.9441	483510.69	0.18		
7.03	40	N/I					98.0456	58604.81	0.02		
7.03	42	N/I					50.1564	1755694.14	0.64		
8.56	44	N/I					92.9596	751670.30	0.28		
8.56	45	N/I					80.1595	90704.06	0.03		
8.56	46	N/I					134.1459	61890.30	0.02		
9.34	48	N/I					91.9721	58858.30	0.02		
9.34	49	N/I					137.1418	44834.55	0.02		
9.35	50	N/I					120.9882	401293.03	0.15		
9.36	51	N/I					94.157	38733.18	0.01		
9.38	52	N/I					121.9411	33600.90	0.01		
10.38	53	N/I					120.9823	2313309.06	0.85		
10.39	54	N/I					82.1633	599523.16	0.22		
10.41	55	N/I					108.4465	389276.94	0.14		
10.58	56	N/I					66.9675	1189809.71	0.44		
10.59	58	N/I					56.0293	1157788.04	0.42		
12.41	59	N/I					136.0433	88041.13	0.03		
12.43	61	N/I					97.1706	51788.47	0.02		
13.35	62	N/I					78.9434	180507.71	0.07		
13.36	63	N/I					77.9464	213614.80	0.08		
13.37	65	N/I					75.9965	42073.27	0.02		
13.39	66	N/I					63.054	89337.46	0.03		
13.40	67	N/I					132.18	96609.75	0.04		
13.41	68	N/I					118.1295	112056.49	0.04		
13.69	69	N/I					92.9963	1627807.12	0.60		
13.69	70	N/I					79.0084	377167.60	0.14		
13.70	71	N/I					139.1397	183588.14	0.07		
13.78	72	N/I					136.0011	1683740.64	0.62		
14.14	74	N/I					112.1396	139489.52	0.05		
14.18	75	N/I					70.08	148943.10	0.05		

Table S4 Continued

Row retention time	Cluster index	Compound name	Data collector	MassDiff	MQScore	Fragmention mass mass	Peak area	Peak area (%)	SpectrumID	Kovat index
14.18	76	N/I				110.0913	87192.34	0.03		
14.24	77	N/I				71.9208	37495.79	0.01		
15.36	78	N/I				83.009	335158.23	0.12		
15.36	79	N/I				51.0384	62470.13	0.02		
15.37	82	N/I				107.045	231324.82	0.08		
15.39	83	N/I				96.0695	138339.17	0.05		
15.40	84	N/I				97.1706	74735.23	0.03		
15.42	85	N/I				109.118	531064.83	0.19		
15.42	86	N/I				137.1425	155247.96	0.06		
15.42	88	N/I				108.3753	353783.28	0.13		
15.43	89	N/I				138.129	65420.45	0.02		
15.44	91	N/I				123.0943	182041.29	0.07		
15.47	92	N/I				124.0019	59760.17	0.02		
15.94	94	N/I				106.9742	215731.47	0.08		
22.64	96	N/I				90.9682	66023.81	0.02		
22.68	97	N/I				161.0078	231601.07	0.08		
22.70	98	N/I				133.0512	61257.47	0.02		

N/I: Not identified; N/D: Not determined

Table S5 The percentage of peak area of limonene from three essential oils and their LC_{50} values at 1 and 2 h of exposure periods

Sample name	Limonene (% peak area)	LC_{50} - 1 h (mg/mL)	LC_{50} - 2 h (mg/mL)
C. reticulata	76.84	47.74±13.99	ND
C. limonia	28.61	ND	142.87±28.73
C. sinensis	68.83	ND	104.86±14.79
Limonene	-	12.69±2.68	ND

ND: Not determined

Table S6 The repellency percentage of cotton balls containing sugar water and an industrial ant repellent product. Data were observed with the naked eye during 1–3 days of outdoor environmental exposure

Experiment	Day 1		Day 2		Day 3	
	2 h	8 h	26 h	32 h	50 h	56 h
1st	43.3	60	100	100	100	100
2nd	53.3	60	100	100	100	100
3rd	46.6	56.6	100	100	100	100
Average	47.3	58.9	100	100	100	100

Table S7 The repellency percentage of cotton balls containing sugar water and the *C. reticulata* essential oil. Data were observed with the naked eye during 1–3 days of outdoor environmental exposure

Experiment	Day 1		Day 2		Day 3	
	2 h	8 h	26 h	32 h	50 h	56 h
1st	63.3	100	100	100	100	100
2nd	63.3	100	100	100	100	100
3rd	66.6	100	100	100	100	100
Average	64.4	100	100	100	100	100