Compositional Analysis and Potent Insecticidal Activity of Supercritical CO₂ Fluid Extracts of Alcea nudi flora L. Leaves

Nazira K. Khidyrova, Saida M. Turaeva, Malohat J. Rakhmatova, Khayrulla M. Bobakulov, Shamansur S. Sagdullaev, Rano P. Zakirova, Khamid U. Khodjaniyazov,* and Kohei Torikai*

ABSTRACT: To mitigate potentially severe food shortages due to the exponential growth of the global population, it is of paramount importance to improve the yield and quality of globally harvested food crops. As pest control contributes to both these aspects, the development of safe and effective pesticides is one of the main strategies pursued in this direction in the context of agricultural chemistry. During our investigation of natural pesticides, a supercritical CO₂ fluid extract of Alcea nudi flora L. was found to exert extremely potent insecticidal activity against aphids (Macrosiphum euphorbiae) and cowpea seed beetles (Callosobruchus maculatus) with LC₅₀ values of 0.03 mg/mL (24 h exposure, contact method). The facts that their insecticidal activity is in the most potent class among the essential oils known to date, and that the extract did not show any toxicity toward beneficial insects such as ladybugs (Coccinella magnifica) and European honeybees (Apis mellifera Linnaeus), indicate that this extract could be a good, natural, and safe new pesticide candidate. A compositional analysis of this extract was carried out using GC/MS.

INTRODUCTION

Due to the improved quality of life, and especially medical care, the global population has reached 7.8 billion and continues to grow.¹ One study estimates that it will grow to 10 and 11 billion in 2050 and 2100, respectively.² To manage the associated potentially severe shortages of food, improving crop yield and quality are of paramount global importance. There are many strategies to increase the yield and quality of crops, among which pest control to decrease crop loss is one of the most essential. Thus, to date, hundreds of artificial pesticides have been developed and made commercially available.³ Chemical pesticides are powerful, scalable, and inexpensive, but they have historically caused health problems in humans, although recently developed ones are often much less harmful.⁴

On the other hand, various plant-derived materials, such as essential oils, plant extracts, and fermentation products (e.g., vinegars) have also been used as natural pesticides and medicines;⁵ these are believed to be superior to their chemical counterparts on account of their greater safety for both humans and the environment. Among these, supercritical CO₂ extracts (SCE) of plants have attracted great attention, as CO₂ can achieve the extraction of unique substances that conventional solvents (e.g., organic solvents and water) cannot afford.⁷ Furthermore, unlike organic solvents, CO₂ is easily

------------------------------------------------------------------------------------------------------------------

© 2022 The Authors. Published by American Chemical Society
19892

http://pubs.acs.org/journal/acsodf

Article

ACS Omega 2022, 7, 19892−19897

https://doi.org/10.1021/acsomega.2c01688

ACS Omega 2022, 7, 19892−19897
maculatus). Furthermore, we examined its insecticidal activity against beneficial insects such as ladybugs (Coccinella magnifica) and European honeybees (Apis mellifera Linnaeus). Identification of molecular components in the SCE was accomplished using gas chromatography/mass spectrometry (GC/MS) and comparison of the MS characteristics and retention indices (RIs) with data in databases. As far as we know, this work is the first to demonstrate the insecticidal activity of extracts of A. nudiflora.

## MATERIALS AND METHODS

### Plant Materials

Leaves of A. nudiflora L. [identified by an experienced botanist, Dr. Alijan M. Nigmatulayev, and kept at the herbarium of the S. Yu. Yunusov Institute of the Chemistry of Plant Substances, Academy of Sciences of the Republic of Uzbekistan (administrative number: 1338)] were collected in the vicinity of Chortak village, Namangan region, Uzbekistan (August 2015), dried in the shade, and crushed (4–6 mm) before the extraction.

### Extraction

The crushed dry leaves of A. nudiflora L. (1 kg) were extracted with fluidic supercritical CO₂ at 40–50 °C under 28–30 MPa for 100 min to give 3.00 g of SCE. Additional samples of dry leaves (10.0 g each) were extracted three times with EtOH (96%, 80 mL, 60 mL, and 60 mL, respectively) under sonication (90 min) or by infusion (24 h) at room temperature. Evaporation at 40 °C afforded an ESE (1.73 g, dark green oily mass) and an EIE (1.44 g, dark green oily mass), respectively.

### Further Separation of the CO₂ Extract

The SCE (2.00 g) was further separated into three fractions using column chromatography on silica gel. Fraction (Fr) 1 (450 mg, yellow oil after the removal of solvents), Fr 2 (275 mg, colorless amorphous mass), and Fr 3 (720 mg, colorless oil) were eluted using hexane–CHCl₃ = 1:1, 1:5, and 1:10, respectively.

### Insects

Dozens of C. maculatus were collected from the institutional storage house and kept separately in a cage with tap-water-washed and dried mung beans (Vigna radiata) to prevent infestation. Adult C. maculatus were reared at 25 ± 1 °C and 55 ± 5% relative humidity with a photoperiod of 12 h. Three-day-old C. maculatus adults were selected and used in assays. The laboratory population of M. euphorbiae was kept on seedlings of the potato variety Sante at 22 ± 2 °C with a photoperiod of 16 h. Adult C. magnifica and A. mellifera Linnaeus were collected at a farm in Tashkent, Uzbekistan and kept in separate cylindrical glass bottles closed with a mosquito net.

### Insecticidal Assays

Each extract (1.00 g) and a drop of Tween 80 or OP-10 were diluted with distilled H₂O to give 100 mL of stock solution (10 mg/mL). These stock solutions were further diluted with H₂O to prepare samples with lower concentrations. The various sample solutions (1.00 mL) were added to Petri dishes (diameter: 9 cm) in which a filter paper had been placed. After 10 min, insects (M. euphorbiae and C. maculatus, 10 adults each), together with their feed plants [potatoes (Solanum tuberosum L.) and mung beans (Vigna radiata), respectively] were placed on each dish, and the insect mortality was determined 5, 10, 30, and 60 min and 24 h after exposure. After obtaining three independent replicates at each concentration, the insecticidal activity of each extract was determined as percentage mortality of the insects.

### Identification of the Components in the CO₂ Extract

A solution of the SCE (1.0 mg) in hexane (1 mL) was injected (1.0 μL) into a GC/MS (5975C inert MSD/7890A GC system, Agilent Technologies, USA, 2009), equipped with a quartz capillary column: HP-INNOWax, 30 m (length) × 0.25 mm (diameter) × 0.25 μm (thickness), Agilent Technologies. The separation and analysis were carried out under the following conditions: injector temperature: 220 °C; temperature mode: 50 °C (1 min) → 200 °C (heating rate: 4 °C/min followed by a constant temperature of 200 °C for 6 min) → 250 °C (heating rate: 15 °C/min followed by a constant temperature of 250 °C for 25 min); recorded EI-MS m/z range: 10–550; carrier gas: helium; flow rate: 1.1 mL/min. The components were identified by comparison of the observed MS characteristics with MS libraries, that is, the Wiley Registry of Mass Spectral Data, 9th Ed., and the National Institute of Standards and Technology (USA) Mass Spectral Library (2011). The Kratz RIs of compounds, which were determined relative to a mixture of n-alkanes (C₈–C₂₀), were also compared with a previous literature report.

## RESULTS AND DISCUSSION

### Insecticidal Activity of the EtOH and CO₂ Extracts

We first examined the insecticidal activity of the EtOH and CO₂ extracts (10 mg/mL each) against aphids, M. euphorbiae (Figure 1), with negative and positive control experiments using H₂O and the known synthetic pyrethroid cypermethrin (0.10 mg/mL).

![Figure 1. Insecticidal activity of the ESE, EIE, and SCE of A. nudiflora against M. euphorbiae (5 min exposure).](https://example.com/image1.png)

After 5 min of exposure, only SCE exhibited potent insecticidal activity, whereas both EtOH extracts, that is, ESE and EIE, were inactive. These results were surprising given that we have detected some insecticidal compounds (e.g., β-amyrin, β-sitosterol, phytol, and lupeol) in our preliminary study of SCE and EIE. We concluded that neither ESE nor EIE were active due to the (very) low concentration of insecticidal compounds. On the other hand, these results were also very interesting as they indicate that supercritical CO₂ is able to extract more/other insecticidal compounds than those mentioned above and/or different unique insecticidal plant substances that EtOH cannot.

### Insecticidal Activity of the CO₂ Extract against M. euphorbiae and C. maculatus

As preliminary insecticidal activity was only detected for SCE, we further investigated the dose responses of M. euphorbiae and C. maculatus to SCE. Treatment of M. euphorbiae with systematically diluted SCE solutions (0.05–5 mg/mL) revealed that the SCE of A. nudiflora L. exhibits potent pesticidal activity (Figure 2). At the highest concentration of 5.0 mg/mL, 87% of the aphids died within 10 min (blue bar), with the death rate reaching 100% after 30 min of exposure (red bar). Even at concentrations of 0.10–1.0 mg/mL, the potent insecticidal activity was maintained especially after 60 min (green bar) and
24 h (purple bar) of exposure, with mortality rates of >80%. At 0.050 mg/mL, the activity dropped to <50%. The lethal concentration (LC$_{50}$) value after 24 h was calculated to be 0.03 mg/mL.

The SCE of A. nudi flora also exhibited potent insecticidal activity against the larger pest C. maculatus (Figure 3). The activity was almost identical to that against M. euphorbiae, with its LC$_{50}$ value for C. maculatus after 24 h also being found to be 0.03 mg/mL. Ikbal and Pavela have reported in their excellent review article that potent insecticidal essential oils show LC$_{50}$ values on the order of 10$^{-1}$ to 10$^{-2}$ μL/mL ($\approx$ mg/mL for solids) against aphids in contact applications.$^{11}$ Only 9% (7 examples) of the 77 examples in the literature showed an LC$_{50}$ of <0.1 μL/mL, and only 4% (3 examples) were more potent than the SCE of A. nudi flora. This comparison clearly shows that among materials of plant origin, the SCE of A. nudi flora exhibits excellent potency against aphids. Moreover, it is noteworthy that the SCE of A. nudi flora is completely harmless to the beneficial insects C. magnifica and A. mellifera L. as well as to human skin, even at a concentration of 10 mg/mL (data not shown). These results indicate that the SCE of A. nudi flora is a good candidate as a novel natural insecticide, although the scalability of the extraction should further be addressed.

### Insecticidal Activity of Further-Fractionated SCE

To investigate the active species, we further separated the SCE using column chromatography on silica gel to give three fractions. The obtained Frs. 1 (eluted with hexane/CHCl$_3$ = 1/1), 2 (hexane/CHCl$_3$ = 1/5), and 3 (hexane/CHCl$_3$ = 1/10) were subsequently subjected to insecticidal assays and the mortality (%) was calculated for insects exposed to the fractions for 24 h. Frs. 1 and 2 exhibited potent insecticidal activity against both M. euphorbiae (Figure 4) and C. maculatus (Figure 5), while Fr. 3 did not. The LC$_{50}$ values of Fr. 1 against M. euphorbiae and C. maculatus were 0.09 and 0.08 mg/mL, while those of Fr. 2 were 0.1 and 0.08 mg/mL, respectively. Since these LC$_{50}$ values are almost identical to those of the parent SCE mixture, these results indicate that less-polar organic compounds extracted by supercritical CO$_2$ fluid are responsible for the insecticidal activity.

### Components in the SCE Detected by GC/MS and a Comparison of Their Insecticidal Activity with Literature Precedents

Finally, we analyzed the chemical components of the SCE of A. nudi flora using GC/MS. Each component was identified via comparison of its Kratz RI with literature values, as well as its EI-MS fragmentation pattern. As a result, 32 compounds were identified (Table 1). Among these, thymol is known to be pesticidal against many pests,
Table 1. Composition of SCE of *A. nudiflora* Analyzed by GC/MS

| No | Compound name               | Structure | Retention time (min) | Kanze retention index (experimental) | Kanze retention index (reported) |
|----|-----------------------------|----------|----------------------|--------------------------------------|---------------------------------|
| 1  | 1-acetylcylobane            | ![1-acetylcylobane](image) | 5.40                 | 1126                                 | 1116 |
| 2  | n-butanol                   | ![n-butanol](image) | 5.89                 | 1136                                 | 1139 |
| 3  | heptan-2-one                | ![heptan-2-one](image) | 6.57                 | 1177                                 | 1182 |
| 4  | n-2-phenyl                  | ![n-2-phenyl](image) | 6.68                 | 1181                                 | 1185 |
| 5  | 3-methyl-butan-2-one        | ![3-methyl-butan-2-one](image) | 6.99                 | 1195                                 | 1202 |
| 6  | 2-octadecanone              | ![2-octadecanone](image) | 7.84                 | 1226                                 | 1232 |
| 7  | pentan-1-ol                 | ![pentan-1-ol](image) | 8.05                 | 1240                                 | 1247 |
| 8  | octanal                     | ![octanal](image) | 9.39                 | 1280                                 | 1287 |
| 9  | 7-phenyl                     | ![7-phenyl](image) | 10.35                | 1313                                 | 1321 |
| 10 | 6-methyl-5-heptene-2-one    | ![6-methyl-5-heptene-2-one](image) | 10.78                | 1328                                 | 1336 |
| 11 | nonanal                     | ![nonanal](image) | 12.44                | 1386                                 | 1391 |
| 12 | 3,5-octadecadiol            | ![3,5-octadecadiol](image) | 12.79                | 1398                                 | 1400 |
| 13 | (3S)-7-octenal              | ![3S)-7-octenal](image) | 13.45                | 1420                                 | 1429 |
| 14 | acetic acid                 | ![acetic acid](image) | 14.37                | 1450                                 | 1446 |
| 15 | methyloctanoate[37]         | ![methyloctanoate](image) | 15.00                | 1471                                 | 1482 |
| 16 | L-methionine (trans-methionine) [38,39] | ![L-methionine (trans-methionine) [38,39]](image) | 15.17                | 1477                                 | 1465 |
| 17 | L-2-hydroxybutanoate        | ![L-2-hydroxybutanoate](image) | 16.03                | 1505                                 | 1523 |
| 18 | trans-2-methyl-4hydroxymethanone[40] | ![trans-2-methyl-4hydroxymethanone](image) | 16.56                | 1585                                 | 1578 |
| 19 | 5,5-dimethyl-2-(3H)-furanone | ![5,5-dimethyl-2-(3H)-furanone](image) | 17.30                | 1593                                 | 1590 |
| 20 | pulegone                    | ![pulegone](image) | 19.69                | 1627                                 | 1654 |
| 21 | (-)-eucismenthol            | ![(-)-eucismenthol](image) | 20.50                | 1654                                 | 1646 |
| 22 | (2E,4S)-2,4-nonadecenal     | ![2E,4S)-2,4-nonadecenal](image) | 21.12                | 1685                                 | 1696 |
| 23 | (S)-octahydroxyoctalinol    | ![23](image) | 22.22                | 1712                                 | 1727 |
| 24 | citral[44]                  | ![citral[44]](image) | 22.33                | 1716                                 | 1733 |
| 25 | n-pentadecanoic acid        | ![n-pentadecanoic acid](image) | 22.74                | 1730                                 | 1734 |
| 26 | cis,cis-deca-2,6-dimetal    | ![cis,cis-deca-2,6-dimetal](image) | 23.29                | 1749                                 | 1756 |
| 27 | trans,trans-deca-2,6-dimetal | ![trans,trans-deca-2,6-dimetal](image) | 24.51                | 1790                                 | 1808 |
| 28 | n-2-hexanoic acid           | ![n-2-hexanoic acid](image) | 25.79                | 1834                                 | 1843 |
| 29 | gerany acetone              | ![gerany acetone](image) | 25.88                | 1850                                 | 1854 |
| 30 | n-octadecanolic acid        | ![n-octadecanolic acid](image) | 32.09                | 2045                                 | 2057 |
| 31 | hexahydrofarnesyl acetone   | ![hexahydrofarnesyl acetone](image) | 34.49                | 2124                                 | 2124 |
| 32 | thymol[45,46]               | ![thymol[45,46]](image) | 35.81                | 2180                                 | 2164 |
such as tobacco cutworms (*Spodoptera litura* Fab), bed bugs (*Cimex lectularius* L), houseflies (*Musca domestica*), red spider mites (*Tetranychus urticae*), Western corn rootworms (*Diabrotica virgifera*), and click beetles (*Agriotes obscurus* L). Pulegone is known to be toxic against *S. litura* Fab., maize weevils (*Sitophilus zeamais*), *M. domestica*, *D. virgifera* and vine mealybugs (*Planococcus ficus*). Menthone acts pesticidally against *C. lectularius* L, *C. lectularius* L, *S. zeamais*, *M. domestica*, and *D. virgifera*, while citral is pesticidal against *M. domestica*, and *D. virgifera*. Therefore, the main active insecticidal compounds in the SCE of *A. nudi flora* are most likely menthone, thymol, pulegone, and citral which differ from those in the EIE and ESE (β-amyrin, β-sitosterol, phytol, and lupeol), albeit that the presence of novel, structurally unknown natural products and/or synergistic effects with some minor components cannot be ruled out at this point. The good insecticidal selectivity of the SCE of *A. nudi flora* against beneficial/pest insects might be derived from the presence of thymol and pulegone, given that they are safe for European honeybees, which has recently been reported.

### CONCLUSIONS

In summary, *A. nudi flora* L. leaves have been extracted with supercritical CO$_2$ under fluidic conditions and been subjected to insecticidal assays for the first time. The supercritical CO$_2$ extract (SCE) of *A. nudi flora* exhibited potent insecticidal activity against aphids (*M. euphorbiaceae*) and cowpea seed beetles (*C. maculatus*) with LC$_{50}$ values of 0.03 mg/mL (24 h exposure, contact method). It is worth noting that the EtOH extract of *A. nudi flora* is not pesticidal and that the SCE is harmless to beneficial insects such as ladybugs (*C. magnifica*) and European honeybees (*A. mellifera Linnaeus*) at the tested concentrations (<10 mg/mL). These findings indicate that the SCE of *A. nudi flora* exhibits promising potential as an excellent lead for a safe pesticide of natural origin. Further investigations into the development of practical methods to enable the large-scale production of *A. nudi flora* SCE and the application of supercritical CO$_2$ extraction to various other plants native to Uzbekistan are currently in progress in our laboratory.

### AUTHOR INFORMATION

**Corresponding Authors**

Khamid U. Khodjaniyazov — A. S. Sadikov Institute of the Bioorganic Chemistry, Academy of Sciences of the Republic of Uzbekistan, Tashkent 100125, Uzbekistan; Faculty of Chemistry, National University of Uzbekistan named after Mirzo Ulugbek, Tashkent 100174, Uzbekistan; Email: hamidkhodjaniyazov@yandex.ru

Kohei Torikai — Faculty of Chemistry, National University of Uzbekistan named after Mirzo Ulugbek, Tashkent 100174, Uzbekistan; Department of Chemistry, Faculty of Science, Kyushu University, Fukuoka 819-0395, Japan; orcid.org/0000-0002-9928-4300; Email: torikai@chem.kyushu- univ.jp

**Authors**

Nazira K. Khidyrova — S. Yu. Yunusov Institute of the Chemistry of Plant Substances, Academy of Sciences of the Republic of Uzbekistan, Tashkent 100170, Uzbekistan

Saida M. Turaeva — S. Yu. Yunusov Institute of the Chemistry of Plant Substances, Academy of Sciences of the Republic of Uzbekistan, Tashkent 100170, Uzbekistan

Malohat J. Rakhmatova — S. Yu. Yunusov Institute of the Chemistry of Plant Substances, Academy of Sciences of the Republic of Uzbekistan, Tashkent 100170, Uzbekistan

Khayrulla M. Bobakulov — S. Yu. Yunusov Institute of the Chemistry of Plant Substances, Academy of Sciences of the Republic of Uzbekistan, Tashkent 100170, Uzbekistan

Shamsur S. Sagdullaev — S. Yu. Yunusov Institute of the Chemistry of Plant Substances, Academy of Sciences of the Republic of Uzbekistan, Tashkent 100170, Uzbekistan

Rano P. Zakirova — S. Yu. Yunusov Institute of the Chemistry of Plant Substances, Academy of Sciences of the Republic of Uzbekistan, Tashkent 100170, Uzbekistan

Complete contact information is available at: https://pubs.acs.org/10.1021/acsomega.2c01688

**Notes**

The authors declare no competing financial interest.

**ACKNOWLEDGMENTS**

We thank Dr. Alimjan M. Nigmatullaev {S. Yu. Yunusov Institute of the Chemistry of Plant Substances, Academy of Sciences of the Republic of Uzbekistan (ICPS), Uzbekistan} for his identification of *A. nudi flora*. This work was supported by a Basic Grant from the Innovation Ministry of the Republic of Uzbekistan (VA-ACS-S-008 to K.K.). K.U.K. and K.T. are grateful to ICPS for financial support to send K.U.K. to K.T.’s laboratory (Kyushu University, Japan) and to invite K.T. to ICPS, which enabled their smooth collaboration.

**REFERENCES**

1. Clark, M.; Tilman, D. Comparative analysis of environmental impacts of agricultural production systems, agricultural input efficiency, and food choice. *Environ. Res. Lett.* 2017, 12, 064016.

2. WIKIPEDIA. Projections of population growth. https://en.wikipedia.org/wiki/Projections_of_population_growth (accessed Mar 17, 2022).

3. Zikankuba, V. L.; Mwanyika, G.; Ntwenya, J. E.; James, A. Pesticide regulations and their malpractice implications on food and environment safety. *J. Agric. Food Chem.* 2018, 66, 6504–6512.

4. (a) Lambeth, C.; Jeanmart, S.; Luksch, T.; Plant, A. Current challenges and trends in the discovery of agrochemicals. *Science* 2013, 341, 742–746. (b) Zhao, X.; Cui, H.; Wang, Y.; Sun, C.; Cui, B.; Zeng, Z. Development strategies and prospects of nano-based smart pesticide formulation. *J. Agric. Food Chem.* 2018, 66, 6504–6512.

5. (a) Sparks, T. C.; Wessels, F. J.; Lorsbach, B. A.; Nugent, B. M.; Watson, G. B. The new age of insecticide discovery—the crop protection industry and the impact of natural products. *Pestic. Biochem. Physiol.* 2019, 161, 12–22. (b) Beck, J. J.; Alborn, H. T.; Block, A. K.; Christensen, S. A.; Hunter, C. T.; Rering, C. C.; Seidl-Adams, I.; Stuhl, C. J.; Torto, B.; Tumlinson, J. H. Interactions among plants, insects, and microbes: Elucidation of inter-organismal chemical communications in agricultural ecology. *J. Agric. Food Chem.* 2018, 66, 6663–6674.

6. Plant extracts are not necessarily safe. For a recent example, see: Phupong, C.; Suenaeg, M.; Bhoopong, P.; Chongkla, W.; Jariyang, G.; Karak, M.; Yoshida, K.; Phupong, C.; Torikai, K. Precise 1H- and 13C-NMR reassignment of dehydrodecarbana by 10 mg INAD-EQUATE and in silico analysis: With an alert for its toxicity. *Tetrahedron* 2020, 76, 131310.

7. (a) Palavra, A. M. F.; Coelho, J. P.; Barroso, J. G.; Rauter, A. P.; Fareleira, J. M. N.; Mainar, A.; Urieta, J. S.; Nobre, B. P.; Gouveia, L.; Mendes, R. L.; Cabral, J. M. S.; Novais, J. M. Supercritical carbon
dioxide extraction of bioactive compounds from microalgae and volatile oils from aromatic plants. *J. Supercri. Fluids* 2011, 60, 21–27.  
(b) Jozwiak, A.; Brzozowski, R.; Bujnowski, Z.; Chojnacki, T.; Swiezeewska, E. Application of supercritical CO2 for extraction of polysiprenoid alcohols and their esters from plant tissues. *J. Lipid Res.* 2013, 54, 2023–2028.  
(c) Abd Hamid, I. A.; Ismail, N.; Abd Rahman, N. Supercritical carbon dioxide extraction of selected herbal leaves: An overview. *IOP Conf. Ser.: Mater. Sci. Eng.* 2018, 358, 012037.

(8) Rahmatova, M. J.; Mamatkulova, N. M.; Khodjaniyazov, K. U.; Mukkaramov, N. I.; Khidyrova, N. K. Accumulation dynamics of polyisoprenoid alcohols and their esters from plant tissues. *Chem. Nat. Compd.* 2013, 49, 1693–1697.  
(f) Quijano, C. E.; Linares, D.; Shakhidoyatov, R. K.; Khushbaktova, Z. A. Results of experimental studies of the immunotropic action of polyisoprenols extracted from Alcea nudiflora extracts. *Pharm. Chem. J.* 2016, 50, 29–32.  
(c) Kukina, T. P.; Sal’nikova, O. I.; Khidyrova, N. K.; Rahmatova, M. D.; Pankrushina, N. A.; Grazhdannikov, A. E. Aliphatic and terpene constituents of Alcea nudiflora extracts. *Chem. Nat. Compd.* 2016, 52, 285–287.  
(d) Khidyrova, N. K.; Rahmatova, M. Z.; Kukina, T. P.; Shakhidoyatov, R. K.; Shakhidoyatov, K. M. Polyisoprenols and triterpenoids from leaves of Alcea nudiflora. *Chem. Nat. Compd.* 2012, 48, 180–184.

(9) Opiyo, S. A. Insecticidal Activity of Ocimum Suave Willd Extracts and Compounds against Sitophilus Zeamais Motschulsky. *Basic Sci. Med.* 2020, 9, 32–37.

(10) (a) Babushok, V. I.; Linstrom, P. J.; Zenkevich, I. G. Retention indices for frequently reported compounds of plant essential oils. *J. Phys. Chem. Ref. Data* 2011, 40, 043101.  
(b) Chung, H. Y.; Yung, I. K. S.; Ma, W. C. J.; Kim, J.-S. Analysis of volatile components in frozen and dried scallops (Patinopecten yessoensis) by gas chromatography/mass spectrometry. *Food Res. Int.* 2002, 35, 43–53.  
(c) Wei, A.; Mura, K.; Shibamoto, T. Antioxidative activity of volatile chemicals extracted from beer. *J. Agric. Food Chem.* 2001, 49, 4097–4101.  
(d) Chung, T. Y.; Eisicher, J. P.; Shibamoto, T. Volatile compounds isolated from edible Korean chamchwi (Aster scaber Thunb). *J. Agric. Food Chem.* 1993, 41, 1693–1697.  
(e) Choi, H.-S. Lipolytic effects of citrus peel oils and their components. *J. Agric. Food Chem.* 2006, 54, 3254–3258.

(f) Quiijano, C. E.; Linares, D.; Pino, J. A. Changes in Volatile compounds of fermented Cereza Agria [Phyllanthus acidus (L.) Skells] fruit. *Flavour Fragr. J.* 2007, 22, 392–394.

(11) Ikbal, C.; Pavela, R. Essential oils as active ingredients of botanical insecticides against aphids. *J. Pestic. Sci.* 2019, 92, 971–986.

(12) Hummelbrunner, L. A.; Isman, M. B. Acute, sublethal, antifeedant, and synergistic effects of monoterpenoid essential oil in honey bee (Apis mellifera) larvae reared in vitro. *Pest Manag. Sci.* 2014, 70, 140–147.

(13) Gaire, S.; Scharf, M. E.; Gondhalekar, A. D. Toxicity and neurophysiological impacts of plant essential oil components on the generalist predator Nesidiocoris tenuis. *Phytoparasitica* 2019, 47, 683–692.