Journal of King Saud University – Science 34 (2022) 101913

Contents lists available at ScienceDirect

Journal of King Saud University – Science

journal homepage: www.sciencedirect.com

Original article

Terpenes and phenolics in alcoholic extracts of pine needles exhibit biocontrol of weeds (*Melilotus albus* and *Asphodelus tenuifolius*) and insect-pest (*Plutella xylostella*)

Tajwar Alam [a], Ghulam Jilani [a,*], Arshad Nawaz Chaudhry [a], Muhammad Sheeraz Ahmad [b], Rukhsanda Aziz [c], Rizwan Ahmad [d]

[a] Institute of Soil & Environmental Sciences, PMAS Arid Agriculture University, Rawalpindi 46300, Pakistan
[b] University Institute of Biochemistry & Biotechnology, PMAS Arid Agriculture University, Rawalpindi 46300, Pakistan
[c] Department of Environmental Science, International Islamic University, Islamabad 44000, Pakistan
[d] Land Resources Research Institute, National Agricultural Research Centre, Islamabad 44000, Pakistan

ARTICLE INFO

Article history:
Received 3 December 2021
Revised 27 January 2022
Accepted 14 February 2022
Available online 18 February 2022

Keywords:
Pinus roxburghii Sargent
Maceration
Bio-pesticide
Germination inhibition
Diamondback moth
Feeding deterrent

ABSTRACT

Massive use of synthetic chemicals exerts deleterious effects on human and environmental health, which calls for the development of novel alternatives to manage agricultural pests. Botanical pesticides have drawn great interest due to their non-toxic and eco-friendly nature. Therefore, under this study, leaf-needles of chir pine (*Pinus roxburghii*) were extracted by different solvents to obtain effective biochemical. These extracts were evaluated for biocontrol of two weed and an insect-pest species. Pine needles were soaked separately in methanol, ethanol, hot and cold water in the volume to weight ratio of 5:1 (solvent:pine needles) for 7 days. Extraction yield, and contents of organic acids, siloxanes and amides were higher with hot and cold water, while alcohols, ketones, terpenes and phenolic compounds were greater in methanolic and ethanolic extracts. These biochemicals with respective extractants increased quantitatively and progressively but not beyond 7 days. Raw extracts (with variable dilution) were employed topically against two weed species (*Melilotus albus* and *Asphodelus tenuifolius*) and an insect-pest (*Plutella xylostella* L.). Greater weed biocontrol efficacy through soaking of *M. albus* and *A. tenuifolius* weed seeds was exhibited by methanolic extract (100% concentration) with the highest germination inhibition (74% and 65%) followed by ethanolic extract (68% and 64%), respectively. The highest mortality (92%) of *P. xylostella* insects was achieved through methanolic extract bath (20% concentration) after 120 h. These results conclude that alcoholic extracts of pine needle contain more effective biochemicals (e.g., phenols and terpenes) with bio-pesticidal characteristics as compared to that from aqueous extracts. Therefore, the alcoholic extracts bear great potential to develop the bio-pesticides as novel and safe alternatives for weeds as well as insect-pest management.

1. Introduction

Pinus roxburghii Sargent (Pinaceae family) is a widely distributed conifer species in the Himalayan Subtropical Pine Forests eco-region stretching over 3000 km with 7.64 million hectares area along the lower elevations of Himalayas ranging from western to south Asia (Afghanistan, China, Pakistan, India Nepal, Bhutan, and Myanmar). (USDA, 2022). With the common name of chir pine, it covers a total area of 0.362 million hectares only in Pakistan (FAO, 2007). It is rich source of various biochemicals; essential oils obtained from its bark and needles are used as antiseptic, diuretic, stimulant, and liver tonic (Labib et al., 2017). Different plant parts, viz., barks, needles, roots, flowers, seeds and stems are used to prepare botanical pesticides belonging to different families, and are utilized either as plant extracts, essential oils or both. Leaf needles of *P. roxburghii* are rich in phenolic compounds, terpenes, vitamin C, organic acids, tannins, alkaloids, and essential oils (Sajid et al., 2018). Despite the rich source of secondary metabolites, very little

* Corresponding author.
E-mail address: jilani@uaar.edu.pk (G. Jilani).

Peer review under responsibility of King Saud University.

Production and hosting by Elsevier

https://doi.org/10.1016/j.jksus.2022.101913

is known about suitable maceration solvents and chemical compounds in *P. roxburghii* needles to use them as bioherbicide and biopesticide. Further, the pine needle residues after extraction are a biomass resource to be used as potting media for plant nurseries or to make compost. Mupondi et al. (2006) processed pine bark along with goat manure, sewage sludge and effective microorganisms for high quality composting.

Weeds cause serious economic losses to the farmers by competing with principal crop for necessary growth factors and also increase the production cost by intensifying the disease and insect-pest problems (Amare et al., 2014). *Melilotus albus* (broad leaf) and *Asphodelus tenuifolius* (narrow leaf) are the weeds growing in wheat fields during winter (rabi) season. Conventionally, these are managed through chemical weedicides, which have deleterious impacts on environment and other non-targeted organisms (Bocker et al., 2019).

Bioherbicides have the potential to decrease the weeds germination because they are target specific and degrade rapidly in the field (Mahapatra et al., 2021). Reduction in the percentage of weed seed germination and seedling establishment was recorded with increasing concentration of crude aqueous extracts of *Maerua edulis* and *Bobgunnia madagascariensis* plants (Mazhawidza and Mvumi, 2017). *Ficus sycomorus* leaves extract contains numerous groups of secondary metabolites which are responsible for their biological activity (Hossain, 2019). Identification of allelopathic plants and studying their growth retarding effects may lead to the development of novel botanical weedicides.

Management of insect-pests has always remained a challenge to the farmers and researchers alike. Diamondback moth (DBM) *Plutella xylostella* L. (Lepidoptera: Plutellidae) is one of the key pests of leafy vegetables (Zhang et al. 2016). It causes qualitative and quantitative losses, directly as well as indirectly. Worldwide, around US $ 4–5 billion were estimated to control DBM that damages leafy vegetables (Zalucki et al., 2012). It is one of the most difficult pests to control because it develops resistance with different commercially available inorganic pesticides including DDT and bacterial insecticide (Bhattarai and Tiwari, 2021) due to intensive use of these insecticides. Large scale production of Brassica vegetable exacerbated the status of DBM pest (Furlong et al., 2013) and change in climate favors the pest proliferation (Machekano et al., 2017). Therefore, control of DBM has become increasingly difficult.

Bio-pesticides have repellent as well as deterrent effects and encourage the activities of natural predators. Due to increased environmental issues, the promotion of insect-pest control agents of botanical origin has become popular in recent years. Botanical compounds with pesticidal activities have been successfully isolated and commercialized. They include azadiractin from neem (*Azadirachta indica*) (Castillo-Sanchez et al., 2015) and pyrethrin from pyrethrum (*Tanacetum cinerariifolium*) (Alao and Adebayo, 2015). Leaf extract of *Lumnitzera racemose* showed potential toxicity against DBM under controlled conditions (Eswaraiah et al., 2020). Phytochemical screening of crude extract from leaves of *Datura metel* yielded different plant secondary metabolites which exhibited insecticidal activity under in-vitro conditions (Alabri et al., 2014). Therefore, present study was undertaken to determine the biochemical composition of *P. roxburghii* needle extracts and to assess their bioefficacy against two weed species, and an insect-pest.

2. Materials and methods

2.1. Collection and processing of pine needles

Leaf needles of *Pinus roxburghii* were collected from Margala hills forest near Islamabad city, Pakistan (73° 02′ E longitude and 33° 36′ N latitude, 508 masl) during November 2018. Needles were air-dried for 2 weeks in shade between 15 and 25 °C. Dry needles were crushed by a grinder to make a fine powder, screened through a 2 mm size mesh sieve, and stored at 4 °C for further use in extraction.

2.2. Maceration of Pinus roxburghii needles

Powdered pine needles were treated separately with four different solvents, viz., cold water (20 °C), hot water (45 °C), methanol (20 °C) and ethanol (20 °C). Purpose to use various extractants was difference in their polarity as water with higher polarity (1.0) renders greater extraction yield, while solvents with lower polarity (<1.0, e.g., ethanol and methanol) produce less extraction yield (Ashraf et al., 2020). Same procedure was followed for all solvents extraction but in case of hot water extraction temperature was adjusted at 45 °C. Maceration with each solvent was performed in separate conical flask in the ratio of 1:5 (pine needle powder:solvent). Each solvent treatment had four sets to make solution through shaking for four different time durations (1, 3, 5 and 7 days) by covering with aluminum foil. After shaking, filtration was done using filter paper Whatman No. 1 and re-filtered three times. Resulting filtrate was concentrated through rotary evaporator, and the crude extract residues were stored at 4 °C in Eppendorf tubes.

2.3. Extract analysis

Biochemical compounds in the extract residues were measured through GC–MS (Agilant 5973–6890), operating at 70 eV in EI mode. Helium gas served as carrier @ 1 mL min^{-1} along with HP-5MS capillary column (30 m, 0.25 mm, 0.25 μm). Initially, temperature of 50–100 °C was achieved @ 5 °C min^{-1}, later 100–250 °C @ 3 °C min^{-1}, and finally maintained at 260 °C for 20 min. The 2 μL aliquot was aspirated into the column at 200 °C, and the resolution for sample components was attained. Various components were identified via retention time comparison against that of authentic samples (Supleco; Bellefonte, USA) carried out through co-elution and MS analysis and database in the NIST library.

2.4. Weedicidal bioassays

Weedicidal bioassay was initiated by pre-testing of serial concentrations against seeds of *Melilotus albus* and *Asphodelus tenuifolius* weeds. Seeds were used after sterilizing in sodium hypochlorite (0.5%) solution for 2 min. Experiment on comparison of different solvent extracts obtained after 7 days incubation was performed with their 33%, 66% and 100% concentrations. First, the extracts were solubilized in Tween 20® (1%) and later diluted to required concentrations in distilled water. Filter papers were soaked separately with 2 mL methanol, ethanol, cold water and hot water extract solutions in the Petri plates being used for weed seeds germination test. Controls were maintained by moistening the filter paper with Tween 20® (1%) solution. In each Petri plate of the tested extract, 20 weed seeds were put for germination in the growth chamber at 24 °C. Germinated seeds were counted daily until 7th day after placement. Seeds germination was considered when growing plumule extended to 2 mm length. Germination (%) was calculated with the following formula:

$$Germination\ (\%) = \frac{Germinated\ seeds}{Total\ seeds} \times 100$$

2.5. Insect culture

Third-instar larvae of diamondback moth (DBM) were reared in Biocontrol Lab of the Entomology Department, PMAS Arid Agricul-

ture University, Rawalpindi, Pakistan. Diamondback moth culture was maintained on young plants (45–50 days old) of cabbage (Asha cultivar) under 25 °C, 65 ± 5% RH and 16/8 h' light/dark conditions following Qureshi et al. (2020).

2.6. Insecticidal bioassays

Pinus roxburghii needles extracts were tested via leaf dip bioassay following the Park et al. (2002) procedure against 3rd instar larvae of DBM. Three concentrations of extracts, viz., 1%, 3%, 5% were prepared from stock solutions of all solvents incubated for 1, 3, 5 and 7 days. Insecticidal bioassay was conducted by testing these serial concentrations along with respective controls (0%). Fresh cabbage (Asha cultivar) leaves were washed and then dried under room temperature. These leaves were dipped into respective concentrations of each extract for 10 s and allowed to dry at filter paper for 60 s, and then placed in Petri dishes having moist filter paper. Ten 3rd instar larvae of DBM were placed in each Petri dish and mortality data were recorded at time intervals of 24, 48, 72, 96 and 120 h. All extracts obtained at 1, 3 and 5 days were found less efficient as compared to day-7 extract. Therefore, concluding experiment was performed using different solvent extracts obtained at 7 days' maceration with 5%, 10%, 15% and 20% concentrations along with respective controls (0%) by following the procedure described earlier. Mortality percentage was calculated using the formula as mentioned below:

$$\textit{Corrected Mortality}\ \ (\%) = \frac{P - Po}{100 - Po} \times 100$$

here,

P = Percent mortality in treatment.
Po = Percent mortality in control.

2.7. Statistical analysis

Data related to extraction yield were analyzed by following the two-factor factorial arrangement under completely randomized design (CRD) with three replications. Weedicidal and insecticidal bioassays were analyzed through two-factor factorial CRD with six replications. Treatment means were compared using LSD test at 5% level of significance through Statstix 8.1 software. Graphical representations were made through MS-Excel software.

3. Results

3.1. Extraction yield with different solvents

Water (hot and cold) as solvents brought a significant ($P < 0.05$) increase in crude extraction yield as compared to methanol and ethanol (Table 1). The highest average extraction yield was obtained through hot water (as solvent) followed non significantly by cold water, but significantly by both methanol and ethanol. In terms of incubation time, averagely there was non-significant difference from day-1 through day-7 except for hot water. Individually, with methanol the highest extraction yield was obtained at day-5 and the lowest at day-1. In case of ethanol, the highest extraction yield was obtained at day-3 and the smallest at day-7. Cold water as solvent rendered the highest crude extraction yield at day-3 and day-5 followed by day-1 and day-7 with a small difference. Hot water when used as solvent, greater extraction yield was obtained at day-7 with the lowest one at day-1. Interaction between solvents and incubation time was statistically significant ($P < 0.05$) with the highest value with hot water at day-7 and the lowest one with ethanol at day-1.

3.2. Biochemical composition of extracts

Compounds obtained from all the extracts after 1, 3, 5 and 7 days' incubation are shown in Table 2. In cold water extract, the identified compounds at day-1 were; acids, amides, ketones, aldehydes, phenols and alcohols. At day-3 and day-5, greater contents of acids, siloxanes, amides, aldehydes, phenols and alcohols were achieved. From day-7, higher quantity of organic acids, siloxanes, amides, and phenolic compounds were obtained. Compounds identified from hot water extract at day-1 and day-3 were; acids, siloxanes, amides, ketones, phenols and alcohols. From day-5, compounds present in greater amount were acids, siloxanes, amides, and phenols while from day-7, acids, siloxanes and phenols were obtained. Compounds identified from the methanol extract of day-1 were; acids, siloxanes, terpenes, phenols, alcohols, ketones, aldehydes and alkanes. Similar compounds were recorded in day-3, 5 and day-7. Compounds obtained from ethanolic extract at day-1, 3, 5 and day-7 were; acids, siloxanes, terpenes, phenols, alcohols, aldehydes, alkanes and ketones. Overall, methanol and ethanol as solvents yielded higher quantity of phenols, terpenes, alcohols, alkanes, aldehydes and ketones as compared to cold- and hot water.

3.3. Efficacy of Pinus roxburghii needle extracts as bio-weedicide

Bioassays concerning weeds germination inhibition showed that all the tested needle extracts of *Pinus roxburghii* had variable weedicidal activities (Fig. 1). However, methanolic extract exhibited the highest germination inhibition (74%) followed by ethanolic extract (68%) both having significant difference with cold water extract (44%) and hot water extract (42%) for *Melilotus albus* at 100% concentrations. Each lower concentration of all the extracts exhibited significantly less germination inhibition if compared to one another. Trend of germination inhibition was as: 100% > 66% > 33% > 0% concentration. In case of *Asphodelus tenuifolius*, methanolic extract displayed the highest germination inhibition (65%) followed by ethanolic extract (64%), however, both differed significantly with cold water extract (34%) and hot water extract (33%) at 100% concentrations. Germination inhibition reduced gradually and significantly with extract concentrations of 66% and 33%, being minimal in control treatment (0% concentration). Current findings revealed that *P. roxburghii* needles possess active biochemical compounds which could serve as the potential source of bio-weedicides. Moreover, methanol and ethanol proved to be more efficient solvent to obtain the promising weedicidal results.

3.4. Efficacy of Pinus roxburghii needle extracts as bio-insecticide

Needle extracts of *Pinus roxburghii* exhibited promising insecticidal activity against *Plutella xylostella* or diamondback moth (DBM) 3rd instar larvae (Fig. 2). Data analysis depicted that efficacy of extracts was less in the beginning 24 h against 3rd instar larvae. There was positive correlation between extracts efficacy with time passed, and it was enhanced with increasing time, and reached the highest value at 72 h with methanolic extract. The highest larval mortality (92%) was recorded by methanolic extract followed by ethanolic extract (76%), cold water extract (66%) and hot water extract (56%) with 20% extract concentrations at an exposure duration of 120 h. In terms of time, the highest mortality was observed during 72 to 96 h in all the solvent extracts. *Pinus roxburghii* needles extract showed mortality to 3rd instar larvae of DBM slowly after 24 h of treatment but toxicity was increased sharply after 72 h. Higher concentration of each extract rendered significantly higher mortality rate as compared to its respective next lower concentration. So, the highest mortality rate was achieved by 20% and the lowest by 5% concentration. *Pinus roxburghii* extracts exhibited

Table 1
Extraction yield (%) from *Pinus roxburghii* needles at various time intervals using different solvents.

Solvents	Day-1	Day-3	Day-5	Day-7	Means
Methanol	4.01 c*	4.04 c	4.08 c	4.06 c	4.05 C*
Ethanol	3.28 d	3.30 d	3.29 d	3.22 d	3.27 D
Cold water	6.05 b	6.10 b	6.10 b	6.04 b	6.07 B
Hot water	6.10 b	6.14 ab	6.12 b	6.28 a	6.16 A
Means	4.86 NS	4.90	4.90	4.90	

*Means with different letters within same column are significantly different ($P < 0.05$; LSD).
NS = Means in the last row have statistically non-significant difference at $P < 0.05$.

Table 2
Average peak area (%) of compounds detected through GC–MS from *Pinus roxburghii* needles using different extraction solvents.

Biochemical Compounds	Day-1				Day-3				Day-5				Day-7			
	Meth	Eth	HW	CW	Meth	Eth	HW	CW	Meth	Eth	HW	CW	Meth	Eth	HW	CW
Acids	21.2	10.1	57.7	44.7	22.5	10.6	59.2	51.3	23.6	11.3	64.1	56.5	23.8	11.3	69.7	57.3
Siloxanes	27.3	20.2	7.9	4.51	27.6	20.7	8.7	7.79	27.9	20.9	9.67	8.86	28.1	21	12.5	9.8
Amides	–	–	0.21	3.62	–	–	0.37	4.81	–	–	0.51	5.21	–	–	–	5.76
Ketones	0.83	0.53	0.15	–	0.88	0.56	0.24	–	0.92	0.59	–	–	0.95	0.59	–	–
Aldehydes	0.69	0.44	–	0.34	0.72	0.45	–	0.46	0.75	0.49	–	0.05	0.76	0.51	–	–
Phenols	2.68	2.02	0.32	0.27	2.73	2.09	0.39	0.3	2.88	2.15	0.4	0.42	2.93	2.17	0.38	0.44
Alcohols	3.17	6.35	0.15	0.24	3.32	6.44	0.21	0.33	3.48	6.61	–	0.43	3.53	6.62	–	–
Alkanes	0.78	0.81	–	–	0.81	0.86	–	–	0.86	0.89	–	–	0.88	0.89	–	–
Terpenes	5.77	4.09	–	–	5.82	4.15	–	–	5.89	4.19	–	–	5.95	4.22	–	–

Solvents: CW, Cold water; HW, Hot water; Meth, Methanol; Eth, Ethanol.

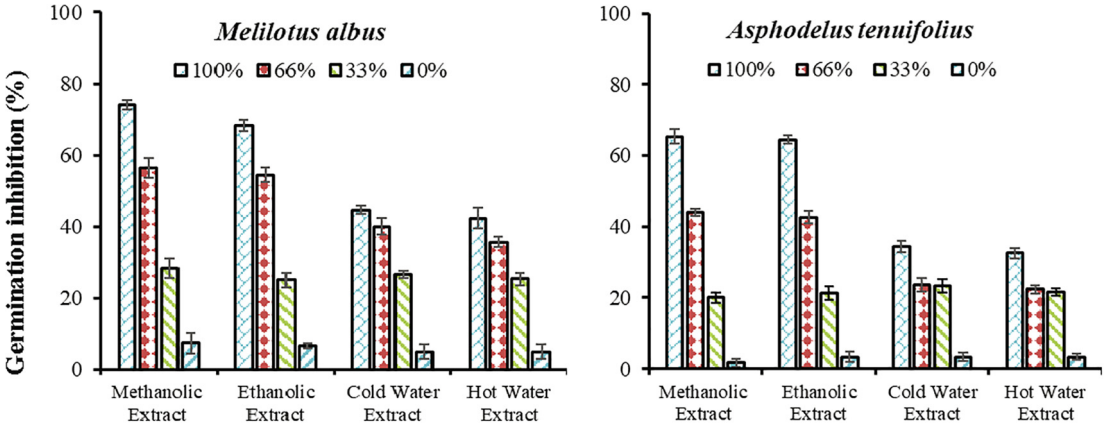

Fig. 1. Effect of different extracts on seed germination inhibition of *Melilotus albus* and *Asphodelus tenuifolius* weeds.

promising activity against the tested pest after 72 h. Current findings revealed that mortality was increased with the increase in concentration as well as exposure time of extract.

4. Discussion

4.1. Impact of solvents on extraction of biochemicals

Employing an appropriate solvent is one of the leading factors for efficient extraction of desired biochemical compounds. Crude extraction yield varies significantly with the polarity of the solvent used; whereas nature of biochemicals extracted varies due to affinity of functional groups of compounds with extractants. Polar solvents (e.g., methanol, ethanol) are more suitable for the extraction of hydrophilic compounds (Sasidharan et al., 2011). During this study, different solvents, viz., methanol, ethanol, hot and cold water were used to extract biochemical compounds from *P. roxburghii* needles. Among from these solvents, significantly higher extraction yield was obtained through hot water (as solvent) followed by cold water, methanol and ethanol. Greater extraction yield with water is due to its higher polarity (1.0); while solvents with lower polarity (<1.0, e.g., ethanol and methanol) produce less extraction yield (Ashraf et al., 2020). Similarly, cold- and hot water as solvent produced higher amounts of organic acids, siloxanes and amides. More efficient extraction of organic acids from sativum - seeds with water has been reported recently by Ashraf et al. (2020). Water extraction also favored to obtain higher amount (7–10%) of tannins from *P. roxburghii* bark (Kumari et al., 2017). Under this study, methanol and ethanol as solvents produced greater amount of terpenes, phenols, and alcoholic compounds. From the viewpoint of natural product, concentration of several biochemicals although with lesser significance as biopesticide (organic acids and siloxanes) was greater with water. Compounds having greater efficiency as biocontrol agents (phenols and terpenes) were obtained in larger content through methanolic and ethanolic extraction.

Methanol is considered as one of the most effective organic solvents for the extraction of valuable phytochemicals from plant material (Parekh et al., 2006). It enables extraction of higher amount of phenols, terpenes and alcoholic compounds, which are

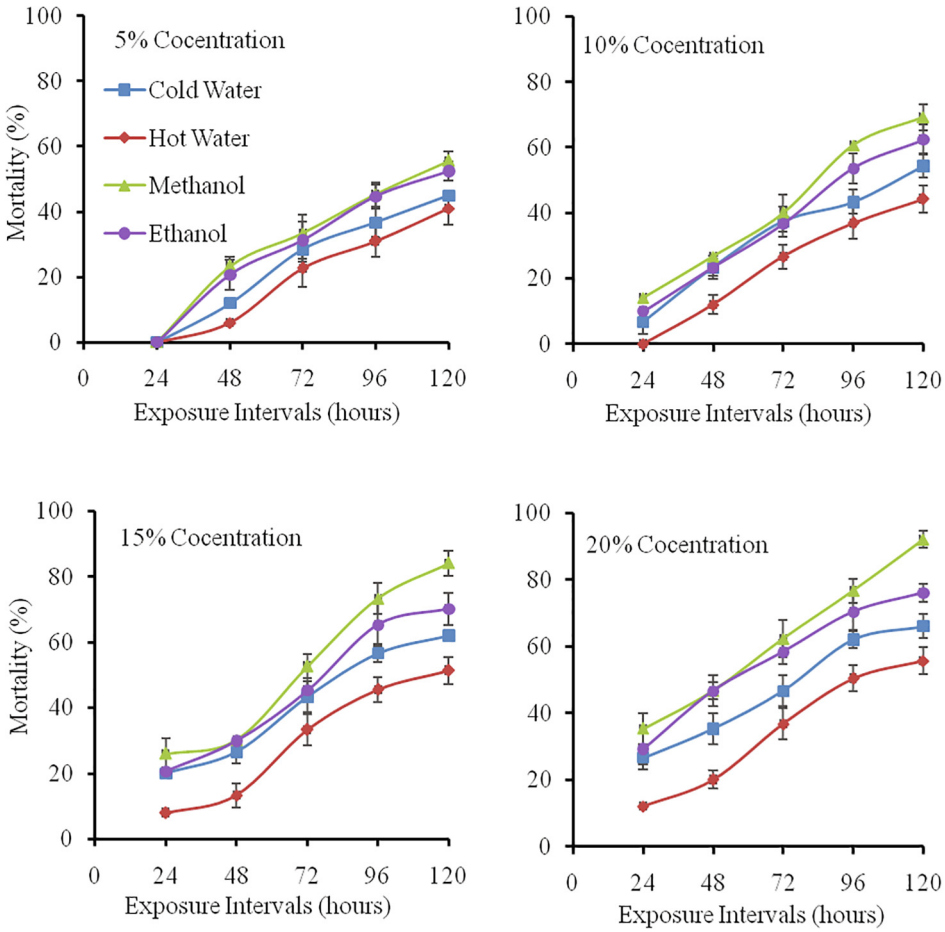

Fig. 2. Effect of different extracts on *Plutella xylostella* mortality at 5, 10, 15 and 20% concentrations.

known to be biocontrol agents (Bogolitsyn et al., 2019). Relatively more phenolic compounds can be extracted through methanol because it acts as a binary solvent (Anokwuru et al., 2011). Moreover, ethanol when used as solvent yielded higher concentration of phenolic compounds as compared to water (Koffi et al., 2010), which are in accordance with current findings. Thus, selection of proper solvent for the targeted group of plant secondary metabolites is quite important.

These findings revealed that extraction of different biochemical compounds from *P. roxburghii* needles depends upon the nature of solvent. Moreover, methanol and ethanol as solvents could be preferably used for extraction of phenolic compounds, terpenes, alcohols and ketones. Whereas, cold- and hot water extraction can be done preferably for organic acids, siloxanes and amides.

4.2. Biocontrol efficacy of different extracts

Several plant-based extracts are being used for weeds management. *Pinus roxburghii* needle extracts efficiently inhibited the germination of two obnoxious weeds (*M. albus* and *A. tenuifolius*). Methanolic extract showed the highest weed seeds germination inhibition followed by ethanol, cold water and hot water. Better performance of methanolic and ethanolic extract could be attributed to the presence of higher amount of terpenes, phenolic and alcoholic compounds in it as compared to that in cold- and hot water extracts. *Pinus roxburghii* bark and needles are good source of polyphenols and terpenes having potential to inhibit the weeds germination (Omezzine et al., 2011). Methanol and ethanol are efficient solvents to extract terpenes and phenolic compounds and had significant weedicidal impacts (Da-Silva et al., 2017). Terpenes, phenols and alcoholic compounds present in plant extracts exhibited greater weedicidal activities (Lim et al., 2017). Polyphenols and terpenes might be affecting the metabolic activities and seed hydration during the process of germination.

Needles extracts of *P. roxburghii* caused variable mortality percentage of the target insect-pest in bioassays. Methanolic extract was more effective to control 3rd instar larvae of *Plutella xylostella* L. when compared with other solvent based plant extracts. It could be attributed to the presence of higher quantity of phenolic compounds and terpenes. Pine needle extracts evaluated in this study have not been used before for insect-pest control as per our knowledge in any part of the world. These results suggest that *P. roxburghii* needles extract could serve as an effective option to insect-pest control. Different plant secondary metabolites and phytochemicals such as essential oils, terpenes, alkaloids, and phenolic compounds have shown pesticidal properties (Copping and Duke, 2007).

5. Conclusions

Needles of *Pinus roxburghii* Sargent were extracted with methanol, ethanol, hot and cold water and analyzed through GC–MS. Results revealed that they contain valuable biochemical compounds including, organic acids, siloxanes, alcohols, phenols and terpenes. More effective compounds could be extracted preferably through methanol/ethanol (alcoholics) for use as bio-pesticide to control 3rd instar larvae of cruciferous crops insect-pest *Plutella xylostella* L. or diamondback moth (DBM). Moreover, these alco-

holic extracts showed promising seed germination inhibition against two notorious weeds, viz., *Melilotus albus* Medik, and *Asphodelus tenuifolius* Cav. Phenols and terpenes affecting negatively to the living cells of weed seeds and insect-pests indicate their toxicity and involvement in the hindrance of metabolic processes. Furthermore, compounds detected in methanolic extract and their greater bio-efficacy indicates that pine needle extract bears great prospects as biocontrol product against targeted weeds and insect-pests under field conditions. These results clearly indicate the potential of pine needles to develop commercial products for weeds and insects biocontrol. Therefore, industrial process would be needed to develop for the large scale production of biopesticides. However, further field studies on larger scale and other weed and insect-pest species are suggested before formulating the commercial products.

Data availability

Information regarding procedures and data are available on request from the corresponding author

CRediT authorship contribution statement

Tajwar Alam: Conceptualization, Investigation, Writing – original draft. **Ghulam Jilani:** Conceptualization, Writing – original draft. **Arshad Nawaz Chaudhry:** Conceptualization, Writing – review & editing. **Muhammad Sheeraz Ahmad:** Conceptualization, Writing – review & editing. **Rukhsanda Aziz:** Writing – review & editing. **Rizwan Ahmad:** Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

References

Alabri, T.H.A., Al Musalami, A.H.S., Hossain, M.A., Weli, A.M., Al-Riyami, Q., 2014. Comparative study of phytochemical screening, antioxidant and antimicrobial capacities of fresh and dry leaves crude plant extracts of *Datura metel* L. J. King Saud Univ. Sci. 26 (3), 237–243.

Alao, F.O., Adebayo, T.A., 2015. Comparative efficacy of *Tephrosia vogelii* and *Moringa oleifera* against insect pests of watermelon (*Citrullus lanatus Thumb*). Int. Lett. Nat. Sci. 35, 71–78.

Amare, T., Sharma, J.J., Zewdie, K., 2014. Effect of weed control methods on weeds and wheat (*Triticum aestivum L.*) yield. World J. Agric. Res. 1, 124–128.

Anokwuru, C.P., Anyasor, G.N., Ajibaye, O., Fakoya, O., Okebugwu, P., 2011. Effect of extraction solvents on phenolic, flavonoid and antioxidant activities of three Nigerian medicinal plants. Nat. Sci. 9, 53–61.

Ashraf, R., Ghufran, S., Akram, S., Mushtaq, M., Sultana, B., 2020. Cold pressed coriander (*Coriandrum sativum* L.) seed oil. In: Cold pressed oils. Academic Press, pp. 345–356.

Bhattarai, A.M., Tiwari, S., 2021. Effects of different bio-rational compounds on mortality of diamond back moth (*Plutella xylostella*) larva under laboratory condition. New thinking and Rejuvenation. RNS 22, 31.

Bocker, T., Möhring, N., Finger, R., 2019. Herbicide free agriculture. A bio-economic modelling application to Swiss wheat production. Agric. Sys. 173, 378–392.

Bogolitsyn, K., Krasikova, A., Gusakova, M., Ivakhnov, A., Gravitis, J., 2019. Selective extraction of terpenoid compounds of *Juniperus communis* L. wood in the medium of a binary solvent (supercritical CO_2 with modifier). Phytochem. Anal. 30, 609–616.

Castillo-Sanchez, L.E., Jiménez-Osornio, J.J., Delgado-Herrera, M.A., Candelaria-Martinez, B., Sandoval-Gío, J.J., 2015. Effects of the hexanic extract of neem (*Azadirachta indica*) against adult whitefly (*Bemisia tabaci*). J. Entomol. Zool. Stud. 3, 95–99.

Copping, L.G., Duke, S.O., 2007. Natural products that have been used commercially as crop protection agents. Pest Manag. Sci.: Formerly Pesticide Sci. 63 (6), 524–554.

da Silva Rodrigues-Corrêa, K.C., Halmenschlager, G., Schwambach, J., de Costa, F., Mezzomo-Trevizan, E., Fett-Neto, A.G., 2017. Dual allelopathic effects of subtropical slash pine (*Pinus elliottii Engelm.*) needles: Leads for using a large biomass reservoir. Ind. Crop Prod. 108, 113–120.

Eswaraiah, G., Peele, K.A., Krupanidhi, S., Kumar, R.B., Venkateswarulu, T.C., 2020. Studies on phytochemical, antioxidant, antimicrobial analysis and separation of bioactive leads of leaf extract from the selected mangroves. J. King Saud Univ. Sci. 32 (1), 842–847.

FAO, 2007. Statistics for APFSOS II - IGF Office, Ministry of Environment, APFSOS II Conference Chiang Mai, Thailand.

Furlong, M.J., Wright, D.J., Dosdall, L.M., 2013. Diamondback moth ecology and management: problems, progress, and prospects. Annu. Rev. Entomol. 58 (1), 517–541.

Hossain, M.A., 2019. A review on *Ficus sycomorus*: A potential indigenous medicinal plant in Oman. J. King Saud Univ. Sci. 31 (4), 961–965.

Koffi, E., Sea, T., Dodehe, Y., Soro, S., 2010. Effect of solvent type on extraction of polyphenols from twenty-three *Ivorian plants*. J. Anim. Plant Sci. 5, 550–558.

Kumari, N., Singh, N., Singh, B., 2017. Phytoconstituents and Pharmacological activity of *Pinus roxburghii* Sarg: A Review. Int. J. Pharm., 241–249

Labib, R., Youssef, F., Ashour, M., Abdel-Daim, M., Ross, S., 2017. Chemical composition of *Pinus roxburghii* bark volatile oil and validation of its anti-inflammatory activity using molecular modelling and bleomycin induced inflammation in Albino Mice. Molecules 22, 64–84.

Lim, C.J., Basri, M., Ee, G.C.L., Omar, D., 2017. Phyto-inhibitory activities and extraction optimization of potent invasive plants as eco-friendly weed suppressant against *Echinochloa colona* (L.) link. Ind. Crop Prod. 100, 19–34.

Machekano, H., Mvumi, B., Nyamukondiwa, C., 2017. Diamondback moth, *Plutella xylostella* (L.) in Southern Africa: research trends, challenges and insights on sustainable management options. Sustainability 9 (2), 91. https://doi.org/10.3390/su9020091.

Mahapatra, S.S., Pattanayak, S., Das, S., Awasthi, L.P., 2021. Impact of crop protection strategies with biopesticides on long, sustainable organic agriculture. In: Biopesticides in Organic Farming CRC Press pp. 29-32.

Mazhawidza, E., Mvumi, B.M., 2017. Field evaluation of aqueous indigenous plant extracts against the diamondback moth, *Plutella xylostella* L. and the rape aphid, Brevicoryne brassicae L. in brassica production. Ind. Crops Prod. 110, 36–44.

Mupondi, L.T., Mnkeni, P.N.S., Brutsch, M.O., 2006. The effects of goat manure, sewage sludge and effective microorganisms on the composting of pine bark. Compost Sci. Utiliz. 14 (3), 201–210.

Omezzine, F., Ladhari, A., Rinez, A., Haouala, R., 2011. Potent herbicidal activity of *Inula crithmoides* L. Sci. Hortic. 130, 853–861.

Parekh, J., Jadeja, D., Chanda, S., 2006. Efficacy of aqueous and methanol extracts of some medicinal plants for potential antibacterial activity. Turk. J. Biol. 29, 203–210.

Park, B.-S., Lee, S.-E., Choi, W.-S., Jeong, C.-Y., Song, C., Cho, K.-Y., 2002. Insecticidal and acaricidal activity of pipernonaline and piperoctadecalidine derived from dried fruits of *Piper longum* L. J. Crop Prot. 21 (3), 249–251.

Qureshi, M.S., Mohsin, A.U., Naeem, M., Shah, K.N., 2020. Parasitism and sex ratio of *Diadegma insulare* (Cresson) (Hymenoptera: Ichneumonidae) against *Plutella xylostella* (Linnaeus) (Lepidoptera: Plutellidae) on cruciferous cultivars under different temperatures. Egypt J. Biol. Pest Control. 13, 1–6.

Sajid, A., Manzoor, Q., Iqbal, M., Tyagi, A.K., Sarfraz, R.A., Sajid, A., 2018. *Pinus roxburghii* essential oil anticancer activity and chemical composition evaluation. EXCLI J. 17, 233–239.

Sasidharan, S., Chen, Y., Saravanan, D., Sundram, K.M., Latha, L.Y., 2011. Extraction, isolation and characterization of bioactive compounds from plants' extracts. Afr. J. Trad. Compl. Alter. Med. 8 (1), 1–10.

USDA, 2022. *Pinus roxburghii* Sarg. Germplasm Resources Information Network. National Germplasm Resources Laboratory, NRS, Beltsville, USA. Accessed. 26-01-2022. URL: https://npgsweb.ars-grin.gov/gringlobal/taxon/taxonomydetail?id=28538.

Zalucki, M.P., Shabbir, A., Silva, R., Adamson, D., Shu-Sheng, L., Furlong, M.J., 2012. Estimating the economic cost of one of the world's major insect pests, Plutella xylostella (Lepidoptera: Plutellidae): just how long is a piece of string. J. Econ. Entomol. 105, 1115–1129.

Zhang, S., Zhang, X., Shen, J., Mao, K., You, H., Li, J., 2016. Susceptibility of field populations of the diamondback moth, *Plutella xylostella*, to a selection of insecticides in Central China. Pestic. Biochem. Physiol. 132, 38–46.