



Evaluation of *Ficus nitida* Allelopathic Potential and the Most Efficient Application Method for Controlling Weeds Associated with Sunflower Plant

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Abstract

Allelopathy is a phenomenon by which plants positively or negatively affect neighboring plants by releasing allelopathic compounds. These allelochemicals are secondary metabolites found in different concentrations in shoots, roots, leaves, flowers, and even pollen grains. Allelochemicals have potential as natural bioherbicides for controlling weeds when applied in the form of extracts or through intercropping, cover cropping, and mulching. The present study was conducted to investigate the allelopathic potential of *Ficus nitida* leaves against *Echinochloa crus-galli* L. and *Corchorus olitorius* L. weeds associated with sunflower plant via two application methods (mixing of *F. nitida* leaf powder with soil and foliar spray of *F. nitida* leaf powder alcoholic extract). Two pot experiments were carried out for the two summer seasons of 2020 and 2021 in the greenhouse of the National Research Centre (NRC), Dokki, Giza, Egypt. Nine treatments were applied in a completely randomized block design. Three treatments were applied before sowing, namely *F. nitida* leaf powder was mixed with the soil at rates of 15, 30, and 45 g/pot. The other three treatments of alcoholic leaf powder extract of *F. nitida* were sprayed twice on both plants and weeds at 10, 20, and 30% (w/v) concentrations. Additionally, three check treatments—healthy (sunflower only), unweeded, and both weeds only—were applied for comparison. The recorded results showed that *F. nitida* had an allelopathic bioherbicidal effect on both weeds. By increasing the concentration of *F. nitida* extract, the bioherbicidal potential increased. Moreover, the recorded results showed that foliar spray of alcoholic extract at 30% concentration was the superior application method for controlling weeds. Mixing of *F. nitida* leaf powder at 45 g/pot ranked second after this superior treatment. A noticeable result is that these two superior treatments improved sunflower growth parameters and yield traits. Quantitative estimation of phenolic compounds and flavonoids demonstrated that the concentration of these allelochemicals is higher in alcoholic extract than in water extract. Moreover, phenolic acid concentrations as detected by high-performance liquid chromatography fractionation are higher in alcoholic extract than in water extract.

Keywords Allelopathy · *Echinochloa crus-galli* · *Corchorus olitorius* · Phenolic compounds · High-performance liquid chromatography

Introduction

Sunflower (*Helianthus annuus* L., family Asteraceae) is one of the most important oilseed crops worldwide and has good adaptability to abiotic stresses (Saady et al. 2021a). As it contains high levels of linoleic acid (55–70%), it is mainly

used for human diet and in biofuel production (Premnath et al. 2016). Sunflower is ranked fourth as a source of vegetable oil worldwide, with 9.2% in 2017/2018 (19 MnT/year) after palm oil (36.5%), soybean oil (27.4%), and rapeseed oil (12.5%) (Inovia and Brétignières 2020). Increased yield could be achieved through introducing high-yielding cultivars and effective management of weeds, diseases, and pests. Weeds are considered to be a serious problem, as sunflower is cultivated at low densities that permit the growth of weeds (Ahmed et al. 2016; El-Rokiek et al. 2018).

Weeds are the greatest competitors of agricultural crops regarding water, nutrients, light, and space, and cause qualitative and quantitative reductions in crop yield (El-

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Metwally and Saady 2021a). Weeds cause sunflower yield losses that reach up to 58% (Daugovish et al. 2003). *Echinochloa crus-galli* and *Corchorus olitorius* are common weeds associated with sunflower plants. *Echinochloa crus-galli* (barnyard grass) belongs to the Poaceae family. *E. crus-galli* is a commonly known noxious weed and negatively affects agricultural productivity through competition for natural resources (Bajwa et al. 2015). *E. crus-galli* is a difficult weed to manage because of its high seed productivity and dormancy level (Hossain and Begum 2015). *Corchorus olitorius* (Egyptian spinach or Molokhia jute) belongs to the Malvaceae family. In Egypt, *C. olitorius* is commonly known as an economic vegetable or medicinal herb. However, it also grows as a weed associated with sunflower, causing substantial yield losses in sunflower crop (Ahmed et al. 2016). Despite the great efforts in controlling weeds in different crop plants using several methods (El-Wakeel et al., 2019a; Ameen et al., 2020; Saady et al. 2021b; El-Metwally et al. 2022a), they are still a main problem in the agricultural ecosystem. There is no doubt that chemical synthetic herbicides achieve good results in controlling weeds in several situations (El-Metwally and Saady 2021b), but the repeated use of overdosed herbicides increases the resistance of weeds to herbicides and generates “super weeds” which contaminate our food (Das et al. 2021). Moreover, chemical herbicides pollute the surrounding environment, which, in turn, is reflected in human and animal health. Therefore, natural ecofriendly approaches are safe alternative strategies for controlling weeds (El-Metwally et al. 2022b). Allelopathy is one of these safe tactics. Allelopathy is a phenomenon by which plants positively or negatively affect neighboring plants by release of allelopathic compounds (Rice 1984). The allelopathic compounds, called allelochemicals, are secondary metabolites that are found in different concentrations in shoots, roots, leaves, flowers, and even pollen grains (Bertin et al. 2003). Sathishkumar et al. (2020) concluded that allelochemicals can be applied as natural bioherbicides through extracts, intercropping, cover cropping, and mulching for controlling weeds.

Although the *Ficus* genus contains about 850 species in the Moraceae family, reports on its allelopathic potential are lacking. *Ficus benghalensis* has an allelopathic inhibitory effect on maize and sunflower plants but does not inhibit the growth of mung bean plants (Mohsin et al. 2016). Moreover, El-Masry et al. (2019) documented the allelopathic bioherbicidal effect of *Ficus nitida* leaves on annual weeds associated with faba bean plants. Ladhari et al. (2020) demonstrated the allelopathic phytotoxic impact of *Ficus carica* on weeds, which may be attributed to phenolic or flavonoid compounds. Since *Ficus nitida* and *Ficus microcarpa* are the most common species in Egypt, the current study sheds light on the allelopathic effect of *Ficus nitida*

as a safe method for weed control. Accordingly, this study aimed to assess the allelopathic potential of *F. nitida* leaf powder as well as alcoholic leaf powder extract of *F. nitida* on the growth and yield of sunflower plants and its associated weeds, i.e., *E. crus-galli* and *C. olitorius*, while determining the most appropriate method of application.

Materials and Methods

Preparation of Dry Plant Material

Fresh mature leaves of *Ficus nitida* were collected from the garden of the National Research Centre, Giza, Egypt, and washed with tap water. For several days, *F. nitida* leaves were dried at room temperature in the shade. Using an electric mill, dried plant tissues were ground separately into a fine powder.

Preparation of Alcoholic Extract

Stock solution (50% w/v) was prepared according to Mekonnen (1999). In a 2L Erlenmeyer flask, 500 g of *Ficus nitida* leaf powder was placed and 1 L of 70% ethanol was added. The shaker (200 revolutions per minute) was used to shake the beaker at room temperature for 48 h. To remove debris, the mixture was filtered through four layers of cheesecloth and centrifuged for 30 min. After that the supernatant was filtered through one layer of filter paper (Whatman No. 1). Following filtration, ethanol was evaporated using a rotary evaporator. Using distilled water, three concentrations of 10, 20, and 30% (w/v) were prepared from a 50% stock solution. The extraction method was repeated as needed to ensure that the extracts were always fresh.

Experimental Procedure

Two pot experiments were performed during the two successive summer seasons of 2020 and 2021 in the greenhouse of the National Research Centre (NRC), Dokki, Giza, Egypt. The experiments were conducted in a completely randomized block design with nine replicates. Pottery pots (30 cm in diameter and 0.07 m² area) were filled with equal amounts of sieved sandy loam soil. Seeds of sunflower Cv Sakha 53 were obtained from the Agricultural Research Centre, Egypt. Five seeds of sunflower were sown 2 cm deep from the soil surface. All pots (except the healthy treatment) were infested with the same weight of cockspur grass (*Echinochloa crus-galli* L.) and Jew's mallow (*Corchorus olitorius* L.) weed seeds and mixed thoroughly. Nine treatments were applied in this investigation. *F. nitida* leaf powder was mixed with the soil surface before sowing in

three different treatments at rates of 15, 30, and 45 g/pot. The corresponding three treatments of *F. nitida* alcoholic extracts were sprayed at 10, 20, and 30% (w/v) after the pots were sown. At 14 and 21 days after sowing (DAS), extracts were applied twice using a hand sprayer at a rate of 50 ml/pot. In addition, distilled water was used to apply three control treatments, including healthy, mixed, and both weeds only, for comparison. All treatments were kept under greenhouse settings, and all cultural techniques were used, particularly fertilization and irrigation.

Studied Parameters

Weeds

Three replicates were collected from each treatment at 45 and 70 DAS. Fresh and dry weights of both *E. crus-galli* and *C. olitorius* (g/pot) were recorded.

Sunflower Plants

Growth Parameters Three replicates of sunflower plants were taken from each treatment at 45 and 70 DAS in order to record the following parameters: plant height, number of leaves per plant, fresh weight, dry weight per plant, and SPAD value (Minolta 2013; only at 45 DAS).

Yield and Yield Attributes Sunflower plants from each treatment were sampled at harvest (100 DAS) to record the head diameter, fresh weight per plant, dry weight per plant, number of seeds per head, weight of seeds per plant, and weight of 100 seeds.

Quantitative Estimation of Total Phenolic Compounds and Total Flavonoids in the Leaf Powder and Alcoholic Leaf Extract of *Ficus nitida* Total phenol and total flavonoids were determined in the leaf powder and alcoholic leaf extract of *Ficus nitida* according to Srisawat et al. (2010).

HPLC Instrumentation for Quantification of Phenolic Acids

High-performance liquid chromatography (HPLC) analyses were conducted on samples of *Ficus nitida* water and alcoholic extracts. A Perkin-Elmer model Lambda 25 (Waltham, MA, USA), double-beam UV/vis spectrophotometer equipped with 10 mm matched silica cells was used for analyte spectra recording. For phenolic acid analysis, a chromatographic system comprised of an Agilent Technologies 1260 series HPLC (Santa Clara, CA, USA) equipped with a 20 μ L sample loop, degasser, quaternary pump, column oven, and diode-array detector was used. At 25 °C, chromatographic separations were performed on an Eclipse C18 column (4.6 mm 250 mm, I.D. 5 mm). At

a flow rate of 0.9 ml/min, the mobile phase consists of water (A) and 0.05% trifluoroacetic acid in acetonitrile (B). The mobile phase was programmed in linear gradient as follows: 0 min (82% A); 0–5 min (80% A); 5–8 min (60% A); 12–15 min (82% A); 15–16 min (82% A); and 16–20 min (82% A). the wavelength detector was monitored at 280 nm. ChemStation software (Agilent) was used for data processing. Analytical grade gallic acid, chlorogenic acid, catechin, methyl gallate, caffeic acid, syringic acid, pyrocatechol, rutin, ellagic acid, coumaric acid, vanillic acid, ferulic acid, naringenic acid, daidzein, quercetin, cinnamic acid, apigenin, kaempferol, and hesperetin were purchased from E. Merck (Darmstadt, Germany) for preparation of standards. Stock solutions of phenolic acid standards (1000 μ g ml⁻¹) were prepared by dissolving appropriate amounts of analytes in methanol and then storing them in a refrigerator at 4 °C. Gradient elution was used in this study: 5 μ L of each prepared standard phenolic acid was injected into the HPLC system and the concentration at a certain area was recorded as shown in Table 1 and Fig. 1. Both *F. nitida* water and alcoholic extracts samples were filtered using 0.42 μ m Millipore filters before injection and 5 μ L of each was injected directly to HPLC system. The column temperature was maintained at 40 °C.

Statistical Analysis

Using the CoStat software program, version 6.303 (2004; <https://www.cohortsoftware.com/costat.html>), the combined ANOVA for the data of the two seasons was analyzed

Table 1 Standard phenolic concentrations (μ g/ml) and areas

Standard phenolic acids	Conc. (μ g/ml)	Area (mm ²)
Gallic acid	15.0	168.18
Chlorogenic acid	50.0	384.69
Catechin	7.0	306.47
Methyl gallate	15.0	251.96
Caffeic acid	18.0	227.47
Syringic acid	17.2	240.93
Pyrocatechol	40.0	259.34
Rutin	61.0	556.13
Ellagic acid	120.0	198.30
Coumaric acid	20.0	627.88
Vanillin	12.9	313.66
Ferulic acid	20.0	310.80
Naringenin	30.0	265.92
Daidzein	35.0	516.94
Quercetin	40.0	328.44
Cinnamic acid	10.0	517.65
Apigenin	50.0	660.21
Kaempferol	60.0	501.87
Hesperetin	20.0	380.89

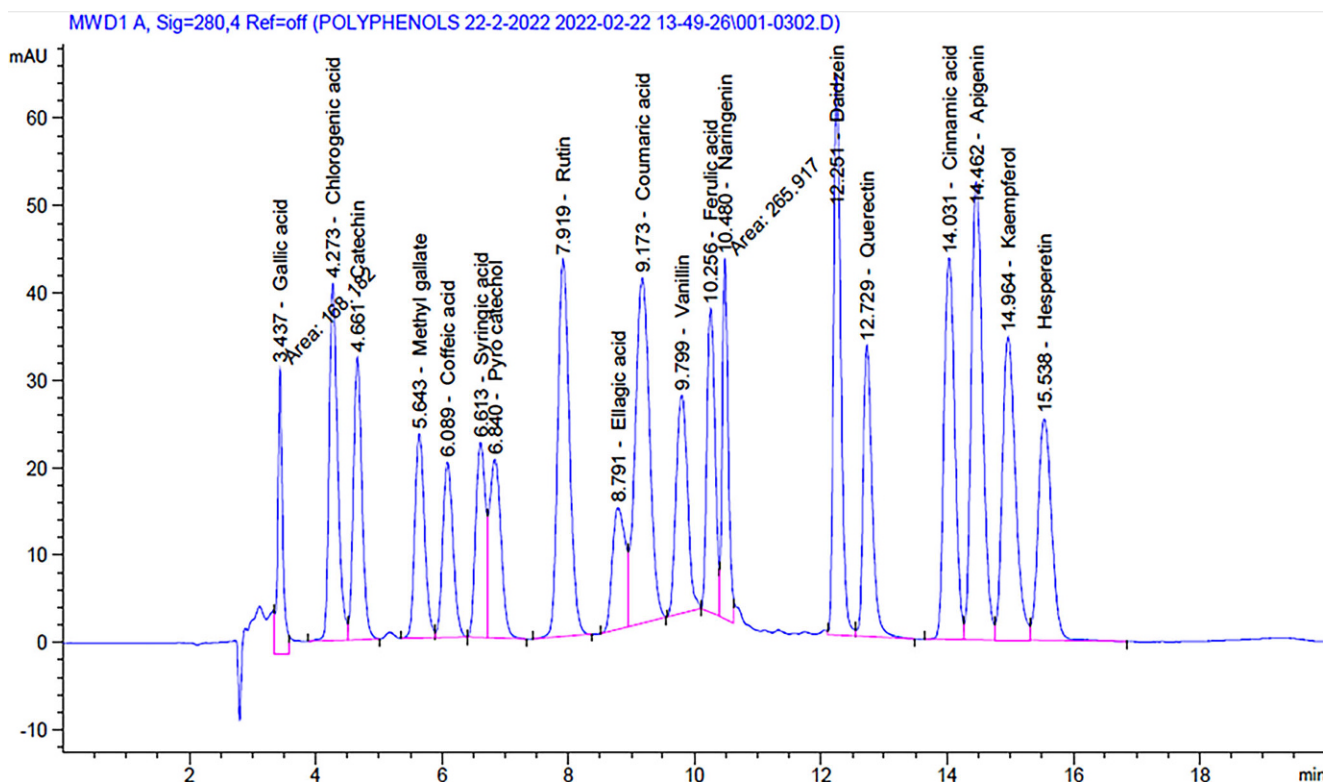


Fig. 1 Standard phenolic acid concentrations ($\mu\text{g/ml}$) and areas

according to Casella (2008). Treatment means were compared using the least significant difference (LSD) at 0.05 probability.

Results

Weeds

Results recorded in Table 2 show that two application methods of *Ficus nitida*—as a powder mixed with soil or foliar spray of alcoholic extract—at successive rates significantly reduced the fresh and dry weight of both *Echinochloa crus-galli* and *Corchorus olitorius* weeds at 45 and 70 DAS as compared to the corresponding weeded control. The rate of reduction of each weed was directly proportional to the applied *F. nitida* concentration. At 45 DAS, *F. nitida* mixing at a rate of 45 g/pot and alcoholic extract at 30% concentration were superior treatments that scored inhibition for both weeds. These superior treatments were followed by *F. nitida* incorporation at rates 30 and 15 g/pot.

At 70 DAS, the response of both weeds became clear. However, *F. nitida* mixing with soil at 45 g/pot was the treatment most effective in controlling *E. crus-galli*, whereas foliar spray application at 30% was the superior treatment in controlling *C. olitorius*. The powder of *F. nitida* mixed with soil at 30 g/pot and foliar spray at 20% were the second-

ranked treatments after highly concentrated treatments for controlling both weeds.

Sunflower Growth Parameters

At 45 DAS, the results in Table 3 illustrate the response of sunflower growth parameters as plant height, number of leaves per plant, fresh weight of plant, dry weight of plant, and SPAD value. The recorded results show that healthy sunflower scored the highest growth parameters. However, the two application methods of *F. nitida* allelopathic materials at the highest rates significantly increased sunflower growth parameters, which are ranked second after the healthy control treatment. It is worth mentioning that mixing of *F. nitida* with soil is more efficient than alcoholic *F. nitida* extract in inducing growth parameters. While at 70 DAS, alcoholic *F. nitida* extract scored higher in growth parameters than mixing *F. nitida* with soil.

As shown in Table 4, at 70 DAS, use of *F. nitida* allelopathic materials either as foliar spray or mixing (at the highest rates) with soil achieved good results by exhibiting higher growth parameters than healthy control. Moreover, it is observed that *F. nitida* foliar spray also performed better than *F. nitida* mixing with soil. *F. nitida* foliar spray at 30% concentration and *F. nitida* mixing with soil at 45 g/pot were superior treatments and increased sunflower dry weight by about 103.23 and 71.10% over unweeded control, respec-

Table 2 Effect of different concentrations of *Ficus nitida* leaf powder or alcoholic extract on fresh and dry weight of *Echinochloa crus-galli* and *Corchorus olitorius* (g/pot) at 45 and 70 days after sowing (combined analysis of the two seasons)

Treatments		At 45 DAS				At 70 DAS			
		<i>E. crus-galli</i>		<i>C. olitorius</i>		<i>E. crus-galli</i>		<i>C. olitorius</i>	
		Fresh weight (g)	Dry weight (g)	Fresh weight (g)	Dry weight (g)	Fresh weight (g)	Dry weight (g)	Fresh weight (g)	Dry weight (g)
Control	Sunflower only	–	–	–	–	–	–	–	–
	<i>E. crus-galli</i> + <i>C. olitorius</i>	84.35	14.61	76.17	17.10	154.51	57.46	106.32	22.36
	Unweeded	52.38	10.26	53.04	10.51	83.65	32.19	71.63	14.93
<i>Ficus nitida</i> mixing with soil (g/pot)	15	30.78	7.24	42.15	7.96	50.39	19.34	62.82	13.35
	30	23.95	5.57	37.65	7.02	36.59	14.18	56.41	11.96
	45	18.68	4.32	28.16	5.04	25.53	9.91	39.12	8.25
Foliar spray of <i>Ficus nitida</i> alcoholic extract	10%	46.60	8.86	36.23	6.55	63.41	25.35	60.25	13.12
	20%	37.83	8.26	33.94	6.20	48.98	18.86	46.64	11.51
	30%	23.72	5.51	18.02	3.88	33.58	12.89	27.31	5.64
LSD at 0.05		2.28	1.15	2.46	1.33	3.93	1.92	3.43	1.25

DAS days after sowing, LSD least significant difference

Table 3 Effect of different concentrations of *Ficus nitida* leaf powder or alcoholic extract on growth parameters of sunflower plants at 45 days after sowing (combined analysis of the two seasons)

Treatments		Growth parameters				
		Plant height (cm)	No. of leaves/plant	Fresh weight of plant (g)	Dry weight of plant (g)	SPAD value
Control	Sunflower only	83.7	21.5	50.89	10.43	38.4
	<i>E. crus-galli</i> + <i>C. olitorius</i>	0.0	0.0	0.00	0.00	0.0
	Unweeded	59.1	14.2	25.57	4.97	31.8
<i>Ficus nitida</i> mixing with soil (g/pot)	15	67.4	16.3	30.81	6.2	33.1
	30	73.2	17.4	36.24	7.24	37.1
	45	82.7	18.5	43.54	8.71	37.9
Foliar spray of <i>Ficus nitida</i> alcoholic extract	10%	62.3	15.7	29.34	5.89	33.9
	20%	71.2	16.8	34.68	6.79	35.7
	30%	78.1	17.9	41.54	8.29	36.0
LSD at 0.05		1.9	0.9	1.51	0.64	1.5

LSD least significant difference

Table 4 Effect of different concentrations of *Ficus nitida* leaf powder or alcoholic extract on growth parameters of sunflower plants at 70 days after sowing (combined analysis of the two seasons)

Treatments		Growth parameters			
		Plant height (cm)	No. of leaves/plant	Fresh weight of plant (g)	Dry weight of plant (g)
Control	Sunflower only	114.7	27.7	70.5	16.98
	<i>E. crus-galli</i> + <i>C. olitorius</i>	0.00	0.00	0.00	0.00
	Unweeded	87.4	21.2	42.8	10.83
<i>Ficus nitida</i> mixing with soil (g/pot)	15	95.3	23.4	53.2	12.92
	30	108.6	25.5	60.0	14.55
	45	117.3	28.2	75.9	18.53
Foliar spray of <i>Ficus nitida</i> alcoholic extract	10%	102.7	24.6	55.2	13.32
	20%	112.9	26.5	61.8	14.98
	30%	125.0	29.4	88.6	22.01
LSD at 0.05		4.4	1.6	1.5	1.33

LSD least significant difference

Table 5 Effect of different concentrations of *Ficus nitida* leaf powder or alcoholic extract on sunflower yield and its attributes (combined analysis of the two seasons)

Treatments		Yield and yield attributes parameters					
		Head diameter (cm)	FW of head (g)	DW of head (g)	No. of seeds/head	Weight of seeds/head (g)	Weight of 100 seeds (g)
Control	Sunflower only	10.7	78.50	18.96	336	13.07	3.12
	<i>E. crus-galli</i> + <i>C. olitorius</i>	0.0	0.00	0.00	000	0.00	0.00
	Unweeded	6.2	28.74	7.95	187	6.78	1.78
<i>Ficus nitida</i> mixing with soil (g/pot)	15	7.5	42.34	10.44	230	8.19	2.22
	30	8.6	59.09	12.58	273	11.15	2.63
	45	11.0	82.84	20.61	445	14.72	3.51
Foliar spray of <i>Ficus nitida</i> alcoholic extract	10%	8.2	51.06	11.47	256	9.81	2.47
	20%	9.4	64.29	15.99	305	11.92	2.94
	30%	11.6	88.14	24.63	470	15.16	3.98
LSD at 0.05		1.1	3.41	1.73	4.3	1.05	0.17

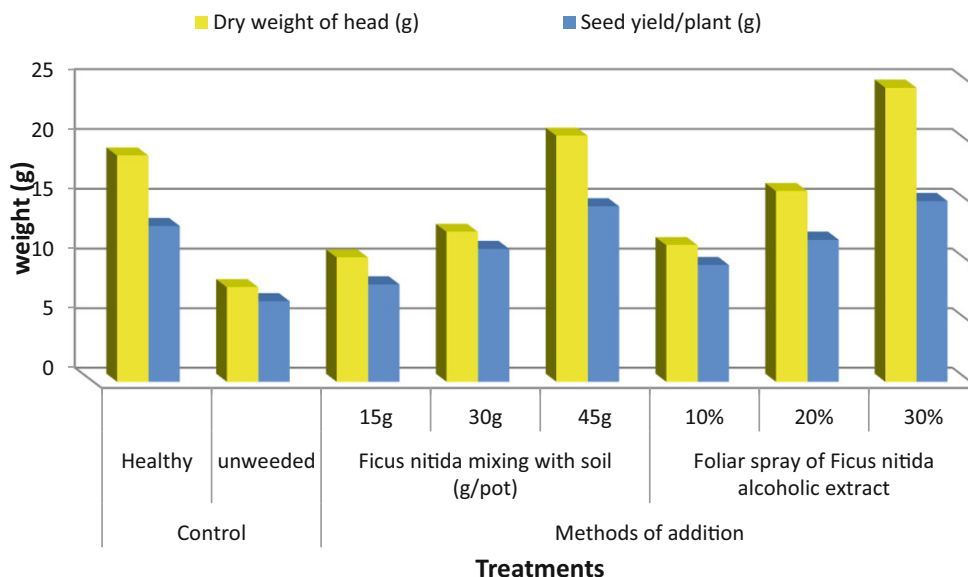
LSD least significant difference, DW dry weight, FW fresh weight

tively. All growth parameters were increased by increasing the concentration of *F. nitida* in both application methods. On the other hand, unweeded control gave the lowest values of all growth parameters of sunflower plants at the two ages of growth.

Sunflower Yield and Yield Attributes

Yield and yield attributes of sunflower, i.e., head diameter, fresh weight of head, dry weight of head, no. of seeds per head, weight of seeds per head, and weight of 100 seeds recorded in Table 5 were significantly increased by all applied concentrations of the two application methods, i.e., either spray of alcoholic *F. nitida* extract or mixing of *F. nitida*, as compared to unweeded control. All sunflower yield

and yield attributes were increased by increasing the concentration of both bioherbicidal application methods. The highest values of all yield and yield attributes of sunflower were recorded for the foliar spray of *F. nitida* alcoholic extract at 30%, followed by *F. nitida* mixing with soil at 45 g/pot, healthy control, and *F. nitida* alcoholic extract at 20% as compared to other treatments (Fig. 2). The previous treatments increased dry weight of the head by 209.81, 159.25, 138.49, and 101.13%; weight of seeds per head by 123.60, 117.11, 92.77, and 75.81%; and weight of 100 seeds by 123.60, 97.19, 75.28, and 65.17%, respectively, as compared to the corresponding unweeded control. Not only did treatments of *F. nitida* alcoholic extract at 30% and *F. nitida* mixing with soil at 45 g/pot alleviate the harmful effect of the two weeds, but they also increased the plant yield over

Fig. 2 Effect of different concentrations of *Ficus nitida* leaf powder or alcoholic extract on dry weight of head (g) and seed yield per plant (g) of sunflower (combined analysis of the two seasons)

that of the corresponding control free from weeds (healthy). These treatments increased dry weight of the head by 29.91 and 8.70%, weight of seeds per head by 15.99 and 12.62%, and weight of 100 seeds by 27.56 and 12.50%, respectively, compared to healthy control. Therefore, it could be concluded that alcoholic leaf powder extract of *F. nitida* (foliar spray) at 30% and *F. nitida* leaf powder mixed with soil at 45 g/pot caused moderate reduction in the growth of both weeds (*E. crus-galli* and *C. olitorius*), as shown in Table 2. This effect was consequently accompanied by the maximum increases in sunflower growth as well as yield and yield attributes (Tables 3, 4 and 5).

Quantitative Estimation of Total Phenolic Compounds and Total Flavonoids in *Ficus nitida*

As shown in Table 6, in quantitative estimation of total phenolic compounds and total flavonoids in the leaf powder and alcoholic leaf extract of *Ficus nitida*, values are higher in the alcoholic extract than in powder water extract.

Table 6 Total phenolic compounds and total flavonoids in *Ficus nitida* seed powder and alcoholic leaf powder extract

Materials	Total phenolic compounds (mg/100 g dry weight)	Total flavonoids (mg/100 g dry weight)
<i>Ficus nitida</i> leaf powder (water extract)	53.90	18.83
Alcoholic leaf powder extract of <i>Ficus nitida</i>	97.82	26.07

Table 7 *Ficus nitida* water and alcoholic extract phenolic acid fractionations

Phenolic acids	<i>Ficus nitida</i> water extract		<i>Ficus nitida</i> alcoholic extract	
	Area (mm ²)	Conc. (µg/ml)	Area (mm ²)	Conc. (µg/ml)
Gallic acid	21.93	19.56	22.63	20.18
Chlorogenic acid	4.05	5.26	4.00	5.20
Catechin	0.00	0.00	0.00	0.00
Methyl gallate	0.00	0.00	1.99	1.18
Caffeic acid	8.00	6.33	15.09	11.94
Syringic acid	6.28	4.48	8.17	5.83
Pyrocatechol	0.00	0.00	0.00	0.00
Rutin	0.00	0.00	0.00	0.00
Ellagic acid	5.24	31.73	59.51	360.10
Coumaric acid	20.12	6.41	19.53	6.22
Vanillin	0.00	0.00	2.86	1.18
Ferulic acid	1.54	0.99	1.55	1.00
Naringenin	3.76	4.24	4.09	4.62
Daidzein	13.10	8.87	0.00	0.00
Quercetin	0.00	0.00	3.33	4.05
Cinnamic acid	2.29	0.44	0.00	0.00
Apigenin	0.00	0.00	0.00	0.00
Kaempferol	0.00	0.00	0.00	0.00
Hesperetin	0.00	0.00	0.00	0.00

Detecting of Phenolic Acids in *F. nitida* Extracts by HPLC

By chromatographic fractionation of water and alcoholic extracts of *F. nitida*, retention time and concentration of each phenolic acid are tabulated in Table 7. Gallic acid, chlorogenic acid, caffeic acid, syringic acid, ellagic acid, coumaric acid, ferulic acid, naringenin, and a few other unknown compounds are detected in water and alcoholic extracts. In addition to the aforementioned phenolic acids, daidzein and cinnamic acid were detected only in water extract, whereas methyl gallate, vanillin, and quercetin were detected in alcoholic extract only. It is worthy of mention that the most commonly identified phenolic acids are higher in concentration in alcoholic extract than in water extract, as shown in Figs. 3 and 4.

Discussion

Assessment of *F. nitida* allelopathic potential is very difficult because of the multiple reactions between the environment and plants. For this reason, scientists seek the most

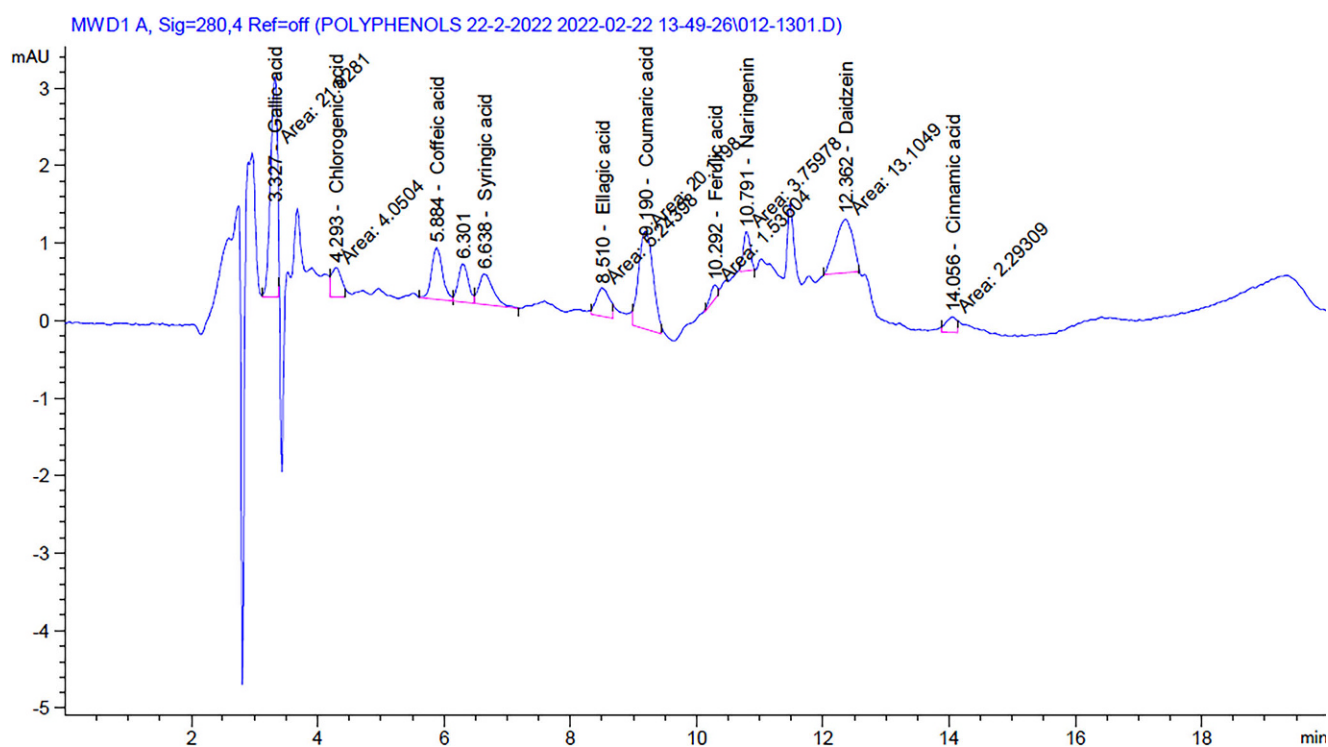


Fig. 3 *Ficus nitida* water extract phenolic acid fractionation

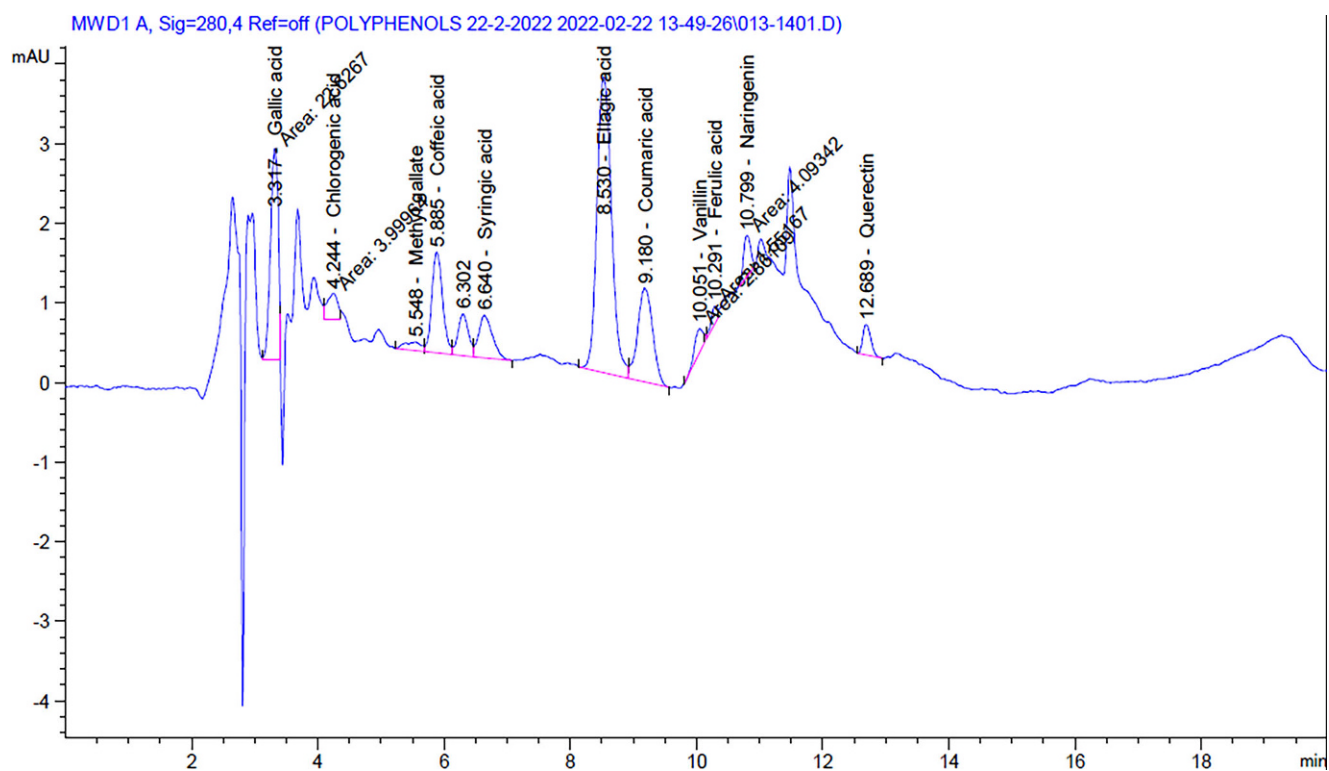


Fig. 4 *Ficus nitida* alcoholic extract phenolic acid fractionation

efficient application method for the natural allelopathic materials under environmental interaction conditions (El-Metwally and El-Wakeel 2019; El-Wakeel et al. 2019b; Saudy et al. 2022). The leaves of *F. nitida* have been proven to have allelopathic efficiency in controlling weeds (El-Masry et al. 2019). Thus, the present study aimed to illustrate the herbicidal effects of allelopathic *F. nitida* leaves and assess the most efficient application method.

The results of the present investigation reveal that *F. nitida* leaf (mixed with soil) and alcoholic leaf powder extract (foliar spray) have a great allelopathic effect in controlling the growth of two annual weeds associated with sunflower plants, i.e., *E. crus-galli* and *C. olitorius*. Moreover, the inhibition rate of weeds at the two stages was increased by increasing the concentration (Table 2). These results are in agreement with the results reported by Manikandan and Jayakumar (2011), who discovered that methanolic leaf and bark extracts of *F. bengalensis* inhibited seed germination, shoot and root length, and biomass weight in *Ipomoea pentaphylla* seedlings. Furthermore, they concluded that the inhibitory effect on weed species is directly proportional to concentration, which could be due to the presence of methanolic soluble allelochemicals such as phenolic acids. According to Jafarihyazdi and Javidfar (2011), allelopathic allelochemicals reduce uptake of nutrients and water by roots; inhibit respiration, photosynthesis, and cell division; and cause slow maturation, which results in delayed or failed reproduction.

Weed management is reflected by positive results concerning growth parameters and yield traits of sunflower (Tables 3, 4 and 5). This enhancement is due not only to the chemical or biological inhibition of weed growth, which increases the plant's competitive ability, but also to the selectivity of the allelochemicals in their action and the plant's responses (Einhellig 1995). However, quantitative estimation of phenolic compounds and flavonoids showed that the allelopathic response of sunflower and its associated weeds may be attributed to these allelopathic compounds, especially as the allelopathic response was increased by increasing the allelochemical concentrations. These results are in agreement with El-Masry et al. (2019), who suggested that allelopathy of *F. nitida* against weeds is correlated with the amount of liberated phenolic acids. Other researchers recorded that *F. benghalensis* leaves and bark extracts have different allelopathic effects on seed germination percentage and early seedling growth parameters of some economic crop plants such as *Zea mays*, *Vigna radiata*, and *Helianthus annuus*. The allelopathic response of these economic crops may be related to allelochemicals such as phenolic acids. These allelochemicals are directly proportional with its concentration (Mohsin et al. 2016; Muhammad et al. 2018).

Most of the allelochemicals detected here are reported as phytotoxic compounds and caused the greatest inhibitory

effect on seed germination, germination rate, and total seedling dry weight of (Cheng and Cheng, 2015; El-Wakeel et al. 2019b). Thus, quantitative and qualitative variation in the detected phenolic compound concentrations may explain the different inhibitory response of weeds to both application methods. Using HPLC, Ladhari et al. (2020) detected phytotoxic allelochemicals in *Ficus carica* that had a bioherbicidal effect on weeds. Moreover, the weed-managing effect is concentration dependent, i.e., the response is directly proportional to concentration (Ladhari et al. 2013). These aforementioned results are in accordance with our findings (Table 7 and Figs. 3 and 4), which prove that *Ficus nitida* leaves contain water-soluble and alcohol-soluble phytotoxic compounds. Consequently, the superiority of the foliar spray application method may be attributed to the concentration of allelochemicals, as most detected phenolic acid concentrations are higher in alcoholic extract than water extract. Also foliar spray may be more efficient than the mixing application due to the microorganism degradation of allelochemicals after they have been liberated into the soil; allelochemicals have a wide range half-lives, from a few hours to a few months (Wang et al. 2007; Bertin et al. 2009). Furthermore, since plant extracts involve several micronutrients, they have an additive beneficial effect on plant growth and development (Elgala et al. 2022).

Conclusion

The results of the present work indicate the possibility of using allelopathic leaf powder and alcoholic leaf powder extract of *F. nitida* as selective bioherbicides in controlling weeds of sunflower. The herbicidal allelopathic activity of *F. nitida* is directly proportional to its concentration. Thus, the highest concentrations of *F. nitida* in both investigated application methods scored the highest herbicidal effect without negatively affecting sunflower growth. However, foliar spray of *F. nitida* alcoholic extract is recommended as being the more effective method in controlling weeds than mixing of *F. nitida* powder with the soil.

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Conflict of interest M.A. El-Wakeel, S.A.A. Ahmed, and N.K. Mes-siha declare that they have no competing interests.

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